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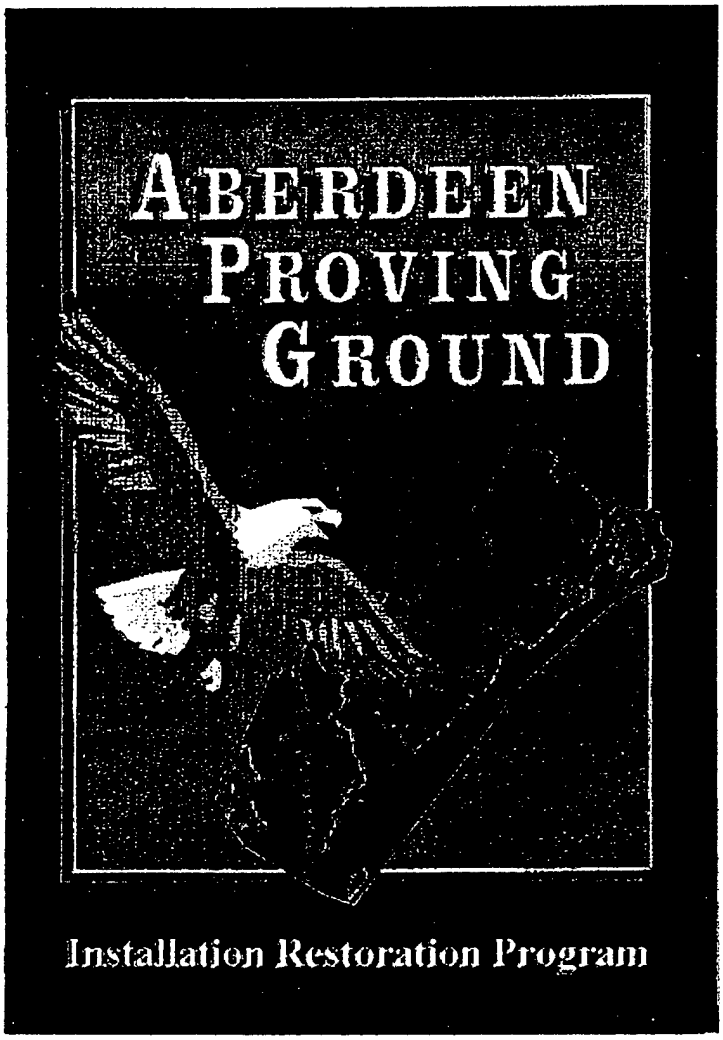


# TERRESTRIAL AND ECOLOGICAL RISK ASSESSMENT AT U.S. ARMY ABERDEEN PROVING GROUND QUALITY ASSURANCE PROJECT PLAN, VOLUME II

## FINAL DOCUMENT

(APPENDICES)

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Aberdeen Proving Ground, Maryland

March 1995

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# APPENDIX A

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* Denotes SOP in draft. Not available for review at this time.

# STANDARD OPERATING PROCEDURE 001
# SAMPLE LABELS

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is included as Figure 001-1. Other formats with similar levels of detail are acceptable.

## 2.0 Material

a. Sample Label
b. Indelible lab marker

## 3.0 Procedure

The following steps describe how to use the sample labelling system:

3.1 As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

a. Project Name

b. Project Number

c. Location/Site I.D. - enter the well # or surface water sampling #, and other pertinent information concerning where the sample was taken.

d. Date of Sample Collection

e. Time of Sample Collection

f. Analyses to be Performed (Note: due to number of analytes, details of analysis should be arranged with lab *a priori.)*

g. Whether Filtered or Unfiltered (water samples only)

h. Preservatives (water samples only)

i. The Number of Containers for the Sample (*e.g.* 1 of 2, 2 of 2)

**3.2** Double-check the label information to make sure it is correct. Detach the label, remove the backing and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

**3.3** Record the Sample Number and designated sampling point in the field logbook, along with the following sample information:

a. Time of sample collection (each logbook page should be dated)

b. The location of the sample

c. Organic vapor meter or photoionization meter readings for the sample (when appropriate)

d. Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)

e. Number of containers required for each sample

f. Whether the sample is a QA sample (split, duplicate or blank)

**3.3.1** A typical logbook entry might look like this:

> 7:35 AM Sample No. MW-3. PID = 35 PPM
> Petroleum odor present. Sample designated MW-3-001.

Note: Duplicate samples will be given a non-existent well number rather than simply using the actual well number with an added prefix or suffix. This will prevent any indication to the lab that this is a duplicate sample. This fictitious well number will be listed in the logbook along with the actual location of the sample.

**3.4** Place the sample upright in the designated sample cooler. Make sure there is plenty of ice in the cooler at all times.

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

**5.1** Note that although incidental odors should be noted in the logbook, it is unwise from a health and safety standpoint to routinely "sniff test" samples for contaminants.

**5.2** No indication of which samples are duplicates is to be provided to the lab.

## 6.0 References

U.S. EPA. 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80

**FIGURE 001-1**
**SAMPLE LABEL**

PROJECT NAME _____ PROJECT NUM. _____

SAMPLE LOCATION/SITE ID _____

DATE: ___/___/___ TIME: ____:____

ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER

FILTERED: [NO] [YES]

PRESERVATIVE: [NONE] [$HNO_3$] [OTHER _____]

SAMPLER: _____

# STANDARD OPERATING PROCEDURE 002
# CHAIN-OF-CUSTODY FORM

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for use of the Chain-of-Custody (COC) Form. An example is provided as part of this SOP. Other formats with similar levels of detail are acceptable.

## 2.0 Material

a. Chain-of-Custody Form
b. Indelible ink pen

## 3.0 Procedure

3.1 Give the site name and project name/number.

3.2 Enter the sample identification code.

3.3 Indicate the sampling dates for all samples.

3.4 List the sampling times (military format) for all samples.

3.5 Indicate "grab" or "composite" sample with an "X."

3.6 Specify the sample location.

3.7 Enter the total number of containers per cooler.

3.8 List the analyses/container volume.

3.9 Obtain the signature of sample team leader.

3.10 State the carrier service and airbill number, analytical laboratory, and custody seal numbers.

3.11 Sign, date, and time the "relinquished by" section.

3.12 Upon completion of the form, retain the shipper copy, and affix the other copies to the inside of the sample cooler, in a zip seal bag to protect from moisture, to be sent to the designated laboratory.

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

None.

## 6.0 References

U.S. EPA. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C., December 1990.

U.S. EPA. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January 1991.

U.S. EPA. 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80

| Proj. no. | Site Name | | | | Num. of Con-tain-ers | | | | | | | | | | LAB : |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Samplers: | | | | | | | | | | | | | | | AIRBILL No: |
| | | | | | | | | | | | | | | | Courier: |
| Date | Time | Comp | Grab | Field Sample No. | | | | | | | | | | | REMARKS |
| | | | | | | | | | | | | | | | |
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| | | | | | | | | | | | | | | | |
| TOTAL | | | | | | | | | | | | | | | |

| Relinquished by: | Date/ time | Received by: | Relinquished by: | Date/ Time | Received by: |
|---|---|---|---|---|---|
| Relinquished by: | Date/ time | Received by: (for lab) | Date/Time | Remarks | |

**FIGURE 002-1**
**EXAMPLE CHAIN-OF-CUSTODY FORM**

# STANDARD OPERATING PROCEDURE 003
# FIELD LOGBOOK

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for recording field survey and sampling information in the Field Logbook.

## 2.0 Material

a. Field Logbook (Teledyne 415 Level Book, or equivalent)[1]
b. Indelible ink pen

## 3.0 Procedure

All information pertinent to a field survey or sampling effort will be recorded in a bound logbook. Each page/form will be consecutively numbered, dated, and signed. All entries will be made in indelible ink and all corrections will consist of line-out deletions that are initialed and dated. The person making the correction will provide a brief explanation for the change. There should be no blank lines on a page. A single blank line or a partial blank line (such as at the end of a paragraph) should be lined to the end of the page. If only part of a page is used, the remainder of the page should have an "X" drawn across it. At a minimum, entries in the logbook will include but not be limited to the following:

a. Project number.

b. Unique, sequential field sample number.

c. Purpose of sampling.

d. Location, description, and log of photographs of each sampling point.

e. Details of the sample site (for example, the elevation of the casing, casing diameter and depth, integrity of the casing, etc.)

f. Name and address of field contact.

g. Documentation of procedures for preparation of reagents or supplies which become an integral part of the sample (e.g., filters and absorbing reagents).

---

[1] Pre-printed, bound forms are approved as well. See SOP 016 for recommended content and format.

h. Identification of sample crew members.

i. Type of sample (for example, groundwater or surface water).

j. Suspected waste composition.

k. Number and volume of sample taken.

l. Sampling methodology, including distinction between grab and composite sample.

m. Sample preservation.

n. Date and time of collection.

o. Collector's sample identification number(s).

p. Sample shipment (for example, name of the laboratory and cartage agent: Federal Express, United Parcel Service, etc.)

q. References such as maps of the sampling site.

r. Field observations (*e.g.* oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, moisture content, layering, U.S.C.S. classification, etc.)

s. Any field measurements made (for example, pH, conductivity, explosivity, water depth, OVA readings, etc.)

t. Signature and date by the personnel responsible for observations.

u. Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a logbook. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The Project Manager will keep a master list of all field logbooks assigned to the Sampling Team Leaders. One logbook kept by the Project Manager will be a master site log of daily activities and will contain the list of field logbooks assigned to Sampling Team Leaders.

**4.0 Maintenance**

Not Applicable.

**5.0 Precautions**

None.

## 6.0 References

U.S. EPA. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C., December 1990.

U.S. EPA. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January 1991.

U.S. EPA. 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80

# STANDARD OPERATING PROCEDURE 004
# SAMPLE PACKING AND SHIPPING

## 1.0 Scope and Application

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

## 2.0 Material

a. Waterproof coolers (hard plastic or metal)
b. Metal cans with friction-seal lids (*e.g.* paint cans)
c. Custody seals
d. Packing material [2]
e. Sample Documentation
f. Ice
g. Plastic Garbage Bags
h. Clear Tape
i. Zip seal plastic bags

## 3.0 Procedure

**3.1** Check cap tightness and verify that clear tape covers label and encircles container.

**3.2** Wrap sample container in bubble wrap or closed cell foam sheets.

**3.3** Enclose each sample in a clear zip-seal plastic bag.

**3.4** Place several layers of bubble wrap, or at least 1" of vermiculite on the bottom of the cooler. Line cooler with open garbage bag, place all the samples upright inside a garbage bag and tie the bag.

**3.5** Double bag and seal loose ice to prevent melting ice from soaking the packing material. Place the ice outside the garbage bags containing the samples.

---

[2] Permissible packing materials are: a) (non-absorbent) bubble wrap or closed cell foam packing sheets; b) (absorbent) vermiculite. Organic materials such as paper, wood shavings (excelsior), and cornstarch packing "peanuts" will not be used.

**3.6** Pack shipping containers with packing material ( closed-cell foam, vermiculite, or bubble wrap). Place this packing material around the sample bottles or metal cans to avoid breakage during shipment.

**3.7** Enclose all sample documentation (i.e., Field Parameter Forms, COCs) in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

**3.8** Seal the coolers with signed and dated custody seals so that if the cooler were opened, the custody seal would be broken. Place clear tape over the custody seal to prevent damage to the seal.

**3.8.1** Refer to SOPs 001, 002, 003, 016 and 39.

**3.9** Tape the cooler shut with packing tape over the hinges and place tape over the cooler drain.

**3.10** Ship all samples via overnight delivery on the same day they are collected if possible.

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

**5.1** Any samples suspected to be of medium/high contaminant concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., paint cans). Label the outer metal container with the sample number of the sample inside.

**5.2** If the sample is suspected of being contaminated with chemical agent, DO NOT use this SOP for packing and shipping methods USE ONLY the packing and shipping methods prescribed in SOP035.

## 6.0 References

U.S. EPA. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C., December 1990.

U.S. EPA. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January 1991.

U.S. EPA. 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80

# STANDARD OPERATING PROCEDURE 005
# DECONTAMINATION

## 1.0 Scope and Application

All personnel or equipment involved in intrusive sampling, or which enter a hazardous waste site during intrusive sampling must be thoroughly decontaminated prior to leaving the site to minimize the spread of contamination and prevent adverse health effects. This procedure describes the normal decontamination of sampling equipment and site personnel.

## 2.0 Material

a. Plastic sheeting, buckets, etc. to collect wash water and rinsates.
b. Approved water.
c. HPLC-grade water.[3]
d. 0.10N Nitric Acid.
e. Non-phosphate laboratory detergent.
f. Reagent grade alcohol [4]
g. Aluminum foil or clean plastic sheeting.
h. Pressure sprayer, rinse bottles, brushes.
i. Plastic garbage bags.
j. 0.01N HCl

## 3.0 Procedure

### 3.1 Sample Bottles

At the completion of each sampling activity the exterior surfaces of the sample bottles must be decontaminated as follows:

**3.1.1** Be sure that the bottle lids are on tight.

**3.1.2** Wipe the outside of the bottle with a paper towel to remove gross contamination.

---

[3] For the purposes of this SOP, HPLC-grade water is considered equivalent to "Deionized ultra filtered water", "Reagent-grade distilled water", and "Deionized organic-free water". The end product being water which is pure with no spurious ions or organics to contaminate the sample. The method of generation is left to the individual contractor.

[4] For the purposes of this SOP, the term "reagent grade alcohol" refers to either pesticide grade isopropanol or reagent grade methanol.

## 3.2 Personnel Decontamination

Review the project Health and Safety Plan for the appropriate decontamination procedures.

## 3.3 Equipment Decontamination

### 3.3.1 Water Samplers

#### 3.3.1.1 Bailers.

After each use, Polytetrafluoroethelyne (PTFE) double check valve bailers used for groundwater sampling will be decontaminated as follows:

a. Discard all ropes used in sampling in properly marked sealable container, or as directed by the health and safety plan. Note: no tubing is to be used in conjunction with a bailer in collecting samples.

b. Scrub the bailer to remove gross(visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent.

c. Rinse off detergent with approved water.

d. Rinse bailer with reagent grade alcohol.

e. Rinse bailer with HPLC-grade water.

f. Rinse bailer with 0.10N Nitric Acid solution.

g. Rinse bailer with HPLC-grade water.

h. Allow bailer to air dry.[5]

i. Wrap bailer in aluminum foil or clean plastic sheeting, or store in a clean, dedicated PVC or PTFE storage container.

j. Dispose of used decon solutions with drummed purge water.

k. Rinse bailer with HPLC-grade water immediately prior to re-use.

---

[5] If the bailer has just been used for purging and is being decontaminated prior to sampling do not air dry. Double rinse with HPLC-grade water and proceed to collect samples.

**3.3.1.2 Pumps**

Submersible pumps will be decontaminated as follows:

a. Scrub the exterior of the pump to remove gross (visible) contamination, using appropriate brush(es) , approved water, and non-phosphate detergent. (Steam cleaning may be substituted for detergent scrub).

b. Calculate the volume of pump plus any tubing which is not disposable and not dedicated to a single well. Pump 3 volumes of non-phosphate laboratory detergent solution to purge and clean the interior of the pump.

c. Rinse by pumping no less than 9 volumes of approved water to rinse.

d. Rinse pump exterior with reagent grade alcohol.

e. Rinse pump exterior with HPLC-grade water.

f. Allow pump to air dry.

g. Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated PVC or PTFE storage container.

h. Prior to reusing pump rinse exterior again with HPLC-grade water. (Double rinse in step "e" above may be substituted for this step).

**3.3.1.3 Dip samplers**

All dip samplers, whether bucket, long-handled, or short-handled (see SOP-007 "Surface Water Sampling") will be decontaminated in the same manner as given in section 3.3.1.1 "bailers" above.

**3.3.1.4 Labware**

Labware such as beakers which are used to hold samples for field measurements, water chemistry, etc. will be decontaminated according to the procedures in 3.3.1.1 "bailers" above.

**3.3.1.5 Water level indicators**

Electric water level indicators, weighted measuring tapes, or piezometers used in the determination of water levels, well depths, and/or NAPL levels will be decontaminated in accordance with section 3.3.1.1 "bailers" above. Clean laboratory wipes may be substituted for brushes. Tapes, probes, and piezometers

should be wiped dry with clean laboratory wipes, and coiled on spools or clean plastic sheeting rather than allowed to air dry.

### 3.3.2 Solid materials samplers

Solid materials samplers include soil sampling probes, augers, trowels, shovels, sludge samplers, and sediment samplers. All will be decontaminated as follows:

a. Scrub the sampler to remove gross(visible) contamination, using appropriate brush(es), approved water, and non-phosphate laboratory detergent.

b. Rinse off detergent with approved water.

c. Rinse sampler with reagent grade alcohol.

d. Rinse sampler with HPLC-grade water.

e. (Non-metallic samplers only) Rinse sampler with 0.10N Nitric Acid solution.

f. (Non-metallic samplers only) Rinse sampler with HPLC-grade water.

g. Allow sampler to air dry.

h. Wrap sampler in aluminum foil clean plastic sheeting, or store in a new zip-seal bag (size permitting) or clean, dedicated PVC or PTFE storage container.

i. Dispose used decon solutions properly according to the site health and safety plan.

j. Rinse sampler with HPLC-grade water immediately prior to re-use.

### 3.3.3 Other sampling and measurement probes

**3.3.3.1** Soil gas sampling probes will be decontaminated as solids sampling devices.

**3.3.3.2** Temperature, pH, conductivity, Redox, and dissolved oxygen probes will be decontaminated according to manufacturer's specifications. If no such soecifications exist, remove gross contaminant and triple rinse probe with

HPLC-grade water. A summary of the decontamination procedures to be used must be included in the instrument-specific SOP.

3.3.3.3 Measuring tapes which become contaminated through contact with soil during field use will be decontaminated as follows:

a. Wipe tape with a clean cloth or laboratory wipe which has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. Rinse cloth in the solution and continue wiping until tape is clean.

b. Wipe tape with a second clean, wet cloth (or lab wipe) to remove soap residues.

c. Dry tape with a third cloth (or lab wipe) and rewind into case, or re-coil tape.

**3.3.4 Drilling Rigs**

All drilling rigs and associated equipment such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out) will be decontaminated prior to site entry after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed prior to site entry there is a likelihood that contamination has accumulated on tires and as spatter or dust enroute from one site to the next (see also Field Investigation Plan, § 6.3.7).

a. Place contaminated equipment in an enclosure designed to contain all decontamination residues (water, sludge, etc.).

b. Steam clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) have been removed.

c. Water used will be taken from an approved source.

d. Containerize, sample, characterize, and dispose of all decontamination residues properly.

**3.3.5 HPLC-grade water Storage**

Dedicated glass storage containers will be used solely for dispensing HPLC-grade water. New HPLC-grade water containers will be decontaminated as follows:

a. Clean with hot tapwater from approved source and non-phosphate laboratory detergent while scrubbing the exterior and interior of the container with a stiff-bristled brush.

b. Rinse thoroughly with approved water.

c. Rinse with 0.01N Nitric acid.

d. Rinse with approved water.

e. Rinse thoroughly with HPLC-grade water.

f. Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

Used HPLC-grade water containers will be decontaminated as follows:

a. Clean the exterior with hot tapwater from an approved source, non-phosphate laboratory detergent, and a stiff-bristled brush.

b. Rinse the exterior thoroughly with HPLC-grade water

c. Rinse the interior twice with pesticide-grade isopropanol.

d. Rinse interior thoroughly with HPLC-grade water.

e. Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

## 4.0 Maintenance

**4.1** HPLC-grade water will be stored only in decontaminated glass containers with aluminum foil lids as stipulated above. The water may not be stored for more than, nor used more than three days after manufacture.

**4.2** HPLC-grade water will be manufactured on-site. An approved tap water source will be used as the influent to the system. Procedures for system set-up, operation and maintenance will conform to manufacturer's specifications.

## 5.0 Precautions

**5.1** Dispose of all wash water, rinse water, rinsates, and other sampling wastes (tubing, plastic sheeting, etc.) in properly marked, sealable containers, or as directed by the health and safety plan.

**5.2** Once a piece of equipment has been decontaminated, be careful to keep it in such condition until needed.

5.3 Do not eat, smoke or drink on site.

**6.0 References**

Site-specific health and safety plan.

# STANDARD OPERATING PROCEDURES 006
# USE OF THE IRDMS NETWORK

## 1.0 LOGIN/LOGOUT OF THE IRDMS (PRI) NETWORK

**1.1** Files may be transferred to, or copied from, the PRI LAN through remote terminals. The connection requires a Hayes-compatible modem operating at 1200 baud, and 3COM network software. The contractor will purchase the 3COM 3+ Remote package, and PRI will supply a "Remote Login Disk" which contains DOS startup files (AUTOEXEC.BAT, COMMAND.COM, IBMBIO.COM, IBMDOS.COM, etc.) and 3COM network programs for startup, remote dialing, etc.

**1.2** To connect to the PRI LAN, the PC must be rebooted from the Remote Login Disk #1. After the standard DOS startup routines are completed, you will get the A> prompt. Change to the drive containing the IRDMS files (C or D).

Type D: <CR>
Get D>

Next, you will start the 3COM linking program.

Type: 3COM XXX (include the space) (where XXX is the contractor i.d.) <CR> Get (after pauses - you will hear the phone ring and data transmission):

Login 1.1 - Copyright (etc.) 3COM
XXX:PRI:IRDMS logged in
D> 3f link e:
E: Linked to \\XXX:PRI:IRDMS
D>

(NOTE: The Remote Login Disk may now be removed from the A: drive)

You are now linked to the E: drive on the PRI LAN, which can be treated as any other drive. For example, you can switch to the E: drive by typing E: <CR>. After a pause (sometimes several seconds), you should get the E> prompt.

Under E: is a subdirectory \TRANSFER where all files are sent (*.TRN) to be transferred. To copy a file SEMAP.TRN from drive D and directory DATA to the network,

Type E: <CR>
Get E>
Type CD\TRANSFER <CR>

Get E> (The subdirectories do not show on the prompt)
Type copy D:\DATA\SEMAP.TRN <CR>
(NOTE: any valid DOS COPY format can be used)

After a while, you may get a message saying that the network is still trying, and allows you to do an abort. Eventually, you should get a file(s) copied message:

Get 1 file(s) copied

To verify that the file was transmitted, you can print the directory listings.
If you are not on the E> (actually E\TRANSFER) prompt, type E: <CR>, then

CD\TRANSFER <CR>, then

Type DIR <CR>
Get (a listing of the \TRANSFER directory contents. SEMAP.TRN should be in the listing.)

To log out after all files have been transmitted:

Type D; <CR>
Get D>
Type LOGOUT <CR>
Get a "logged out" message

You can now (a) do other things with the IRDMS programs, or (b) reboot the machine with the Remote Login Disk removed, to return the PC to local control.

AFTER EVERY TRANSFER OF DATA TO THE PRI LAN, call PRI at (410) 679-3030 (ask for the contact for the specific installation) and give them the installation and file type (e.g., SL map file data) and the DOS name of the file that was transmitted. The contractor should receive by Fax a confirmation that the data was received; if not, call PRI and ask for it.

# STANDARD OPERATING PROCEDURE 007
# SURFACE WATER SAMPLING

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for sampling surface water. This procedure can be applied to the collection of surface water samples from streams, rivers, ditches, lakes, ponds and lagoons. Surface water samples provide an indication of the amount of contaminant in the surface water. It is therefore important to collect a representative sample.

## 2.0 Material

a. Sample bottles
b. Stainless steel or PTFE-lined bucket
c. Long-handled dip sampler (PTFE or stainless steel)
d. Short-handled dip sampler (PTFE or stainless steel)
e. Peristaltic pump with 0.45$\mu$M filters and disposable tygon tubing
f. 0.45$\mu$ disposable filters
g. Cooler with ice

## 3.0 Procedure

**3.1** For all surface water samples, mark the sampling locations on a site map. Photograph and describe each location, and place a numbered stake above the visible high water mark on the bank closest to the sampling location, and/or mark adjacent trees with surveyor's flagging. The photographs and descriptions must be adequate to allow the sampling station to be relocated at some future date by someone other than the original sampling crew. Use the long handled dip sampler where access is poor or non-contact with water is suggested in the health and safety plan.

**3.2** Sampling should performed deliberately and methodically to minimize disturbance of bottom sediments, yet as quickly as possible to ensure a representative sample. To prevent contamination of the exterior of the sample container, and/or potential contamination of the surface water sample by laboratory contaminants on the exterior of the bottle, the sample container should never be dipped into the water, rather a decontaminated, long-handled or measuring cup-type PTFE or stainless steel sampler, or a sampling bucket should be used to collect unfiltered samples.

**3.3 Sampling with the PTFE or stainless steel sampler** (long-handled or measuring cup-type):

**3.3.1** Remove the cap from the sample bottle.

3.3.2 Dip a sample of surface water using the sampler.

3.3.3 Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.

3.3.4 Add preservative as required by SOP039. Replace cap, and place in cooler immediately.

3.4 **Sampling with stainless steel or PTFE-lined bucket:**

3.4.1 Remove cap from sample bottle.

3.4.2 Gently dip collection bucket in the water. Fill bucket and carefully lift from water body.

3.4.3 Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.

3.4.4 Add preservative as required by SOP039. Replace cap, and place in cooler immediately.

**-- OR --**

3.4.5 Use smaller sampling cup to transfer sample from bucket to sample bottle as in section 3.3 above.

3.5 Both filtered and unfiltered samples will be taken for metals analyses. Bulk samples for filtration will be collected using the stainless steel or PTFE-lined bucket method described in section 3.4 above. Sample filtration must be performed immediately upon retrieval of the bulk sample as follows:

3.6 Filtration will be performed immediately after collecting sample. Set up filtration equipment prior to collecting sample. Filtration may be accomplished by gravity (see 3.7), or if necessary, due to slow filtering, a peristaltic pump will be used to pressure filter the sample (see 3.8). Vacuum filtration will not be used due to the possibility of analyte volatilization.

3.7 **Gravity filtration will be accomplished as follows:**

3.7.1 Using decontaminated forceps, place a 0.45$\mu$M membrane in a decontaminated filter funnel.

3.7.2 Slowly pour sample into the funnel and collect filtrate directly into appropriate sample container(s).

3.7.3 Add preservative(s) as required by SOP039. Immediately cap container and place in cooler.

3.7.3 Dispose of filter membrane.

**3.8 Pressure filtration will be accomplished as follows:**

3.8.1 Using previously assembled disposable tubing, 45$\mu$ in-line filter, and peristaltic pump, filter sample from collection bucket into appropriate container.

3.8.2 Adjust pump rate to avoid aeration of sample.

3.8.3 Fill container, preserve as indicated in SOP0039, immediately cap container and place in cooler.

3.8.4 Dispose of filter and tubing.

3.9 Refer to SOP 1-5, 16, and 39.

## 4.0 Maintenance

Refer to manufacturer's specifications for maintenance procedures on generators and pumps.

## 5.0 Precautions

5.1 Avoid disturbing bottom sediments.

5.2 Consult the health and safety plan (HASP) prior to collecting any samples for PPE such as dermal and respiratory protection and personal flotation devices when sampling in or near deep water or from boats.

5.3 Always decontaminate the sampling and filtration equipment, and change gloves between sampling locations to minimize the risk of cross contamination.

5.4 Always set up generators downwind of working area. Never service generators onsite.

## 6.0 References

None.

# STANDARD OPERATING PROCEDURE 008
# pH MEASUREMENT USING BECKMAN Φ™ 12 pH/ISE METER

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for measuring the pH of all types of aqueous solutions, including drinking water, saline water, industrial and domestic wastes. pH is a measure of the hydrogen ion content of a solution, and thus gives a general indication of the acidity or alkalinity of a water sample.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. Φ 12 pH Meter
b. Combination (pH) electrode
c. Automatic temperature compensator (ATC) probe
d. Commercial Buffer solutions (standards) of pH 4.00, 7.00, and 10.00
e. HPLC-grade water
f. Beakers
g. Wash bottle
h. Laboratory wipes for blotting electrodes

## 3.0 Procedure

**3.1 Calibration check.** Calibration of the pH meter will be checked on a daily basis. A two point calibration should be used as follows:

**3.1.1** Prepare beakers of buffer solutions of pH 4.00, 7.00, and 10.00.

**3.1.2** Calibration should closely bracket the expected pH range of the samples to be taken.

**3.1.3** Turn on instrument, clear instrument

**3.1.4** Rinse the electrode with distilled water and blot excess.

**3.1.5** Immerse probes in beaker of pH 4.00 or 7.00 standard, swirl gently.

**3.1.6** Press pH key, then STD key.

**3.1.7** Keep the probes in the sample until the reading stabilizes. The reading should be the pH of the standard.

**3.1.8** Rinse the electrode with distilled water and blot excess.

**3.1.9** Repeat the procedure with pH 7.00 and 10.00 standards

**3.1.10** Record the initial readings.

**3.1.11** If the measured values vary from the expected value by greater than 0.2 pH units, recalibrate the instrument with fresh aliquots of buffer solution. If the discrepancy persists, alert the Field Operations Leader, who has the option of trying to fix the meter or obtaining a second pH meter.

**3.1.12** Record all measurements in the field logbook.

**3.1.13** Verify calibration by reading the pH of the third buffer solution.

**3.1.14** Refer to SOP 003 and 016.

**3.2** **pH measurements** will be taken using the two-point standardization method as follows:

**3.2.1** Connect the ATC and pH electrodes to the appropriate inputs.

**3.2.2** Turn on instrument, clear instrument.

**3.2.3** Prepare two small beakers of standard buffer solutions. Ideally the pH values of these standards will "bracket" the expected pH value of the sample and be as close as possible to the pH of the sample.

**3.2.4** Rinse a small beaker with distilled water, then sample water. Fill the beaker with sample water.

**3.2.5** Rinse the probes with distilled water. Blot excess.

**3.2.6** Immerse electrode and ATC in first standard. Swirl gently. Press pH key and STD key. Wait for display to stop flashing.

**3.2.7** Rinse the probes with distilled water. Blot excess.

**3.2.8** Immerse electrode and ATC in second standard. Swirl gently. Press STD key. Wait for display to stop flashing.

**3.2.9** Rinse the probes with distilled water. Blot excess.

**3.2.10** Immerse the probes in the sample and swirl gently, keeping the probes in the sample until the display stops flashing.

**3.2.11** Record the sample pH and temperature after stabilization. Note any problems such as meter drift.

**3.2.12** Rinse the probes with distilled water. Blot excess.

**3.2.13** Repeat steps 3.2.9 through 3.2.12 for additional samples.

**3.3** **Decontaminate probe** according to manufacturer's specifications.

**3.4** **Decontaminate beakers** according to SOP005, section 3.3.1.4 "labware".

## 4.0 Maintenance

The following steps will be taken to maintain the pH meter.

**4.1** Check the batteries each time the meter is used.

**4.2** Keep the probe stored in a 0.1 M KCl solution adjusted to pH 4 when the meter is not in use. Alternatively, the electrode may be rinsed with deionized water and the protective cap put on, trapping any residual water inside it (do not blot the electrode dry prior to putting the cap on).

## 5.0 Precautions

**5.1** Remove coatings of oil material or particulate matter that can impair electrode response by gentle wiping or detergent washing, followed by distilled water rinsing.

**5.2** As noted in Section 1 (above), these procedures may not apply to alternate manufacturers' equipment.

**5.3** Calibration is always performed using the pH 7.00 and one end point (pH4.00 or 11.00) standard never calibrate the instrument using two end points only.

## 6.0 References

Beckman Instruments, Inc., Users Manual for Φ™ 10 pH Meter, Φ™ 11 pH meter, and Φ™ 12 pH/ISE Meter

US EPA. 1983. Methods for Chemical Analysis of Water and Wastes, March, 1983.

Franston, Mary Ann H., et al. (eds), Standard Methods for the Examination of Water and Wastewater, 15th Edition, American Public Health Assn., American Water Works Assn., and Water Pollution Control Federation, 1981

# STANDARD OPERATING PROCEDURE 009
# TEMPERATURE MEASUREMENTS

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for measuring the temperature of a solid or liquid sample, in particular, for measuring water temperature. Groundwater temperature does not vary dramatically over the course of a year. Therefore, groundwater temperature can be used to help identify an aquifer, identify stream reaches where groundwater inflow is occurring, identify thermal gradients in lakes or ponds, and also indicate when sufficient water has been removed from a well during purging.

## 2.0 Material

Digital reading, thermocouple thermometer in combination meter or in a stick. Accuracy= +/- 0.5°C

## 3.0 Procedure

**3.1** Rinse the probe with distilled water.

**3.2** Insert the probe into the sample, and leave it in the sample until the temperature stabilizes.

**3.3** Record the temperature reading, being sure to indicate °C or °F.

**3.4** Decontaminate the probe according to SOP005 "Decontamination" section 3.3.1.4 "Labware."

**3.5** Refer to SOP 003, 005, and 016.

NOTE: °C = (°F - 32) (5/9)
°F = ((9/5)C°) + 32

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

None

## 6.0 References

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, March, 1983.

# STANDARD OPERATING PROCEDURE 010
# WATER LEVEL AND WELL-DEPTH MEASUREMENTS

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## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for measuring water level and well-depth. This procedure is applicable to the sampling of monitoring wells and must be performed prior to any activities which may disturb the water level, such as purging or aquifer testing.

## 2.0 Material

a. Electric Water Level Indicator (dipmeter) with cable measured at 0.01 foot increments.

**-or-**

Weighted Steel Tape and chalk.

**-or-**

Transducer and datalogger.

b. Oil-water interface probe
c. Plastic Sheeting
d. Photoionization detector (PID) or intrinsically safe flame ionization detector (FID)

## 3.0 Procedure

### 3.1 Preliminary Steps

**3.1.1** Locate the well and verify its position on the site map. Record whether positive identification was obtained, including the well number and any identifying marks or codes contained on the well casing or protective casing. Gain access to the top of the well casing.

**3.1.2** Locate the permanent reference mark at the top of the casing. This reference point will be scribed, notched or otherwise noted on the top of the casing. If no such marks are present, measure to the top of the highest point of the well casing and so note this fact in field logbook. Determine from the records and record in the notebook the elevation of this point.

**3.1.3** Record any observations and remarks regarding the completion characteristics and well condition, such as evidence of cracked casing or surface seals, security of the well (locked cap), and evidence of tampering.

3.1.4 Keep all equipment and supplies protected from gross contamination; use clean plastic sheeting. Keep the water level indicator probe in its protective case when not in use.

## 3.2 Operation

3.2.1 Sample the air in the well head for gross organic vapors by lifting the well cap only high enough for an organic vapor meter (PID or FID) probe to be entered into the well casing. This will indicate the presence of gross volatile contaminants as well as indicating potential sampler exposure.
Remove cap. Allow well to vent for 60 to 90 seconds. Resample headspace. Record both readings. If the second reading is lower than the first, use the second reading to determining whether respiratory protection will be required during subsequent water level and well depth determinations, and sampling. Note that all headspace sampling must be performed at arm's length and from the upwind side of the well if possible.

3.2.1.1 Refer to SOP 011, 023, or 024 as appropriate.

3.2.2 If non-aqueous phase liquid (NAPL) contamination is suspected [6], use an interface probe to determine the existence and thickness of NAPLs.

3.2.2.1 Open the probe housing, turn the probe on, and test the alarm. Slowly lower the probe into the well until the alarm sounds. A continuous alarm indicates a NAPL while an intermittent alarm indicates water. If a NAPL is detected, record the initial level (first alarm). Mark the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable sufficiently to record the depth.

3.2.2.2 Continue to slowly lower the probe until it passes into the water phase. Slowly retract the probe until the NAPL alarm sounds and record that level in the manner as described above.

3.2.2.3 Record the thickness of the LNAPL (see section 3.3.1).

3.2.2.4 Continue to slowly lower the interface probe through the water column to check for the presence of DNAPL.

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[6] Interface probes will be used in all wells for first round sampling, regardless of site history. If no NAPLs are detected during the first round of sampling, this step may be omitted during subsequent sampling events unless conditions such as site history or headspace vapors would indicate otherwise.

**3.2.2.5** Measure and record the thickness of the DNAPL layer (if any) as described above.

**3.2.2.6** Slowly raise the interface probe, recording the depth to each interface as the probe is withdrawn. If there is a discrepancy in depths, clean the probe sensors and re-check the depths.

**3.2.2.7** NOTE: Air/liquid interface depth is more reliable if probe is lowered into liquid. NAPL/water drepths are more accurate if probe is moved from water into NAPL.

**3.2.2.8** Always lower and raise interface probe slowly to prevent undue mixing of media.

**3.2.2.9** Always perform NAPL check in wells installed in areas with suspected NAPL contamination. Always perform NAPL check if headspace test reveals presence of volatiles. Always perform NAPL check the first time a well is sampled. **If** a well has been sampled previously **and** no NAPLs were present **and** none of the preceeding conditions are met, the NAPL check may be omitted.

**3.2.3** If no NAPL is present use an electronic water level detector as follows.

**3.2.3.1** Remove the water level indicator probe from the case, turn on the sounder, and test check the battery and sensitivity scale by pushing the red button. Adjust the sensitivity scale until you can hear the buzzer.

**3.2.3.2** Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the meter buzzes. Very slowly, raise and lower the probe until the point is reached where the meter <u>just</u> buzzes. Marking the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable and record the depth.

**3.2.4** Alternatively use a steel tape with an attached weight if aquifer gradients are lower than 0.05 ft/ft. Due to the possibility of adding unknown contaminants from chalk colorants, only white chalk is permitted.

**3.2.4.1** Rub chalk onto the first 1 foot of the steel tape and slowly lower the chalked end into the well until the weighted end is below the water surface. (A small splash can be heard when the weighted end hits the water surface.)

**3.2.4.2** Using the method described above read and record the length from the steel tape.

**3.2.4.3** Remove the steel tape. The chalk will be wet or absent where the tape was below the water surface. Locate, read, and record this length. Subtract wetted length from total length and record the difference. This is the depth to water table.

**3.2.5** Transducers and dataloggers will be used where water level fluctuations over time are to be measured, such as tidal fluctuation studies (SOP043) and slug tests (SOP033). Note that transducers are inappropriate for measuring well depth.

**3.2.5.1** Slowly lower the transducer into the well until it is below the lowest possible piezometric level (typically 2-3 ft below the water table).

**3.2.5.2** Tape the umbilical to the protective casing to prevent the transducer from falling further.

**3.2.5.3** Attach the umbilical leads to the datalogger.

**3.2.5.4** Turn datalogger on.

**3.2.6** To measure the well depth, lower electric water level indicator probe or tape until slack is noted. Very slowly raise and lower the cable until the exact bottom of the well is "felt." Measure (cable) or read the length :(tape) and record the depth. Note that if the electric water level indicator is used to determine depth of well, the offset distance between the tip of the probe and the electrode must be added to the reading to determine actual depth.

**3.2.7** Withdraw the probe or tape.

**3.2.8** Decontaminate the probe(s) and cable(s).

## 3.3 Data Recording and Manipulation

**3.3.1** Record the following computations:

date and time
weather
method of measurement
casing elevation
NAPL surface elevation = casing elevation - depth to NAPL
NAPL thickness = depth to bottom of NAPL - depth to top of NAPL
water level elevation = casing elevation - depth to water

well bottom elevation = casing elevation - depth to bottom (or read directly from tape)

3.4 Refer to SOPs 3, 5, and 16.

**4.0 Calibration**

No calibration is needed.

**5.0 Precautions**

5.1 Depending upon the device used, correction factors may be required for some measurements.

5.2 Check instrument batteries prior to each use.

5.3 Exercise care not to break the seals at the top of the electric water level indicator probe.

**6.0 References**

McAlary, T. A., and Barker, J. F., 1987. "Volitalization Losses of Organics During Ground Water Sampling from Low Permeability Materials" in Ground Water Monitoring Review, Fall,1987

Thornhill, Jerry T., 1989. Accuracy of Depth to Groundwater Measurements; In "EPA Superfund Ground Water Issue" EPA/540/4-89/002

# STANDARD OPERATING PROCEDURE 011
# PHOTOIONIZATION DETECTOR (HNu Model PI-101 and HW-101)

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for field operations with a photoionization detector (HNu Systems Model PI-101 or HW-101). The photoionization detector (PID) detects total ionizables, hence it is used to monitor both organic and inorganic vapors and gases to determine relative concentrations of air contaminants. This information is used to establish level of protection and other control measures such as action levels. The PID cannot effectively detect compounds having ionization potentials above the photon energy level of the lamp used; therefore, methane, which has an ionization potential of 12.98 eV, is undetectable by PIDs, whose lamps are capable of producing 9.5, 10.2, or 11.7 eV.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. HNu Systems Model PI-101 or HW-101 survey probe with 9.5, 10.2, or 11.7 eV lamp.
b. Lead-acid gel-cell battery
c. Calibration Gas (*e.g.* isobutylene - 101 ppm) w/regulator
d. Tygon tubing
e. Tedlar bag (optional)
f. Instrument logbook

## 3.0 Procedure

These procedures are to be followed when using the HNu in the field.

### 3.1 Start Up Procedures

**3.1.1** Before attaching the probe, check the function switch on the control panel to ensure that it is in the off position. Attach the probe by plugging it into the interface on the top of the readout module.

**3.1.2** Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scale. If not, recharge the battery. If the red indicator light comes on, the battery needs recharging or service may be indicated.

**3.1.3** Turn the function switch to any range setting. Listen for the hum of the fan motor. Check meter function by holding a solvent based marker pen near the sample intake. If there is no needle deflection, look briefly into the end of the

probe (no more than one or two seconds) to see if the lamp is on. If it is on, it will give a purple glow. Do not stare into the probe any longer than two seconds. Long term exposure to UV light can damage the eyes. (See also note 5.4)

**3.1.4** To ZERO the instrument, turn the function switch to the standby position and rotate the zero adjustment until the meter reads zero. A calibration gas is not needed since this is an electronic zero adjustment. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Allow the instrument to warm up for 3-5 minutes to ensure that the zero reading is stable. If necessary, readjust the zero.

**3.2 Operational Check**

**3.2.1** Follow the start up procedure in section 3.1

**3.2.2** With the instrument set on the 0-20 range, hold a solvent-based magic marker near the probe tip. If the meter deflects upscale, the instrument is working.

**3.3 Field Calibration Procedure**

**3.3.1** Follow the start-up procedures in section 3.1 and the operational check in section 3.2.

**3.3.2** Set the function switch to the range setting for the concentration of the calibration gas.

**3.3.3** Attach a regulator HNu P/N 101-351 or equivalent (flow = 200 to 300 ml/min) to a disposable cylinder of isobutylene (HNu 101-351 or equivalent). Connect the regulator to the probe of the HNu with a piece of clean Tygon tubing. Turn on the valve of the regulator.

**3.3.4** After five seconds, adjust the span dial until the meter reading equals the benzene concentration of the calibration gas used, corrected to its equivalence which should be marked on the canister (Isobutylene ~0.7X benzene).

**3.3.5** Record in the field log: the instrument ID No.; serial number; the initial and final span settings; the date; time; location; concentration and type of calibration gas used; and the signature of the person who calibrated the instrument.

**3.3.6** If the HNu does not function, check-out, or calibrate properly, the project equipment manager is to be notified as soon as possible. Under no circumstances is work requiring monitoring with a PI-101 or HW-101 to be done with a malfunctioning instrument.

### 3.4 Calibration to a Gas Other Than Isobutylene

The HNu may be calibrated to any certified calibration gas. However, after calibration all subsequent instrument readings will be relative to the calibration gas used.

**3.4.1** Calibrate according to procedure 3.3

**3.4.2** Partially fill and flush to two times a gas bag (Tedlar recommended) with the certified National Institute of Standards and Technology (NIST) (formerly NBS) traceable calibration gas. Then fill the bag with one to three liters of the calibration gas. If the gas is toxic, this must be done in a fume hood.

**3.4.3** Feed the calibration gas into the probe with the range set for the value of the gas. After five seconds, adjust the span control until the meter reads the value of the calibration gas.

**3.4.4** Record the results of the calibration on the calibration/maintenance log and attach a new calibration sticker (if available) or correct the existing sticker to reflect the new calibration data. All subsequent readings will be relative to the new calibration gas.

### 3.5 Operation

**3.5.1** Follow the start up procedure, operational check and calibration check (refer to 3.1).

**3.5.2** Set the function switch to the appropriate range. If the concentration of gases of vapors is unknown, set the function switch to 0-20 ppm range. Adjust if necessary.

**3.5.3** While taking care not to permit the HNu to be exposed to excessive moisture, dirt, or contaminants, monitor the work activity as specified in the Site Health and Safety Plan.

**3.5.4** When the activity is completed or at the end of the day, carefully clean the outside of the HNu with a damp disposable towel to remove all visible dirt. Return the HNu to a secure area and place on charge. Place the instrument on charge after each use; the lead acid batteries cannot be ruined by over charging.

**3.5.5** With the exception of the probe's inlet and exhaust, the HNu can be wrapped in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation. If the instrument becomes contaminated, make sure to take necessary steps to decontaminate it. Call the

Equipment Administrator if necessary; under no circumstances should and instrument be returned from the field in a contaminated condition.

**3.6** Refer to SOP 3 and 16.

## 4.0 Maintenance

The following sections cover basic maintenance and servicing procedures; they are to be performed only by persons who have been specifically trained in the procedures. In general, disassembly procedures not covered in this text are to be left to trained service personnel, including the contractor's equipment administrator or equipment managers as appropriate.

The appropriate calibration/maintenance logs are to be filled in completely whenever a PI-101 or HW-101 receives servicing. This is true of both contractor-owned and rental instruments.

The equipment manager should be called to arrange for a fresh instrument when necessary. The contractor's equipment facility is responsible for arranging all repairs which cannot be performed by the project equipment manager.

### 4.1 Routine Service

The PID's performance is affected by a number of factors. These include but are not limited to the decay of the UV lamp output over time and the accumulation of dust and other particulate material and contaminates on the lamp and in the ion chamber. Because of these factors, the PID should not be left in the field for a period of more than two weeks before being replaced with a fresh instrument. If a site is going to be inactive for a period of more than a week, all monitoring instruments are to be returned to the project equipment manager or his trained designee for servicing and/or reassignment. The following procedures are to be performed at the designated intervals for routine service.

| Procedure | Frequency |
|---|---|
| Operational Check | Prior to use and at instrument return |
| Field Calibration | Prior to use and at instrument return |
| Full Calibration | Bi-weekly (return instrument to equipment manager for replacement with a fresh unit) |
| Clean UV Lamp and Ion Chamber | Bi-weekly or as needed |
| Replace UV Lamp | As needed |

**4.1.1** UV Lamp and Ion Chamber Cleaning

During periods of analyzer operation, dust and other foreign materials are drawn into the probe forming deposits on the surface of the UV lamp and in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, drifting, and which show apparent moisture sensitivity. These deposits interfere with the ionization process and cause erroneous readings. Check for this condition regularly to insure that the HNu is functioning properly. If the instrument is malfunctioning, call your respective equipment manager to arrange to have a fresh replacement.

4.1.2 Lamp eV Change

If different applications for the analyzer would require different eV lamps, separate probes, each with its own eV lamp, must be used. A single readout assembly will serve for any of the probes (9.5, 10.2 and 11.7 eV). A change in probe will require resetting of the zero control and recalibrating the instrument. The 11.7 eV lamp will detect more compounds than either of the two lower eV lamps. However, the 11.7 eV probe needs more frequent calibration, it burns out much faster than the lower eV lamps.

## 5.0 Precautions

**5.1** The HNu PI-101 and HW-101 are designed to sample air or vapors only. DO NOT allow any liquids or low boiling vapors to get into the probe or meter assembly.

**5.2** High concentrations of any gas can cause erroneous readings. High humidity can also cause the instrument readings to vary significantly from the actual concentration of gases or vapors present. This is true even through the HNu cannot react to water vapor.

**5.3** High humidity, dust, and exposure to concentrations of low boiling vapors will contaminate the ion chamber, causing a steady decrease in sensitivity.

**5.4** Continued exposure to ultraviolet light generated by the light source can be harmful to eyesight. If a visual check of the UV lamp is performed: Do not look at the light source from a distance closer than 6 inches with unprotected eyes. Use eye protection (UV-blocking sunglasses or safety glasses). Only look briefly - never more than about 2 seconds.

**5.5** Place the instrument on charge after each use; the lead batteries cannot be ruined by over charging.

**5.6** If at any time the instrument does not check out or calibrate properly in the field, the equipment manager is to be notified immediately and a replacement provided for the malfunctioning instrument. Under no circumstances should field work requiring continuous air monitoring for organic vapors and/or gases be done with a malfunctioning HNu, without a HNu or an approved comparable instrument.

## 6.0 References

Manufacturer's Equipment Manual(s).

# STANDARD OPERATING PROCEDURE 012
# SPECIFIC CONDUCTANCE MEASUREMENTS

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for measuring the specific conductance of any aqueous solution, including drinking water, saline water, industrial and domestic wastes. Conductivity is the ability of an aqueous solution to pass an electrical current. The current is primarily carried by dissolved inorganic ions such as chlorides, nitrates, sulfates, along with cations such as sodium, calcium, magnesium and others. Organic compounds do not carry current and therefore have almost no conductivity.

## 2.0 Material

a. Conductivity meter with integral temperature compensation - Accuracy = $\pm$ 2% at 25°C (77°F)
b. Conductivity cell
c. Appropriate conductivity reference solution
d. HPLC-grade water (see SOP005 footnote 2)
e. Thermometer (optional, see 5.2)

## 3.0 Procedure

### 3.1 Calibration

The specific conductivity meter should be calibrated at the beginning of each day[7] as follows:

**3.1.1** Thoroughly rinse the probe with Appropriate conductivity reference solution

**3.1.2** Zero meter if appropriate.

**3.1.3** Measure the specific conductance of fresh Appropriate conductivity reference solution record it in the field notebook, and adjust the calibration knob until the meter reads properly.

**3.1.4** Rinse probe with HPLC-grade water.

**3.1.5** Measure the specific conductance of HPLC-grade water and record in the field logbook. If Specific conductivity of HPLC-grade water is not 0 ($\pm$2%) recalibrate instrument.

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[7] The meter should be recalibrated any time the readings are suspect (*e.g.* out of expected range)

**3.2 Operation**

The specific conductivity meter will be operated as follows:

**3.2.1** Thoroughly rinse the probe and sample beaker with sample water.

**3.2.2** Measure the temperature of the sample water. Convert Fahrenheit temperature readings to Celsius using C = 5/9 (F - 32) if Celsius temperature is not obtained directly.

**3.2.3** Place the probe in the sample beaker with sufficient sample to completely submerge the probe. Swirl the probe to remove any air bubbles trapped in the probe.

**3.2.4** Select the highest multiplier scale on the meter and turn the instrument on. Progressively use lower multiplier scales until a mid-scale deflection is obtained.

**3.2.5** If appropriate, check probe accuracy by pressing cell test button. If value change is > 10% check probe.

**3.2.6** Record the temperature and conductivity values.

**3.2.7** Specific conductivity values are corrected for temperature using:

$$K_{25°C} = \frac{K\ measured}{1 + 0.0191\ (t-25)}$$

where:

K = conductivity in $\mu$mhos
t = temperature, °C

**3.2.8** Decontaminate the probe. (See SOP005 § 3.3.3.2)

**3.3** Refer to SOPs 003, 005, and 016.

**4.0 Maintenance**

The following steps will be taken to properly maintain the conductivity meter:

**4.1** Check the batteries each time the instrument is used.

4.2 Inspect the probe on a daily basis for damage or loss of platinum black plating from the electrode. If the platinum is damaged, alert the Field Team Leader and arrange to get a new cell.

4.3 Follow manufacturers specifications regarding storage of probe between uses.

**5.0 Precautions**

5.1 Be certain there is no air in the cell before taking a reading.

5.2 If conductivity meter does not have integral temperature compensation, use a thermometer to determine temperature of the sample.

**6.0 References**

USEPA, 1983. Methods for Chemical Analysis of Water and Wastes, March, 1983.

Manufacturer's Manual

# STANDARD OPERATING PROCEDURE 013
# COLLECTION OF MONITORING WELL SAMPLES

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for the collection of groundwater samples from monitoring wells.

## 2.0 Material

a. Conductivity meter
b. Thermometer (optional)[8]
c. pH meter with ORD probe
d. Turbidity meter
e. Dissolved Oxygen meter
f. Water-level indicator
g. Transparent bailer with a double check valve
h. PVC bailer (for purging only)
i. Stainless steel bailer (for purging and sampling)
j. Polytetrafluoroethelyne (PTFE) bailer with PTFE-coated stainless steel cable, double check valve top and controlled flow bottom discharge attachment[9] for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles) (for purging and sampling)
k. Polypropylene rope
l. Submersible pump and hose (for purging only)
m. Peristaltic pump with tubing for filtering samples
n. Variable speed, low flow submersible pump (*e.g.* Grundfos MP1 ground-water sampling pump) (for purging and sampling)
o. Bladder pump (dedicated to one well only)
p. 0.45$\mu$M filters
q. Sample bottles and labels
r. Logbook or book of field parameter forms
s. Generator
t. Tygon tubing
u. Plastic sheeting
v. Photoionization Detector (PID) Organic Vapor Analyzer

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[8] Temperature compensation and measurement capabilities are generally available as integral functions of pH meters and conductivity meters. If this is the case, a separate thermometer is not required.

[9] Although use of a controlled flow bottom discharge valve is historically preferred, use of such a device can cause aeration of the sample.

## 3.0 Procedure

**3.1 General:** Ground-water sampling will follow these general steps:

- Arrive on site
- Set up apparatus (generators, pumps, etc.)
- Glove
- Perform all steps of SOP 010 - organic vapor check, water level and well depth measurements
- Sample NAPLs (as required)
- Begin purge procedure
  - If using bailer to purge and sample see § 3.6.
  - If using pump to purge and bailer to sample see § 3.7.
  - If using bladder or low-flow pump to purge and sample see § 3.8.
- Decon/reglove
- Take samples
  - If with bailer see § 3.6.5
  - If with bladder or low flow pumps see §3.8
- Decon/dispose of wastes, move equipment to next site.

**3.2 General Rules for Groundwater Field Parameter Logbook** (see SOP 016 for further procedures):

**3.2.1** Only one site or installation per logbook, and only one sampling location per page or form (if using pre-printed forms). The same book maybe used for more than one sampling event.

**3.2.2** First five pages will be reserved for index, general notes, etc. Sign and date each entry.

**3.2.3** Last five pages will be reserved for recording calibration data for the pH, temperature, turbidity, ORD, DO, and conductivity meters. Use the page number or a separately recorded "Cal Reference Number" to refer to each calibration.

**3.2.4** (As appropriate). Insert the cardboard flap under the form being filled out, so that writing does not go through to the pages below.

**3.2.5** (As appropriate). Fill in the forms from front to back of the logbook, tearing out the white copy for each sample when the sample has been collected. This copy goes in the cooler with the sample, directly to the laboratory. The original copy must be torn out before you write on the back of the duplicate form.

**3.2.6** (As appropriate). Duplicate copies, index pages, and calibration sheets remain intact.

## 3.3 Groundwater Sampling General Rules

**3.3.1** Refer to SOPs 001-005, 008-012, 036, 037, and 039

**3.3.2** Groundwater samples will be collected from the least contaminated wells first, progressing to the most contaminated [10].

**3.3.3** Upon arrival at the well site, immediately set up and organize the purging, sampling, and filtration equipment. If needed, due to muddy or contaminated ground, remoteness from sampling vehicle, and\or for placement of hose(s) and\or power cord if a pump is used, place clean plastic sheeting at, or around the well, to serve as a clean staging area for purging and sampling equipment, as conditions warrant. Care must be exercised not to step on plastic sheeting.

**3.3.4** If the well is remote from the sampling vehicle set up the filtration equipment and place rope, wrapped bailer, and pre-labeled sample containers on the plastic sheet, uphill from the well.

**3.3.5** When a pump is to be used situate the portable generator on level ground approximately 15 feet away from and downwind from the well. All generator maintenance (oil and fueling) is to be performed off site. If the hose(s) and/or power cord of the pump are not on a reel, place the pump with its hose and power cord on the plastic sheeting downhill from the well.

**3.3.6** Glove. Check well headspace for organic vapor which may pose a health and safety hazard and indicate the presence of NAPL. Measure depth(s) to and thickness(es) of NAPL(s) as appropriate. Measure the depth to water and depth of well. From the water depth, well diameter, sand pack length, etc., calculate the equivalent volume (1 EV) of water in the well.

1 EV = volume in casing + volume in saturated sand pack. Therefore; if the **water table lies below the top of the sandpack**, use the following equation:

$$1\ EV = (\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_w) * (0.0043)$$

If the **water table lies above the top of the sandpack** use this equation:

$$1\ EV = [(\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_s)] * (0.0043)$$

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[10] First round samples are to be collected from upgradient wells first, moving to downgradient wells under the assumption that upgradient wells will be less contaminated than downgradient wells. Results of first round analysis may mandate a change in sampling sequence.

where: $R_s$ = radius of sandpack in inches
$R_w$ = radius of well casing in inches
$h_s$ = height of sandpack in inches
$h_w$ = water depth in inches

0.0043 gal/in$^3$
Assumed filter pack porosity = 30%

Tables and graphs showing equivalent volumes for typical well constructions are available.

Alternate equations for calculating EV are acceptable, two alternates are given in SOP 010

**3.3.7** Samples will always be collected in order of decreasing volatility (*i.e.*, the samples to be analyzed for the volatile constituents should be collected first.) Deliver the VOC sample to the vial by allowing the water to trickle down the inside wall of the vial at a rate no greater than approximately 100 ml/min. Other samples may be delivered at a faster rate. Sampling rates will at no time exceed 1 L/min. Procedures for each class of samples are contained in Appendix A of the GWP, the QAPP, and SOP 039.

**3.3.8** When collecting samples for volatile analysis care should be taken to prevent analyte loss by volatilization. The following procedures should be adhered to when collecting these samples [11]:

**3.3.8.1** Avoid excessive aeration and agitation of sample.

**3.3.8.2** Fill vial so that a reverse meniscus is present by adjusting the flow rate from the sampling device.

**3.3.8.3** Place septum on vial so that the PTFE side is in contact with the sample. After the cap is on the bottle, check for air bubbles in the sample. If air bubbles are present, properly dispose of that sample and recollect the sample in the same vial.

**3.3.8.4** Make sure vial is labeled and immediately transfer the vial to the cooler with ice.

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[11] Although EPA Region III policy is to preserve VOA samples by acidifying to pH < 2, the possibility of generating mustard agent by reverse hydrolyzation of thyodiglycol mandates that at the Edgewood Area, APG this not be done. Instead, the holding time on all VOA samples **shall** not exceed 7 days.

**3.3.9** Filtered and unfiltered samples will be taken for inorganics (metals) analyses. The samples will be filtered through an in-line 0.45$\mu$M filter (preferred method), or by gravity through a 0.45$\mu$M membrane placed in a filter funnel. Use forceps to place the membrane into the funnel and pour sample through funnel until appropriate volumes have been filtered.

If necessary, due to slow filtering, a peristaltic pump may be used to filter the sample through an in-line filter. Connect the pump to the generator, attach tygon tubing to the bottom discharge valve on the bailer. Start pump and collect sample from the end of the in-line filter directly into the proper container, preserved (as required by SOP 039), and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

**-- OR --**

If a low flow pump is used collect the samples, filtered samples will be taken by installing a 0.45$\mu$M filter in-line and pumping the water through the filter. Collect sample from the end of the in-line filter directly into the proper container, preserved (as required by SOP 039), and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

**3.3.10** Unfiltered samples will be collected by slowly pouring the sample water into the appropriate sample container, being careful not to agitate or cause bubbles to form. Do not overfill bottles. Make sure sample bottle is labeled and the cap is on tightly. Then place the sample in cooler with ice immediately.

**3.3.11** All samples will be delivered to the laboratory as soon as possible. If possible, samples will be shipped on the same day as they are collected. If samples must be retained due to weekend sampling (Friday through Sunday), the lab shall be notified as to the time sensitive nature of the samples.

**3.3.12** Refer to SOP 1-5, 16, 31, and 39 .

**3.4 Sampling of Non-Aqueous Phase Liquids**

**3.4.1** If NAPLs are detected in the well, a sample from all layers must be collected prior to any purging activities. Non-aqueous phase liquids (NAPLs) may be indicated by the presence of volatiles in the well headspace, and confirmed by the oil/water interface probe (see SOP 10 § 3.2 - 3.2.2.3).

**3.4.1.1** Collecting LNAPLs will be accomplished using a transparent bailer with a double check valve. This bailer will be slowly lowered until the bottom of the bailer is 1-2 in. below the LNAPL-water interface, as determined in SOP 010 then slowly withdrawn. Verify that the interface was sampled by visual inspection of the bailer contents through the side of the bailer. Measure the thickness of the LNAPL in the bailer and note in the Field Notebook. Sample for laboratory analysis. An additional field verification may be performed by decanting the remainder of the contents of the bailer into a glass jar, adding a hydrophobic dye such as Sudan IV, or Redoil, shaking the sample and looking for coloration of NAPL. Alternate field tests are: examine the sample under ultra violet light (many fluoresce), or allow the sample to stand overnight, and examine for interface and/or volatiles in the headspace the following day. Refer to following sections on purging and sample collection for set up and general operation.

**3.4.1.2** Collecting dense non-aqueous phase liquids (DNAPLs) will be accomplished using a transparent bailer with a double check valve. The bailer must be lowered very slowly to the bottom of the well and raised slowly out of the well in a controlled fashion. Sample for analysis as above. The same field check described above may be employed for DNAPL. Refer to following sections on purging and sample collection for set up, and general operation.

**3.4.1.3** If NAPLs are present in the well, **and** a low-flow pump is to be used for purging and sampling, the well will be allowed to re-equilibrate prior to purging and sampling. This will be accomplished by allowing the well to stand undisturbed for at least 8 hours prior to purging and sample collection.

## 3.5 Well Purging - General Rules

Water within the casing of a well will stagnate, degas, lose volatiles, possibly precipitate metals due to changes in redox potential, and may react with the screen and/or casing material. It is therefore necessary to purge a sufficient volume of this stagnant water from the well and/or casing to ensure that a representative sample of formation water can be obtained. Traditionally, the volume of water to be purged was arbitrarily set at 3 to 5 equivalent volumes. Recent advances in sampling technologies have caused a re-thinking of such arbitrary purge volumes. It is for this reason that Monitoring of select chemical and physical properties of the sample medium will be used instead of strict volumes to determine when a representative sample may be taken from a well.

**3.5.1** Acceptable purge/sampling devices include: bailers, high-discharge submersible pumps (purge only), and variable speed, low-flow pumps which include both submersible pumps (purge and sample), and dedicated bladder pumps (purge and

sampling). It is recommended to purge and sample at similar rates with one type device per well. An acceptable exception to this general rule is to use a high-discharge submersible pump to purge a deep, fast-recharging well, and a bailer to sample the same well.

**3.5.2** Peristaltic, gas-lift, and centrifugal pumps can cause volatilization, produce high pressure differentials, and can result in variability in the analysis of some analytes of interest. These types of pumps shall not be used to purge or sample wells.

**3.5.3** To prevent ground-water from cascading down the sides of the screen in to an open hole, thereby aerating the sample, purge rates will closely match recharge rates. If the static water level is within the casing, the initial purge rates may be set high enough to lower the water level to the top of the screen, then reduced to maintain that level.

**3.5.4** Purging will be accomplished with either a submersible pump, a low-flow (submersible or bladder) pump, or bailer. The choice of bailer or pump will be based on depth to water table, volume to be purged, and permeability of the aquifer. If the well recharges rapidly and/or has greater than 20 gallons (estimated EV) to be purged, water may be removed with a submersible pump or a low-flow pump. If the well recharges slowly and/or has less than 20 gallons to be purged, water will be removed with a bailer or a low-flow pump.

**3.5.5** Purging will be accomplished with as minimal disturbance to the surrounding formation as possible.

**3.5.6** Purge water will be containerized[12] on site until analysis of samples is completed. At that time, if the samples are non-hazardous, the water may be disposed of through the waste water treatment plant on-post. If the purge water is found to be hazardous, it will be disposed of as hazardous waste in a licensed TSDF.

**3.5.7** If the water level is within the screened interval and the well recharge rate is less than 0.1 L/min purge the well using a low-flow pump as follows:

**3.5.7.1** Draw the water down to within 1 foot of the top of the pump.

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[12] If, after two rounds of quarterly samples, the water has proven to be uncontaminated, and the purge volume does not exceed 10,000 Gal/day, the purge water may be discharged on the surface, at least 50 ft downhill from the well. If the water is contaminated but does not exceed 100 ppm total VOC, and other contaminants are non toxic to aquatic life as defined in COMAR 26.08.02.03-2, Table 1, MDE may be petitioned on a case-by case basis for a waiver for surface discharge. This letter will be drafted by the contractor for DSHE signature.

**3.5.7.2** Allow the well to recover.

**3.5.7.3** Check and record field parameters (§ 3.7.3).

**3.5.7.4** Repeat steps 3.5.7.1 through 3.5.7.3 then collect samples for metals analysis only [13].

**3.5.7.5** Note the event in the field log book, and report the problem to the APG project manager. If this extremely low recharge problem consistently occurs in a given well, the well may be considered for re-development and/or replacement.

**3.5.7.6** If adjacent wells have elevated VOC levels, additional soil gas surveys will be considered in the vicinity of the low recharge well to help determine the need for replacement.

## 3.6 Purging and Sampling With Bailers

**3.6.1** Bailers may be used for both purging and sampling wells if: a) the well recharge rate is less than 4 L/min, b) depth to the water table is less than 50 ft, and c) less then 20 gal are to be purged (5 EV < 20 gal) [14].

**3.6.2** When purging with a bailer, either a PVC, PTFE, or stainless steel bailer may be used. The bailer will be attached to either a spool of PTFE-coated stainless steel cable or polypropylene rope. If using cable, attach it to the bailer using stainless steel cable clamps. Thoroughly decon the cable after each use, prior to rewinding cable onto spool. Cable clamps and raw cable ends may serve to trap contamination. Exercise particular caution in deconning these areas. If using rope, attach the rope to the bailer using a bowline knot, dispense the needed length (a few feet more than the well depth) and cut the remainder away, then, at the end opposite the bailer, make a slip knot and place it around the well casing or protective posts to prevent losing the bailer and rope down the well. The polypropylene rope will be not reused, it will be properly disposed of. Either type of bailer will be repeatedly lowered gently into the well until it fills with water, removed, and the water will be discharged into an appropriate container until purging is complete. Care must be taken not to unduly agitate the water, as this

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[13] Analyte losses due to volatilization in a drained well are too high for valid VOC sampling (M$^{c}$Alary and Barker, 1987).

[14] These numbers are based on the following assumptions: 1) In purging, it is preferable to remove water at approximately the recharge rate. 2) Four L/min is estimated as the approximate maximum rate at which water can be removed with a bailer from depths of 20-50 feet. 3) Twenty gallons is estimated to be at the limit of the sampler's endurance, at which point fatigue and sloppiness of technique begin.

tends to aerate the sample, increase turbidity, makes stabilization of required parameters (3.6.3) difficult to achieve, and generally prolongs purging.

**3.6.3** After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature (SOP 009), conductivity (SOP 012), pH (SOP 008), turbidity (SOP 036), redox potential (Eh) (SOP 038), and dissolved oxygen level (SOP 037) at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10% in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

**3.6.4** Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles).

**3.6.5** Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly. Refill bailer as needed.

**3.6.5.1** Please see footnote 2. If the controlled flow bottom discharge attachment is used for VOC sampling, attach it to the bottom of the bailer. Using the stopcock valve on the bailer to control the flow, fill sample vials as described above in § 3.3.8.

**3.6.5.2** Remove check valve top and pour unfiltered sample into inorganics sample bottles.

**3.6.5.3** Collect filtered samples as described in § 3.3.9 (above).

**3.6.6** Decon bailer and cable in accordance with SOP 005 § 3.3.1.1

## 3.7 Purging With Pump, Sampling With Bailer

**3.7.1** If the recharge rate of the well is greater than 30 L/min, or the water level is deeper than 50 ft, or more than 20 gal or purge water will be generated (5 EV > 20 gal), then purging and sampling may be accomplished using a submersible pump / bailer combination.

**3.7.2** When purging with a pump, gradually lower the intake until it is submerged within the screened interval. Lower an electronic water level probe to the top of the screen (as determined from completion records) to the monitor water level, start pump, and slowly lower the pump as the water level continues to fall. Care should be exercised to lower the water column to the top of the screened interval (water level probe will stop beeping) but not below the top of the screen if possible. This will ensure that the stagnant layer has been removed, but should minimize the detrimental effects of over pumping the well. Secure hose(s) and/or power cord to casing and place discharge hose into the proper container, downhill and as far away from the well as possible. Determine and record the discharge rate.

Discharge rate = volume of container/time to fill container

The discharge rate will be established at approximately equal to or just greater than the well's recharge rate (determined from well development). If well development records are incomplete, recharge rate can be determined by monitoring the rise/fall of the water level within the casing as one purges the well. If the water level is static at a given pumping rate, but fluctuates up or down as pumping rate is decreased or increased, the pumping rate at which the water level is static is the recharge rate.

**3.7.3** After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10% in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

**3.7.4** Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles). Filtration of metals samples will be accomplished using either an in-line filter attached to the bottom of the bailer, or a funnel and appropriate filter (see § 3.3.9 above).

**3.7.5** Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly, fill sample containers as described above in § 3.6.5

**3.7.6** Decon bailer and cable in accordance with SOP 005 § 3.3.1.1. Decon pump in accordance with SOP 005 § 3.3.1.2.

### 3.8 Purging and Sampling With Low-Flow Pump

To obtain representative samples, subsurface disturbances should be kept to a minimum, thereby preventing sample alteration due to sampling actions. The reasoning behind the use of low-flow pumps to purge and sample monitoring wells is that these pumps minimize physical disturbance (turbulence) at the sampling point and chemical changes (aeration) in the medium. For these reasons, the low-flow pump is the preferred method for both purging and sampling in most cases. For the purposes of this SOP, "low-flow pumps" are defined as either dedicated bladder pumps or variable speed submersible pumps. Practical operational flow rates for these sampling devices range from 0.1 L/min to 30 L/min.

**3.8.1** Low-flow pumps may be used for purging and sampling any well having recharge greater than 0.1 L/min, which is the practical lower limit of pump performance. Below that pumping rate, pump inefficiencies and/or overheating may alter the physical and chemical properties of the sample. If the pump is continuously operated at sampling rates higher than the well recharge rate, the water level will be lowered in the well, possibly allowing aeration of the sample which is unacceptable sampling procedure. Low-flow pumps are suitable for sampling wells with recharge rates lower than 0.1 L/min if precautions are taken to avoid aeration of the sample.

**3.8.2** Low flow submersible pumps will be used as follows:

**3.8.2.1** Lower the pump into the well, slowly so as not to agitate the water, until the pump is at the mid-point of the screened interval or the mid-point of the water column if the static water table lies below the top of the screen [15]

**3.8.2.2** Attach the pump's umbilical cord (which will consist of power cord and sampling tubing) to the protective casing, or lock the cord spool so that the pump cannot move vertically in the well during sampling.

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[15] This assumes a 10-ft. screened interval. If the screened interval is greater than 10-ft., multiple samples should be taken as follows:

- If the screen is 10 - 12 ft., sample the canter of the water column, as outlined above.
- If the screen is longer than 12-ft., and the water column is 10-ft or less, sample the center of the water column.
- If the screen is longer than 12-ft., and the water column fills the screen, or extends above the screen, sample at 1/3 and 2/3 the height of the water column, or about every 6-ft.

**3.8.2.3** Lower the water level probe into the well behind the pump until it just touches water. This will allow the sampler to monitor the water level while purging and sampling, and prevent the inadvertent drying of the well.

**3.8.2.4** Begin purging at the pump's lowest setting, then gradually increase rate[16] until the pumping rate matches the aquifer recharge rate. **If the water level is above the top of the screen**, the pumping rate may be allowed to slightly exceed recharge rate, lowering the water level to no less than 1 foot above the screen, then reduced until it matches recharge rate and purging continued. **If the water level is below the top of the screen**, always keep the purge rate lower than well's recharge rate.

**3.8.2.5** Monitor stabilization parameters listed in § 3.6.3 beginning immediately, using an in-line monitoring system. Record parameters regularly, at a rate of one set of parameters per each 1-3 liters of water removed from the well. When these parameters stabilize to within 10% over 3 consecutive readings, reduce[17] flow rate to 0.1 L/min (if needed) and begin collecting VOC samples directly from the discharge line.

**3.8.2.6** If the well recharges at a rate less than 0.1 L/min, purge until the water level is even with the top of the screen, allow the well to recover and sample immediately.

**3.8.2.7** Remove and decon water level probe (SOP 005 § 3.3.1.5) and pump (SOP 005 § 3.3.1.2).

**3.8.3** The length of tubing used in conjunction with the low-flow pump will be appropriate to the depth of the well (*i.e.* A 100 ft roll of tubing may not be used in sampling a 30 ft well. A 50 ft roll would be used instead, thereby generating less decon solution, and providing less opportunity for physical and chemical changes in the sample due to contact with the spooled tubing (see § 3.8.4)). This means that the contractor will have on hand: a) spools of varying length (*e.g.* 25, 50, 75, and 100 ft spools) or b) several short *e.g.* 10 ft lengths of tubing with a secure means of connecting them end-to-end.

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[16] Some sources indicate that the pumping rate should not exceed 1 L/min, with 0.5 L/min being preferable. The optimal purge rate is highly aquifer dependent, and may range from less than 0.5 L/min to greater than 10 L/min. The purge rate for a given well will; therefore, be a field decision, based on well development, purge, and sampling records rather than SOP mandate.

[17] Sampling should occur at the same rate as purging as long as aeration of sample does not occur.

**3.8.4** When a sampling event occurs during summer months, in full sun, shade will be provided for the spooled tubing. Otherwise the tubing will be an effective water heater, warming the ground-water sample, creating the potential for volatilization of organics.

**3.8.5** Spooled tubing will be monitored to ensure that no air bubbles are trapped at the top of a coil. Trapped air bubbles can enhance volatilization of organics.

**3.8.6** If a dedicated bladder pump is used, follow steps 3.8.2.3 through 3.8.2.5. for purging and sampling.

## 4.0 Maintenance

Refer to manufacturer's requirements for maintenance of pumps and generators.

## 5.0 Precautions

Refer to the HASP for appropriate PPE.

## 6.0 References

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# STANDARD OPERATING PROCEDURE 014
# COLLECTION OF PRODUCTION WELL SAMPLES

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for the collection of groundwater samples from production wells. This protocol will allow for collection of samples from both active production wells (§ 3.4) and inactive production wells (§ 3.10).

## 2.0 Material

a. Conductivity meter
b. Temperature meter
c. pH meter
d. Turbidity meter
e. ORD probe
f. Dissolved oxygen meter
g. Sample bottles and labels
h. Logbook or field parameter form

## 3.0 Procedure

3.1 Upon arrival at the well site, immediately set up and organize the sampling and ancillary equipment. If needed, due to muddy or contaminated ground and/or remoteness from sampling vehicle place plastic sheeting at, or around the sampling location as conditions warrant. Exercise caution not to step on and contaminate the sheeting.

3.2 If the well is remote from the sampling vehicle set up the filtration equipment and place sample containers on the plastic sheet, uphill of the sampling location.

3.3 If a pump is to be used for filtration, situate the portable generator on level ground approximately 15 feet away from and downwind from the sampling location. All generator maintenance (oil and fueling) is to be preformed off site.

3.4 If the well is currently in use. As close as possible to the well, open a tap to a high flow rate and allow the well to purge.

3.5 Obtain a sample of groundwater for temperature, conductivity, ORD, DO, turbidity, and pH measurements. Record values in sampling logbook.

3.6 Take samples for physical stabilization (water quality) parameters every 5 minutes during the well purging process.

3.7 Allow the well to purge until the water quality parameters of pH, temperature, conductivity, turbidity, oxidation-reduction potential, and dissolved oxygen measurements stabilize within 10% in three consecutive 5-minute sampling periods, purging will be considered complete and sampling may proceed.

3.8 Slow water flow rate to a trickle.

3.9 For procedures for collecting samples, with the exception of the sample source being a bailer: Refer to SOP 013 3.2.1; 3.5.1 and 3.5.3 through 3.5.8.

3.10 If the well is not currently in use. Use a pump and bailer, or low-flow pump for sampling. Refer to SOP 013 for purging and sampling protocol.

3.11 decontaminate equipment

3.12 Refer to SOP 1-5, 13, and 16.

**4.0 Maintenance**

Not applicable.

**5.0 Precautions**

Not applicable.

**6.0 References**

USATHAMA. 1990. Installation Restoration Quality Assurance Program, December 1985, 1st edition, March 1987, 2nd edition).

# STANDARD OPERATING PROCEDURE 015
# DOCUMENT CONTROL SYSTEM

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for identifying and storing a complete set of documents relating to project tasks. Each document will receive a unique identification number made up of elements describing the document.

## 2.0 Materials

Not applicable.

## 3.0 Procedure

3.1 Each project-related document will be given to the Document Control Officer.

3.2 The Document Control Officer will record information for each document on a Document Control Sheet which will be retained as a backup record.

3.3 The information from each Document Control Sheet will be maintained in a computer database.

3.4 The individual Document Control Number will be entered on the Document Log Sheet and will be written on the document.

3.5 The storage location for each document will be recorded on the Document Control Logsheet and the documents will be stored in the recorded location.

3.6 The database file will be backed up on a regular basis to prevent accidental loss of the data.

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

None.

## 6.0 References

None.

# STANDARD OPERATING PROCEDURE 016
# SURFACE WATER, GROUNDWATER, AND SOIL/SEDIMENT FIELD LOGBOOKS

## 1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure is to delineate protocols for recording surface water, groundwater, soil/sediment sampling information, as well as instrument calibration data in the Field Logbooks. Example forms are given. Alternate, equivalent forms are acceptable.

## 2.0 MATERIAL

a. Applicable Field Logbook
b. Indelible ink pen

## 3.0 PROCEDURE

All information pertinent to soil/sediment, groundwater, or surface water sampling will be recorded in the appropriate logbook. Each page/form of the logbook is consecutively numbered. All entries will be made with an indelible ink pen. All corrections will consist of line-out deletions that are initialed and dated.

A. Soil/Sediment Logbook

1. Field Parameters (refer to forms 16-a and b)

a. If using carbon paper or self-duplicating forms, before entering data in logbook, insert a sheet protector between form sets to isolate first blank form from remaining forms.

b. HIGH CONCENTRATION EXPECTED?: answer "Yes" or "No."

c. HIGH HAZARD?: answer "Yes" or "No."

d. INSTALLATION/SITE: record the complete name of the installation or site.

e. AREA: record the area designation of the sample site.

f. INST CODE: record the 2 letter installation code appropriate for the installation or site. Correct abbreviations can be found on pages 3-6 of the IRDMS User's Guide for chemical data entry.

g. FILE NAME: record "CSO" for a soil sample or "CSE" for a sediment sample.

h. SITE TYPE: record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on pages 18-21 of the IRDMS User's Guide for chemical data entry. This entry must match the Site Type on the map file form.

i. SITE ID: record a code up to 10 characters or numbers which is unique to the site.

j. FIELD SAMPLE NUMBER: record a code specific for the sample.

k. DATE: enter the date the sample was taken.

l. TIME: enter the time (12 hour or 24 hour clock acceptable as long as internally consistent) the sample was taken.

m. AM PM: circle "AM" or "PM" to designate morning or afternoon (12 hour clock).

n. SAMPLE PROG: record "GQA" (Groundwater Quality Assessment) or other appropriate sample program.

o. DEPTH (TOP): record the total depth sampled.

p. DEPTH INTERVAL: record the intervals at which the plug will be sampled.

q. UNITS: record the units of depth (feet, meters)

r. SAMPLE MEASUREMENTS: check the appropriate sampling method.

s. CHK: check off each container released to a laboratory.

t. ANALYSIS: record the type of analysis to be performed on each sample container.

u. SAMPLE CONTAINER: record the sample container type and size.

v. NO.: record the number of containers.

w. REMARKS: record any remarks about the sample

x. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: record the total number of containers.

y. SITE DESCRIPTION: describe the location where the sample was collected.

z. SAMPLE FORM: record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System (USCS).

aa. COLOR: record the color of the sample as determined from standard Munsell Color Charts.

bb. ODOR: record the odor of the sample or "none". See SOP 001 § 5.0 "Precautions".

cc. PID (HNu): record the measured PID(HNu) values.

dd. UNUSUAL FEATURES: record anything unusual about the site or sample.

ee. WEATHER/TEMPERATURE: record the weather and temperature.

ff. SAMPLER: record your name.

2. Map File Form (refer to form 16-c)

a. The mapfile logbook form will be located on the reverse of the field parameter logbook form, or on an adjoining page of the field logbook (if level book is used)

b. SITE ID: record the Site ID from the field parameter form.

c. POINTER: record the field sample number for the sample being pointed to.

d. DESCRIPTION/MEASUREMENTS: describe the location where the sample was taken, along with distances to landmarks.

e. SKETCH/DIMENSIONS: diagram the surroundings and record the distances to landmarks.

f. MAP REFERENCE: record which U.S.G.S. Quad Map references the site.

g. COORDINATE DEFINITION: write the compass directions the X- and Y-Coordinates of the map run.

h. COORDINATE SYSTEM: write "UTM" (Universal Transverse Mercator).

i. SOURCE: record the 1 digit code representing the Map Reference.

j. ACCURACY: give units (*e.g.*write "1-M" for 1 meter).

k. X-COORDINATE: record the X-Coordinate of the sample site location.

l. Y-COORDINATE: record the Y-Coordinate of the sample site location.

m. UNITS: record the units map sections are measured in.

n. ELEVATION REFERENCE: record whether topography was determined from a map or a topographical survey.

o. ELEVATION SOURCE: record the 1 digit code representing the elevation reference.

p. ACCURACY: record the accuracy of the map or survey providing the topographical information.

q. ELEVATION: record the elevation of the sampling site.

r. UNITS: write the units in which the elevation is recorded.

s. SAMPLER: write your name.

B. Surface Water Logbook (refer to form 16-b and c)

1. Field Parameter Logbook

a. CAL REF: record the calibration reference for the pH meter.

b. pH: record the pH of the sample.

c. TEMP: record the temperature of the sample in degrees Celsius.

d. COND: record the conductivity of the water.

e. For all other sections, see 3.B.1.

2. Map File Form - See 3.A.2.

C. Groundwater Logbook (refer to form 16-b and d)

1. Field Parameter form - See 3.B.1.

2. Map File form (refer to form 16-c)

a. WELL NO. OR ID: record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on pages 18-21 of the IRDMS User's Guide for chemical data entry.

b. SAMPLE NO.: record the reference number of the sample.

c. WELL/SITE DESCRIPTION: describe the location where the sample was taken, along with distances to landmarks.

d. X-COORD and Y-COORD: record the survey coordinates for the sampling site.

e. ELEV: record the elevation where the sample was taken.

f. UNITS: record the units the elevation was recorded in.

g. DATE: record the date in the form MM/DD/YY.

h. TIME: record the time, including a designation of AM or PM.

i. AIR TEMP.: record the air temperature, including a designation of C or F (Celsius or Fahrenheit).

j. WELL DEPTH: record the depth of the well in feet and inches.

k. CASING HT.: record the height of the casing in feet and inches.

l. WATER DEPTH: record the depth (underground) of the water in feet and inches.

m. WELL DIAMETER: record the diameter of the well in inches.

n. WATER COLUMN HEIGHT: record the height of the water column in feet and inches.

o. SANDPACK DIAM.: record the diameter of the sandpack. Generally, this will be the same as the bore diameter.

p. EQUIVALENT VOLUME OF STANDING WATER: use one of the following equations, to determine one equivalent volume (EV):

1 EV = volume in casing + volume in saturated sand pack. Or to restate:

$$1\ EV = (\pi R_w^2 h_w + 0.30\pi(R_s^2 - R_w^2)h_s) * (0.0043)$$

where: $R_s$ = radius of sandpack in inches
$R_w$ = radius of well casing in inches

$h_s$ = height of sandpack in inches
$h_w$ = water depth in inches

$0.0043 = gal/in^3$
and filter pack porosity is assumed as 30%

**-- OR --**

$$\text{Volume in casing} = (0.0043\ gal/in^3)(\pi)(12\ in/ft)(R_c^2)(W_h)$$

where $R_c$ = radius of casing in inches, and
$W_h$ = water column height in feet

$$\text{Vol. in sandpack} = (0.0043\ gal/in^3)(\pi)(12\ in/ft)(R_b^2 - R_c^2)(W_h)(0.30)$$

(if $W_h$ is less than the length of the sandpack),

**-- PLUS --**

$$\text{Vol. in sandpack} = (0.0043\ gal/in^3)(\pi)(12\ in/ft)(R_b^2 - R_c^2)(S_h)(0.30)$$

(if $W_h$ is greater than the length of the sandpack).

where $R_b$ = radius of the borehole, and
$S_h$ = length of the sandpack.

Show this calculation in the comments section.

q. VOLUME OF BAILER OR PUMP RATE: record bailer volume or pump rate.

r. TOTAL NUMBER OF BAILERS OR PUMP TIME: record the number of bailers required to remove 3 equivalent volumes (EV) of water from the well or the total purge time and volume as applicable.

s. WELL WENT DRY? write "YES" OR "NO."

t. NUMBER OF BAILERS OR PUMP TIME: record the number of bailers or pump time which made the well go dry.

u. VOLUME REMOVED: record the volume of water (gal) removed before the well went dry.

v. RECOVERY TIME: record the time required for the well to refill.

w. PURGE AGAIN?: answer "YES" or "NO."

x. TOTAL VOL. REMOVED: record the total volume of water (in gallons) removed from the well.

y. CAL REF.: record the calibration reference for the pH meter.

z. TIME: record time started (INITIAL T(0)), 2 times DURING the sampling and the time sampling ended (FINAL).

aa. pH: record the pH at start of sampling (INITIAL), twice DURING the sampling and at the end of sampling (FINAL).

bb. TEMP: record the water temperature (Celsius) at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

cc. COND: record the conductivity of the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

dd. D.O.: record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

ee. TURBIDITY: record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

ff. ORD: record the oxidation/reduction(RedOx) potential of the water sample at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

gg. HEAD SPACE: record any positive readings from organic vapor meter reading taken in well headspace prior to sampling.

hh. NAPL: Record the presence and thickness of any non aqueous phase liquids (LNAPL and DNAPL)

ii. COMMENTS: record any pertinent information not already covered in the form.

jj. SIGNATURE: sign the form.

D. Field Calibration Forms (refer to form 16-e)

a. Record time and date of calibration.

b. Record calibration standard reference number.

c. Record meter I.D. number

d. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line.

e. Record value of reference standard (as required).

f. COMMENTS: Record any pertinent information not already covered on form.

g. SIGNATURE: sign form.

## 4.0 MAINTENANCE

Not Applicable.

## 5.0 PRECAUTIONS

None.

## 6.0 REFERENCES

User's Guide to the Contract Laboratory Program, USEPA, July, 1984.

## FIELD PARAMETER/LOGBOOK FORM 16-a
## SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? _____ HIGH HAZARD? _____

INSTALLATION/SITE _____ AREA _____

INST CODE _____ FILE NAME _____

SITE TYPE _____ SITE ID _____

FIELD SAMPLE NUMBER _____

DATE (MM/DD/YY) ____/____/____ TIME _____ AM PM SAMPLE PROG. _____

DEPTH (TOP) _____ DEPTH INTERVAL _____ UNIT _____

SAMPLING METHOD:

SPLIT SPOON _____ AUGER _____ SHELBY TUBE _____ SCOOP _____ OTHER _____

| CHK | ANALYSIS | SAMPLE CONTAINER | NO. | REMARKS |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

TOTAL NUMBER OF CONTAINERS FOR SAMPLE _____

### DESCRIPTION OF SITE AND SAMPLE CONDITIONS

SITE DESCRIPTION : _____

SAMPLE FORM _____ COLOR _____ ODOR _____

PID (HNu) _____ UNUSUAL FEATURES _____

WEATHER/TEMPERATURE _____

SAMPLER _____

## FIELD PARAMETER/LOGBOOK FORM 16-b
## GROUNDWATER AND SURFACE WATER SAMPLES

HIGH CONCENTRATION EXPECTED? _____ HIGH HAZARD? _____

INSTALLATION/SITE _____ AREA _____

INST CODE _____ FILE NAME _____

SITE TYPE _____

SITE ID _____ FIELD SAMPLE NUMBER _____

DATE (MM/DD/YY) ____/____/____ TIME _____ AM PM SAMPLE PROG. _____

DEPTH (TOP) _____ DEPTH INTERVAL _____ UNITS _____

### SAMPLING MEASUREMENTS

CAL REF. _____ pH _____ TEMPERATURE °C _____ CONDUCTIVITY _____

OTHER _____

| CHK | ANALYSIS | SAMPLE CONTAINER | NO. | REMARKS |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

TOTAL NUMBER OF CONTAINERS FOR SAMPLE _____

### DESCRIPTION OF SITE AND SAMPLE CONDITIONS

SITE DESCRIPTION _____

SAMPLING METHOD _____

SAMPLE FORM _____ COLOR _____ ODOR _____

PID (HNu) _____

UNUSUAL FEATURES _____

WEATHER/TEMPERATURE _____ SAMPLER _____

## MAP FILE LOGBOOK FORM 16-c
## SURFACE WATER, SOIL, AND SEDIMENT SAMPLES

SITE ID _____ POINTER _____

DESCRIPTION/MEASUREMENTS _____

_____

_____

_____

SKETCH/DIMENSIONS :

MAP REFERENCE _____

COORDINATE DEFINITION (X is _____ Y is _____)

COORDINATE SYSTEM _____ SOURCE _____

ACCURACY _____

X-COORDINATE _____ Y-COORDINATE _____ UNITS _____

ELEVATION REFERENCE _____

ELEVATION SOURCE _____ ACCURACY _____

ELEVATION _____ UNITS _____

SAMPLER _____

## MAP FILE AND PURGING LOGBOOK FORM 16-d
## GROUNDWATER SAMPLES

WELL COORD. OR ID _____ SAMPLE NO. _____

WELL/SITE DESCRIPTION _____

_____

X-COORD. _____ Y-COORD. _____ ELEV. _____ UNITS _____

DATE ____/____/____ TIME _____ AIR TEMP. _____

WELL DEPTH _____ FT. _____ IN. CASING HT. _____ FT. _____ IN.

WATER DEPTH _____ FT. _____ IN. WELL DIAMETER _____ IN.

WATER COLUMN HEIGHT _____ FT. _____ IN. SANDPACK DIAM. _____ IN.

EQUIVALENT VOLUME OF STANDING WATER _____ (GAL) (L)

VOLUME OF BAILER _____ (GAL) (L) or PUMP RATE _____ (GPM) (LPM)

TOTAL NO. OF BAILERS (5 EV) _____ or PUMP TIME _____ MIN.

WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME _____

/OL. REMOVED _____ (GAL) (L) RECOVERY TIME _____

PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (GAL) (L)

| DATE & TIME | QUANTITY REMOVED | TIME REQ'D | pH | Cond | Temp | ORD | Turb | DO | Character of water (color / clarity / odor / partic.) |
|---|---|---|---|---|---|---|---|---|---|
| (before) | | | | | | | | | |
| (during) | | | | | | | | | |
| (during) | | | | | | | | | |
| (during | | | | | | | | | |
| (after) | | | | | | | | | |

COMMENTS _____

_____

SIGNATURE _____

# EXAMPLE
# FIELD CALIBRATION FORM 16-e
# FOR
# pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, ORD, AND DISSOLVED OXYGEN METERS

| INITIAL CALIBRATION | FINAL CALIBRATION |
|---|---|
| DATE: | DATE: |
| TIME: | TIME: |

## pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

| pH STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| 7.0 | | | |
| 10.0 | | | |
| 4.0 | | | |

## CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

| COND. STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| | | | |
| | | | |

## TEMPERATURE METER CALIBRATION

METER ID _____

| TEMP. STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| ICE WATER | | | |
| BOILING WATER | | | |
| OTHER _____ | | | |

# EXAMPLE
# FIELD CALIBRATION FORM 16-e
# FOR
# pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, ORD, AND DISSOLVED OXYGEN METERS

## TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID

| STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

## ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID

| STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

## DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID

| STANDARD | INITIAL READING | RECALIB. READING | FINAL READING |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

COMMENTS _____

_____

_____

_____

SIGNATURE _____

# STANDARD OPERATING PROCEDURE 017
# GROUND PENETRATING RADAR SURVEY

## 1.0 PURPOSE

This guideline provides a description of, and technical management guidance on the use of Ground Penetrating Radar surveys during hazardous waste site investigations at U.S. Army installations.

## 2.0 SCOPE

This guideline provides a description of and the principles of operation, instrumentation, applicability, and implementation of ground penetrating radar (GPR) geophysical surveys. GPR surveys can be used to map subsurface stratigraphy; to rapidly locate buried metallic objects, such as pipes, drums, ordnance, and tanks; to locate buried waste disposal structures; to locate voids within the subsurface; and in some cases can directly detect contaminants.

The document is intended to be used by a site manager to develop an understanding of the method sufficient to permit work planning and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation, which will vary between sites, between target depths and characteristics, and between instruments. The description focuses on methods and equipment that are readily available and typically applied; it is not intended to provide a complete discussion of the state of the art. Specialized expertise is required during both planning and execution of geophysical surveys to develop a target-specific, site-specific, and instrument-specific scope of work, with detailed operating procedures, which will best achieve the goals of the survey.

## 3.0 DEFINITIONS

Dielectric Constant. The measure of the ability of a material to store a charge when an electromagnetic field is applied. This is the measure of the property which determines the reflection, adsorption, and transmission characteristics of the radar signal in the subsurface. Dielectric constants for common materials are listed in Table 017-1.

Ground Penetrating Radar (GPR) Survey. A geophysical survey technique where an (electromagnetic) radar pulse is transmitted into the subsurface and the reflected pulse is measured and recorded.

Penetration or Exploration Depth. The maximum depth at which an object of interest can be detected using a given GPR configuration. The penetration depth is a function of the electrical properties of the subsurface materials and of the GPR signal strength and antenna design.

Radar Trace. The display of reflected signal strength on a graph of lateral distance along the ground-vs-travel time.

Transceiver. Antenna design consisting of a transmitter mounted on or within the antenna - also known as a monostatic configuration. A bistatic antenna configuration consists of the transmitter in a separate housing from the antenna.

Travel Time. The time required for a radar signal to travel from the antenna to a target, reflect, and return to the antenna. Travel time is a function of the depth of the target and the electromagnetic characteristics of the subsurface.

## 4.0 RESPONSIBILITIES

Site Manager. Responsible for the scoping of geophysical surveys during development of the work plan, with the help of the RI leader, site geologist, and site geophysicist.

Site Geophysicist. As a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of these techniques for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical specialists and other personnel as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialists.

Field Operations. Leader responsible for the overall management and coordination of the field work and enforcement of proper work and Health and Safety practices - including coordination, observation, or supervision of Explosive Ordnance Disposal (EOD) or military personnel, subcontractors, or co-contractors as required.

## 5.0 THEORY AND PRINCIPLES OF OPERATION

### 5.1 Description of the Ground Penetrating Radar (GPR) Method

Commercially-available GPR units operate on the principle of time-domain reflectometry, in which the difference in strength and the time delay between a transmitted electromagnetic pulse and its reflection from an object are measured. The time delay, t, is directly related to the propagation velocity of the electromagnetic waves, v, and to the distance between the transmitter and the subsurface object or reflector, D, as follows:

$$t = \frac{2D}{v}$$

Because GPR antenna are generally placed near the ground surface, the distance, D, corresponds to the depth of buried targets that reflect the radar signals.

The strength of a radar signal is a complex function of the distance traveled through the medium and the dielectric constant, the magnetic permeability, and the electrical conductivity of the medium. Radar signals are attenuated rapidly in materials with high dielectric constants. The attenuation of radar signals in subsurface media is dependent on their mineralogy and the water content. Thus, materials such as dry sands and gravels are least absorptive of radar signals, whereas wet clays are highly absorptive. The absorptive properties of the medium limits the penetration depth, i.e., the depth at which targets may be detected. The strength of a radar reflection is also a function of the composition, size shape, and depth of the target. Reflections are strong not only from objects exhibiting large difference in dielectric constant from the surrounding medium, but also which are large in size compared to the radar signal wave length.

The GPR repetitively transmits very short-duration (typically 5 to 10 nanosecond) pulses of high-frequency (typically 80 MHz to 1 GHz) electromagnetic energy through an

antenna that is moved along the ground surface at a constant speed. Reflected pulses are detected by the same antenna at a location corresponding to the distance traveled by the antenna during the transmission and reflection of the pulse, at which point another pulse is transmitted. At a typical antenna speed of 2 miles per hour (3 feet per second), a complete pulse transmit/receive cycle occurs about every 2 inches along the path of the antenna. GPR antenna may have either a monostatic or bistatic configuration. In the monostatic configuration, the antenna is a transceiver that utilizes a fast acting switch changing it to a receiving antenna immediately following pulse transmission and back to transmitting antenna for generation of the next pulse. The bistatic configuration utilizes separate transmitter and receiver antennas. The received signal is transmitted to the central control unit where the initial signal processing takes place and the pulse is typically recorded with digital tape recorders and displayed on an oscilloscope and/or graphic recorder. The reflected data may also be run through a video display units for real-time viewing. Recording enables the geophysicist to play back data later for further processing or analysis.

Common radar antennas operate at approximate center frequencies of between 80 and 1000 MHz. The higher frequencies provide the best resolution for smaller targets, however, the penetration depth is roughly inversely proportional to frequency. Thus, the design of any GPR survey requires an analysis of the trade-off between target resolution and depth of penetration so that the optimal antenna frequency may be selected.

## 5.2 Applicability

The antenna frequency and equipment configuration used for ground penetrating radar (GPR) surveys, as well as the survey methodology, is dependent upon site-specific conditions and the particular objectives of the survey. In addition, analysis of data requires interpretive skills developed through extensive training in the geosciences/geophysics and exposure to the application of GPR to a variety of field problems. The supervising geophysicist should have experience applying the technique at sites presenting a wide range of soil conditions and where the objectives of surveys have involved identification of subsurface features that include distinct targets such as buried metal objects and more subtle features such as back-filled excavations. Because of the highly interpretative nature of the GPR work it is difficult, therefore, to establish a standard operating procedure that would apply to all site surveys. The following material, however, offers some general guidelines for the conduct of GPR surveys, including information on the principles of operation, instrumentation, application and interpretation associated with standard GPR methods.

Because the GPR method utilizes high-frequency radio waves that are of higher frequency than the wave frequencies associated with seismic surveys or resistivity soundings, target resolution is better than that provided by these other two techniques. However high frequencies are more susceptible to signal attenuation. In typical clay materials, for example, the useful penetration depth is about 3 feet using a 100 megahertz (MHz) antenna. The same antenna used to survey over an area of dry sand soils (< 20 percent moisture); however, will allow for a penetration depth of about 30 feet.

Ground penetrating radar (GPR) is typically useful in detecting three classes of subsurface anomalies - metallic objects, layers/areas/objects of differing electrical properties, and disruptions in the layering of the subsurface materials. Buried metallic objects, such as pipes, barrels, bomb casings, and underground storage tanks, typically show prominent hyperbolic signatures due to the high contrast in electrical conductivity between metals

and soil materials. Buried materials of differing electrical properties, such as concrete or brick walls, or water or air-filled pvc pipes, can also be detected. In some cases, contamination in the soils or floating on the water table can be directly detected using GPR. For example, free petroleum product strongly attenuates the radar signal and can often be detected floating on the water table. GPR is also useful for delineating disruptions in the natural soil layering, due to excavations, disposal trenches, or voids.

Characterization of the subsurface with GPR involves the generation of plots of distance along a survey line vs. reflected wave travel time and signal strength (amplitude) for return pulses from subsurface reflectors. The "reflective" property of subsurface materials or objects is associated with strong contrasts in the electrical properties between materials or objects that are in contact. These strong contrasts may be associated with naturally occurring stratigraphic or structural boundaries or from the disruption of natural materials and/or the presence of man-made materials (i.e., buried drums or contaminants). The dielectric constants for natural and man-made materials provide a parameter for quantifying and comparing electrical properties of subsurface materials. Disruptions of natural earth materials (e.g., excavation of and back-filling of trenches or formation of voids through subsidence) change the materials bulk physical and chemical properties, and, consequently, their electrical properties. These contrasts make GPR an effective means for identifying the boundaries of abandoned landfills or locating drums or subsurface contaminant plumes. Depending on the depth of penetration that may be achieved, the method may be used to map the interface between the saturated and unsaturated zones, map the depth to bedrock, locate sinkholes, or map fracture systems more efficiently than may be accomplished with other conventional methods such as soil boring programs.

A major limitation of GPR is subjective process involved in data interpretive. Weak reflections from objects of interest may be indistinguishable from noise in the radar record or masked by stronger reflections from other objects. However, special computerized signal processing and enhancing routines originally developed for seismic processing now enable the geophysicist to enhance weaker signals, but the success of the data interpretative process ultimately depends heavily on the experience of the geophysicist.

## 6.0 INSTRUMENTATION

The standard array of GPR instrumentation consists of a transmitter/antenna unit(s), a DC power supply, a control unit and the signal processing circuitry connected to the antenna by a cable; an oscillograph or video display unit, and a plotter or an analog or digital recording device. The field equipment may be vehicle mounted but is small enough that it may be hand carried into areas not having vehicle access. Examples of GPR units are Geophysical Survey Systems, Incorporated's (GSSI) SIR System 8 and System 10 ground-penetrating radar systems. Both GSSI systems utilize impulse radar technology to sense and record continuous, high resolution profiles of subsurface materials.

The primary variable in GPR instruments is the antenna configuration. Antennas are currently available in both monostatic (one-piece) and bistatic (two-piece) configurations and are generally designed to detect radar signals ranging from about 80 to 1000 MHz.

## 7.0 DATA ACQUISITION

### 7.1 Field Procedures

7.1.1 Planning. The planning phase of a GPR survey requires the development of information pertaining to the geophysical characteristics of the site and how they might vary across the area to be surveyed. The type and structure of soils, geologic formations, and the approximate elevation of the water table are critical factors that should be established. In addition to developing information about the characteristics of earth materials that are present at the site, the geophysicist needs to develop information about the targets of interest and factors that may interfere with execution of the survey or cause excessive noise in the acquired data. If possible, it is useful to know the depth, size and shape, and type of potential targets to be detected. The depth and size of the objects are very important such that the appropriate antenna configuration and spacing between survey lines may be selected. Surveys over known objects on site are useful to cross-check the survey detection limit and depth calculations; however, they are not absolutely necessary. Other specifications include accuracy of locational resolution desired, probable weather conditions during site activities, and the type and sophistication of data processing required for data interpretation and presentation.

The time and effort required to perform GPR field surveys depends on several factors including the sophistication of the equipment used, the type of target, and logistical considerations. Additional time and effort are required to process and interpret data. Sophisticated data processing, detailed interpretations, and high-quality displays require considerable computer usage and may require more time to complete than the actual field survey.

7.1.2 Site Layout. GPR surveys are performed by establishing a grid of parallel survey lines across the site and moving the radar antenna along each of these lines. The spacing between the lines is dependent upon the size and depth of the targets of interest and the objectives of the survey (i.e. reconnaissance or detailed survey). GSSI systems employ an electronic marking device that records a mark on direct field graphic records or within digital radar files. Generally, double marks or clicks are made at the beginning and end of the survey lines and single clicks are recorded as the antenna passes over predetermined distance intervals. Following acquisition of data in the field the data is downloaded from digital tape to PC storage for subsequent processing and analysis in the office.

7.1.3 Depth Determination. To determine the depth of anomalies noted on radar profiles, it is necessary to convert the data from recorded travel times, to depths. This is done by determining the transmission velocity(ies) associated with the earth materials. The velocity of electromagnetic waves within the subsurface at the site may be determined through excavation of observed targets to determine their depth of burial. Several excavations may be made and an average or range of transmission velocities for materials at a given site may be determined. Once velocity values are determined, interpretations or determinations concerning the depths of other signatures or anomalies may be made. Two other simpler methods, discussed in the following section, provide a simple means of determining the radar-wave velocities through subsurface materials and, therefore a means for converting travel-times to depths. These are two graphical approaches, one involving examination of hyperbolic signatures and the other involving the examination of changes in the travel-time within a given subsurface section as the transmitter and receiver antenna (bistatic configuration) are moved

away from each other. The later is referred to as wide angle radar reflection (WARR) measurements.

7.1.4 GPR Survey Measurements. Once an optimal GPR survey configuration has been decided upon, The survey itself is straightforward, barring changes in the subsurface electrical characteristics. The unit is activated and the antenna is towed along one of the prearranged grid lines. An electronic mark is placed on the data at the beginning and end of each survey line, and every 10 or 20 feet along the line, so that the position of the data and anomalies on each radar line is readily apparent. The locations of surface metal, utilities, walkways, etc. should be noted on the field logs during the survey.

### 7.2 Data Format

Although much of the data obtained in GPR surveys are automatically recorded by the instrumentation, additional information to unambiguously identify and to interpret each trace should be recorded in the field logbook. At a minimum, the field logbook should contain the following information:

- Project name, number and location
- Company or organization
- Date and time of day
- Operator's name
- Line and trace designation (also recorded directly on the signal recording medium)
- Equipment serial numbers
- Antenna frequency
- Direction and speed of antenna movement
- Weather and temperature
- Site map coordinates at the beginning and end of the trace
- Other pertinent notes, remarks or comments
- Electromagnetic velocity in the subsurface medium at the nearest calibration point.
- Map of grid and survey lines referenced to permanent features (wells, buildings, etc.)
- Data acquisition parameters (gain, range, filters used)

## 8.0 DATA PROCESSING AND INTERPRETATION

### 8.1 General

Reflected radar signals are electronically processed and displayed as an intensity-modulated time spectrum, where the time corresponds to target depth as described above. The series of signals corresponding to the reflected pulses as the antenna moves along a path forms a three-dimensional data set containing distance of traverse, depth, and intensity information.

Typically, the data are recorded on magnetic tape and/or displayed on a graphic recorder with distance displayed along the X-axis, time (i.e., depth) displayed along the Y-axis, and the intensity given by the degree of darkness of the trace. In a typical survey, a series of parallel traverses are made with the GPR, and the series of graphical traces provide X-Y-

Z locational, as well as intensity of reflection information for targets of interest. Interpretation of anomalies in GPR traces requires considerable subjective evaluation by a trained geophysicist. Extensive experience is essential to distinguishing target reflections from inherent system noise and interferences. In many cases, the anomalies due to targets of interest are small compared to varying reflections from the antenna system, the ground surface, geologic perturbations, and other interferences. Similarly, an acceptable interpretation of target depth from travel-time data requires knowledge of the varying geophysical characteristics across the site area surveyed.

A radar antenna transmits a "cone" rather than a thin beam of electromagnetic energy. The result is that reflections are obtained from objects not directly below the antenna. As the antenna moves across the plane of an object, reflections are obtained for a considerable distance along the antenna path. The radar reflections will generate a hyperbolic signature pattern. The size and shape of this hyperbolic signature varies as a function of the wave velocity within the subsurface materials, the size and the orientation of the object relative to the radar wave pulse, and the rate, t, at which the antenna configuration is moved along the surveyed line. The signal travel times will vary corresponding to the distance between the antenna and the object.

## 8.2 Hyperbolic reflectors

A discrete spherical target, therefore, will generate a hyperbolic reflection patterns with the apex of the hyperbola corresponding to the location and depth of the object. Multiple or odd-shaped targets or targets of considerable size in comparison to the radar wavelength) will generate complex reflection patterns consisting of overlapping hyperbolas. Thus, a true "picture" of subsurface objects is not obtained, and experience is necessary to translate the complex tracings into information concerning target depths, size, or shape.

## 8.3 Depth Determination

The calculation of the depth of exploration for a particular GPR configuration may be accomplished by knowing the dielectric constant of the earth materials or the depth to a particular reflector that is clearly visible on the radar record. In addition, the depth may be calculated through identification and graphical measurement of hyperbolic signatures or through the development wide angle refraction and reflection (WARR) data. The values for resistivity (inverse of conductivity), dielectric constant, and wave velocity in Table 017-1 are typical values; however, the real values for subsurface materials may change significantly over short lateral distances. The use of the published values exclusively may not yield depth calculations of sufficient precision.

8.3.1 WARR Method. The WARR measurement requires the use of actual data from the surveys conducted at each of the areas, are used to determine the velocities of the pulse, depth to significant reflectors, and the total depth of penetration. WARR measurements are performed with a bistatic (separate transmitter and receiver) antenna configuration. The two antennae are moved away from each other at a constant rate as a record is developed. By knowing the distance between the transmitter and receiver at a given time during the development of the record, the geophysicist may calculate the electromagnetic wave velocity(ies) using a $t^2$-$x^2$ plot. The recorded reflections are graphically plotted with the square of the distance between transmitter and receiver on the x-axis and the square of the time period on the y-axis. The inverse of the slope of the line of the reflection is the velocity of propagation. A simple calculation relating distance versus time will yield a velocity of the propagated pulse. Once the

transmission velocity is known, depths to reflectors may be determined. The WARR measurements require the presence of good horizontal reflectors.

8.3.2 Hyperbola Geometry Method. The second method, the identification and measurement of hyperbolas, also provides good estimates of GPR penetration depth. The shape and position of the hyperbolic signatures, caused by cylindrical objects (e.g. pipes or drums), is a function of the wave velocity within the subsurface, the size of the object, and the rate at which the antenna is moved. This type of calculation is possible because the GPR pulse has a broad cone-shaped radiation pattern and cylindrical objects have numerous surfaces that are normal to the pulse as the antenna is moved along a survey line: first approaching, directly over, and then away from the buried object.

The cone-shaped radiation pattern has an included angle of approximately 90 degrees. For flat or horizontal reflectors, only the vertical component of the pulse is reflected back to the receiver and the other sub-vertical components of the pulse are reflected or refracted away from the antenna not producing a record. Therefore, the two-way reflection sensitivity, or field of view, for flat surfaces is small. For cylindrical objects in the subsurface the sub-vertical components of the pulse may also be reflected back to the receiver as the antenna approaches the object. Because the round trip travel-time for the reflected pulse decreases as the antenna approaches the object, reaches a minimum directly over the object, and then increases again as the antenna moves away from the object, the record that is produced has a hyperbolic (inverted horse shoe) signature or pattern. The hyperbolic pattern generated on a radar record, distance (x) vs time (y), is a representation of the maximum round trip travel for the pulse when it first intersects the pipe, decreasing as the antenna approaches the axis of the pipe, and then increasing to a maximum again as it moves away from the pipe. Therefore, the position and shape of the hyperbola in the radar record are directly related to the wave transmission characteristics of the subsurface materials. Utilizing this simple geometric relationship the geophysicist may calculate transmission velocities for the various areas and the depths of penetration for the pulse and the depth to significant anomalies. Essentially, this results in a conversion of time on the y-axis of the GPR records to depth.

($T_z$) represents the time of travel (or distance once velocity is known) for the "first" or "last" reflection that is produced when the center axis of the antenna is 45 degrees from the curved object (e.g. pipe). This reflection is recorded in the GPR record as the ends of the limbs of the hyperbola. This value is also referred to as the slant range or distance between the target and the antenna. The distance represented by ($T_z$) is greater than the actual depth of the target. As the antenna approaches the object the radar signature (top of the hyperbola) approaches the actual depth of the object. ($T_y$) represents the time of travel (or distance once the velocity is known) when the antenna is directly over the reflector.

If it is assumed that the subsurface materials over path distance (Z) have on average the same electrical properties as those for path distance (Y) and the dimensions of the pipe are considered insignificant relative to the other dimensions, then the following geometric relations are true:

$$X^2 + Y^2 = Z^2$$

and

$$\frac{T_y}{T_z} = \frac{Y}{Z}$$

Combining these equations and solving for Y (the actual depth of the pipe), the following equation may be derived:

$$Y = X * (1/((T_z/T_y)^2 - 1)^{0.5})$$

X = distance along the ground in any convenient unit.
Y = depth of pipe in distance units.
$T_z$= travel time or "slant range" to pipe in any units.
$T_y$= travel time when the antenna is directly over the pipe.

The distance along the surface and the lengths of the $T_z$ and $T_y$ scaled directly from the record are all that are needed to calculate the depth to the object causing the hyperbola. While these graphical measurements and calculations are relatively simple, they may become tedious when performed on all of the hyperbolae. This process has been automated with RADAN software available from GSSI.

## 9.0 SURVEY DESIGN

### 9.1 Prerequisites

Appropriate planning of GPR surveys requires a basic understanding of the site subsurface and hydrogeologic features, including the probable lateral variability. A statement of work should be generated that describes, in as much detail as possible, the known site conditions that may affect the subsurface electrical properties and the objectives of the proposed survey efforts, and allows for changing the survey parameters if the subsurface conditions change or are not found to be as described. The type and degree of data interpretation and the desired format for data presentation should also be specified if possible.

### 9.2 Instrument Selection

The important instrument selection decision for the survey lies in the determination of the optimal antenna. Generally, the higher the antenna frequency, the smaller the object that it can resolve, and the smaller its depth of penetration. The optimal antenna is the one with the highest resolution that will have sufficient depth of exploration. However, the depth of penetration for a given antenna will vary widely between sites, primarily due to soil moisture content and the amount of clay/sand ratio of the soils. Overnight rainfall can often elevate the soil moisture content such that an antennas depth of penetration can be degraded by 30% in a few hours. No one antenna configuration is suitable for all cases, even at the same site. Ideally, the survey crew should carry multiple antennas to the site for the survey so that an optimal configuration will always be available.

### 9.3 Grid Design

The survey grid should be designed such that the GPR measurements are spaced to adequately define the distribution and extent of the exploration targets. For convenience,

an orthogonal survey grid, located relative to a known location, is usually established over the survey area and the GPR measurements and anomalies are located by their grid coordinates (x,y). This is a convenient method for recording the data in an organized manner, for graphically displaying the data, and for ease of locating the anomalies detected. For determination of geologic features or to detect large targets, reconnaissance-type, low-resolution surveys are typically performed with a track spacing of 5 to 20 feet. Surveys to detect small discrete targets or to resolve target details require a track spacing of 1 to 5 feet.

## 10.0 QUALITY CONTROL

The application of quality control principles to GPR surveys involves:

### 10.1 General

Requiring that trained geophysicists be utilized in all aspects of the survey including the preparation of the project workplans and survey designs, conduct of the survey, interpretation of the data, and presentation of the results. Workplans need to be written such that some degree of latitude is given to the survey team so that they may adjust to site conditions.

- Clearly defining the objectives of the survey in terms of the: (a) degree of detail needed and the size of the area to be surveyed; (b) type, depth, size and composition, of targets of interest (if known); (c) resolution required; (d) schedule limitations; (e) degree of sophistication required for data presentation and interpretation; and (f) specific deliverables required.

- Defining specific field quality control procedures.

- Justifying rejection of any data from a data set. Field data sheets should contain all observed data and the conditions that could impact data validation.

- Recording all field data in permanent ink in a bound logbook and tape logs with each page signed and dated by the survey team leader.

- Properly calibrating the GPR unit. In general, the objectives of most geophysical surveys may be achieved by obtaining relative measurements across the area surveyed. Absolute calibration is, therefore, of lesser importance. However, a properly calibrated instrument provides an added measure of data validity.

- Evaluating all sources of noise, interference, and obstructions at a site and noting their potential effects on certain measurements made along a surveyed line. These real-time field observations later allow for correcting the data results for noise, validating suspected external sources, and in detecting problems that may jeopardize the objectives of the survey.

### 10.2 Calibration

Calibration of the radar traces for depth determinations is performed as follows:

- For reconnaissance surveys or for surveys where lateral resolution is more important than depth, the traces may be roughly calibrated by estimating the velocity of electromagnetic waves in the media at the site from published values for similar materials. The crudeness of the calibration is evidenced by considering that the velocity may vary by more than an order of magnitude, depending on the soil/rock properties and the moisture content.

- For surveys requiring reasonable resolution of target depth, the travel time to targets of known depths must be determined at each site. As the radar trace is made over the known targets, the reflection patterns provide direct depth-calibration points on the trace. Sites with uniform lithology may require only a few depth calibrations, but generally it is necessary to perform these calibrations at several locations and at several depths throughout the area of interest. Each radar trace should be referenced to the calibration most representative of the trace coordinates at the site. The preferred method is to use buried objects of known depth as calibration targets, or to excavate to detected objects and measure their depth below ground surface. A less desirable (but often necessary) procedure is to bury standard targets at various depths within the area of interest.

- In addition, WARR measurements may be conducted at several locations over the entire area of the site to determine how the transmission velocities change. If necessary specific transmission velocities may be determined for each subarea within the surveyed area.

### 10.3 Daily Quality Control

All radar traces and interpreted data sets should be accompanied by quality control data that indicate the level of quality of the data. Periodic replicate measurements should be made so that measurement precision may be established. Time and/or depth calibrations should be performed on a daily basis.

A calibration that yields significant changes in instrument parameters or travel time may indicate the need for repetition of data or increased density of travel time calibrations in the area of interest. Graphical data should be reviewed during the field activities to determine that data quality is adequate, and whether the survey results appear to be consistent with conceptual models.

## 11.0 HEALTH AND SAFETY CONSIDERATIONS

### 11.1 General

All procedures for hazardous waste site entrance, traverse, and egress that apply to general field operations established in the Site Health and Safety Plan also apply to conduct of geophysical surveys. Because GPR surveys are non-intrusive, the potential for exposure to chemicals of concern is generally low. Conducting GPR surveys consists of

traversing the site on foot or in vehicles, and there are extended periods during which personnel are subject to adverse environments at the site. Geophysics survey personnel will adhere to the guidelines of the Site Health and Safety Plan for field activities.

Towing the GPR antenna by hand may involve considerable physical activity or hazards, especially on sloping or rough terrain. The geophysical methods discussed herein do not require extremely strenuous activity, and exposure to heat or cold is similar to that during other field activities. Extreme weather conditions will have adverse effects on the time required to obtain validated data, there by increasing the duration of personal exposure to the elements and to hazardous site influences.

### 11.2 Explosive Ordnance Disposal (EOD)

For surveys in areas where ordnance disposal or the presence of ordnance is suspected, EOD personnel will clear site access and survey areas prior to survey activities as per the Health and Safety Plan. The GPR methods described here involve the transmission of a weak electromagnetic radar signal whose characteristics vary between antenna transceivers. These transceivers generally emit a total electromagnetic signal of 0.1-4.0 milliwatts over a frequency spectrum ranging from 80-1000 MHz. These signal strengths are total output strengths measured at the transceiver. The signal strength at any given frequency is measured in microwatts and falls off rapidly with depth in the subsurface. It is possible that this signal may not be safely broadcast in the vicinity of certain types of (live) ordnance, particularly those which may have active radar proximity detonators. The individual GPR instruments proposed for the survey(s) will be cleared for use, for each site where ordnance may be present, by the facility Health and Safety officer.

## 12.0 POTENTIAL PROBLEMS

GPR surveys are subject to a wide variety of potential problems that may impact conduct of the survey and/or proper interpretation of the survey results. The two most significant problems include:

- Noise and Interferences. GPR measurements may be affected severely both by natural and by man-made sources of electromagnetic interference. Sources of system noise that degrade the quality of radar traces include improper spacing of antennas above ground, improper cable placement, location of antennas too close to other system components, and facility instrument operation. Because reflections are obtained from any object with a dielectric constant differing from the surroundings, large masses or a high density of buried or surface rocks, metal, debris, wet soil, or structures can mask targets of interest. Some antennas are not shielded on their top surface and, as a result, are subject to interfering reflections obtained from overhead objects such as trees, power lines, and buildings. Topographic and geologic features can also interfere with acquisition of high-quality target detection data. Small depressions in the ground surface, the presence of boulders, clay lenses and moist soil zones affect both the capability to detect the target and determine its depth. Sources of electromagnetic energy in the vicinity, such as radio or television transmitters, or navigational radar antennas may result in spurious signals in the radar traces. In some cases, these problems may be minimized by judicious selection of radar and/or data communications frequency, and by scheduling the surveys during period of transmission inactivity.

- Rebar. The presence of steel reinforcing mesh or concrete rods in concrete(rebar) is a common problem for GPR surveys. The rebar generates regularly spaced hyperbolas in

the data. If the rebar is thick and closely spaced, the reflections from the rebar can completely obscure the underlying material.

- Weather Conditions. Because water absorbs radar signals, wet weather has a very serious effect on the ability to perform GPR surveys. Physical difficulties in executing a survey over wet terrain also may be expected. Therefore, survey activities should be planned, if at all possible, during periods when dry weather can be expected. The survey schedule should also account for moist soil conditions and changes in these conditions.

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TABLE 017-1

| | Resistivity (ohm/meter) | Relative Dielectric Constant | Electromagnetic Wave Vel. (cm/ns) |
|---|---|---|---|
| Air | ---- | 1 | 30 |
| Asphalt | ~10 | 2.5-3.5 | 16-19 |
| Concrete | ~10 | 3-9 | 10-17 |
| Conglomeratic soil | 100-1000 | 9-14 | 8-10 |
| Sandy soil | 50-400 | 11-18 | 7-9 |
| Silty soil | 20-200 | 14-36 | 5-8 |
| Clayey soil | 1-30 | 25-56 | 4-6 |
| Sandstone | 200-1000 | 9-14 | 8-10 |
| Limestone | 2000-10000 | 6-11 | 9-12 |
| Ice | ---- | 3.2 | 17 |
| Water | ---- | 81 | 3.3 |
| Sea Water | 5x10-2 | 81 | 3.3 |

*Reference: 1987, Operation Manual, Model 2441 GeoRadar-I, OYO Corporation.

# STANDARD OPERATING PROCEDURE 018
# ELECTROMAGNETIC INDUCTION (TERRAIN CONDUCTIVITY) SURVEYS

## 1.0 PURPOSE

The purpose of this standard operating procedure is to provide a general description and technical management guidance on the use of electromagnetic induction (terrain conductivity) surveys during hazardous waste site investigations at U.S. Army installations.

## 2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability, and implementation of electromagnetic induction methods used during hazardous waste site investigations to determine subsurface conductivity. Measurements of subsurface conductivity can be used to determine the presence and approximate extent of subsurface contaminants, buried drums, and metal containers, along with depth to the water table and structural characteristics of the subsurface environment.

The document is intended to be used by a site manager to develop an understanding of the method sufficient to permit work planning and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation, which will vary between sites, between target depths and characteristics, and between instruments. The description focuses on methods and equipment that readily available and typically applied; it is not intended to provide a complete discussion of the state of the art. Specialized expertise is required during both planning and execution of geophysical surveys to develop a target-specific, site-specific, and instrument-specific scope of work, with detailed operating procedures, which will best achieve the goals of the survey.

## 3.0 DEFINITIONS

Apparent Conductivity. The quantity measured during an electromagnetic induction survey; proportional to the actual conductivities of subsurface materials.

Conductivity. The property of a material to conduct an electric current, roughly equal to the reciprocal of resistivity.

Current. The quantity of charge transmitted per unit time.

Electromagnetic Induction (EMI). The process of transmitting a primary electromagnetic field which induces a secondary magnetic field in magnetic or paramagnetic objects or volume.

Electromagnetic Induction Survey. A geophysical exploration method whereby secondary electromagnetic fields are induced in the subsurface and whose strength is a measure of ground conductivity.

Horizontal Dipole Coil Orientation. The horizontal dipole coil orientation induces a field response which is greatest in the near-surface and falls off monotonically with depth.

In-phase Signal Component. The 180 degree (in-phase) component of the induced electromagnetic field. The in-phase component is highly sensitive to buried metal, but has no direct relationship to the earth conductivity.

Potential. Intensive property of electric fields, equating to the ability to do work.

Profiling. A survey technique where the EMI instrument, using a fixed coil spacing, takes measurements along a survey line to determine how the readings vary with location.

Quadrature Phase Signal Component. The 90 degree component of the induced electromagnetic field which is linearly proportional to the earth conductivity.

Resistivity. The property of a material which resists the flow of an electric current.

Sounding. A survey technique which measures the conductivities from progressively greater depths by increasing the coil spacings of the EMI instrument about a given location.

Vertical Dipole Coil Orientation. In the vertical dipole coil orientation, the EMI instrument has a greater depth of exploration than in the horizontal dipole mode, and the near-surface materials add relatively little to the total measurement.

## 4.0 RESPONSIBILITIES

Site Manager. Responsible for the scoping of geophysical surveys during development of the work plan, with the help of the RI leader, site geologist, and site geophysicist.

Site Geophysicist. As a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of these techniques for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical specialist and other personnel as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialists.

Field Operations. Leader responsible for the overall management and coordination of the field work and enforcement of proper work and Health and Safety practices - including coordination, observation, or supervision of Explosive Ordnance Disposal (EOD) or military personnel, subcontractors, or co-contractors as required.

## 5.0 THEORY AND PRINCIPALS OF OPERATION

### 5.1 Description of Electromagnetic Induction (EMI) Method

In the Electromagnetic Induction (EMI) method, the electrical conductivity of a geohydrologic section is measured by transmitting a high-frequency electromagnetic field into the earth, which induces eddy currents that generate secondary electromagnetic fields. These secondary electromagnetic fields can then be detected by a receiver. The primary electromagnetic field is transmitted by an aboveground transmitter coil, and the resulting secondary electromagnetic fields are detected by an aboveground receiver coil. Thus, EMI measurements do not required direct ground contact, as is the case for resistivity measurements, and surveys across a line or area may be performed quite rapidly.

EMI instruments are calibrated to read subsurface conductivity directly in units of millimhos per meter, where

1000 millimo per meter = 1 / ohm-meter

This relation indicates that the conductivity obtained from EMI measurements varies inversely with the resistivity measured using a resistivity survey. However, because the subsurface sections associated with the two methods are generally of different depth or cross-sectional area, there is not an exactly inverse relationship between conductivity and resistivity surveys.

The conductivity value obtained by an EMI instrument depends on the combined effects of the number of soil and rock layers, their thicknesses and depths, and the inherent conductivities of the materials. The quantity actually measured is an apparent conductivity of the earth volume between the ground surface and an effective penetration depth, which is defined as the depth at which variations in conductivity no longer have a significant effect on the measurement. The sampling depth is related to the spacing between the transmitter and receiver coils of the instrument, approximately as follows:

$$\text{Sampling depth} = 1.5 \text{ (coil spacing)}$$

Vertical profiling can be accomplished by multiple measurements about a point, with varying coil spacings. Horizontal profiling is performed by making measurements along traverses with a fixed coil spacing.

**5.2 Applicability**

The measurement of subsurface conductivity at a hazardous waste site provides a valuable contribution to site characterization for the following reasons:

- Conductivity is a function of the geohydrologic section and is overwhelmingly influenced by the presence of water (where buried metal is not present). Therefore, conductivity can provide indirect evidence on the porosity and permeability of subsurface materials and the degree of saturation. These parameters, in turn, are directly related to subsurface lithology and to the potential for infiltration/migration of contaminants from a source area.

- Conductivity is influenced by the presence of dissolved electrolytes in soil or rock pore fluids. Contaminant plumes in the vadose (unsaturated) and saturated zones can be mapped if there is sufficient change in conductivity to be detected by EMI measurements. In general, contaminant plumes of inorganic wastes are most easily detected because conductivity may be increased by one to three orders of magnitude above background values. The limit of detection is a change from a background of 10%–20%. Plumes of non-polar organic constituents from spills or leaking containers may be detected if sufficient soil moisture has been displaced to affect the ground conductivity to a measurable degree.

- Conductivity can be used to detect the presence of buried wastes if the degree of saturation, containerization, or inherent electrical properties of the wastes produce sufficient variation from the soil matrix. Practically, only large sources, such as a buried disposal trench or a group of buried drums, can be detected by these methods. The degree of detail provided by typical surveys cannot distinguish the size, shape, or mass of sources except in a qualitative manner.

For these reasons, conductivity surveys should be investigated as potentially appropriate site characterization tools when any of the following information is desirable:

- Detection and mapping of contaminant plumes; the rate of plume movement may also be deduced from measurements made over time.
- Estimates of depth, thickness and resistivity of subsurface layers, depth to the water table, or probable geologic composition of a layer.
- Detection, mapping an depths of burial pits, landfills, clay caps or lenses, or deposits of buried waste.
- Determination of locations for drilling to intercept contamination or to investigate aquifer properties.
- Corroboration of limited chemical and geohydrologic data at a site.

## 6.0 INSTRUMENTATION

EMI instruments are available in two forms:

### 6.1 Fixed Coil

Single-piece models operable by one person, with fixed coil spacings of 1, 4, and 12 feet; these provide sampling depths on the order of 1.5, 6, and 15 feet, respectively. The Geonics EM-38 (1 foot coil spacing) and EM-31 (12 foot coil spacing) are examples of this type of instrument.

### 6.2 Variable-coil

Dual-coil models, operable by two persons, with variable coil spacing up to about 40 feet (sampling depth up to about 60 feet). The Geonics EM-34 is an example of this type of instrument.

The 12-foot fixed coil and the dual-coil apparatus are most commonly used in hazardous wastes site investigations. In either case, an additional person to record data and identify measurement locations is highly desirable and more time efficient. The instruments are calibrated to read directly in conductivity units, and values are typically read and recorded on a data sheet. Some units have been modified to provide direct digital recording on magnetic tape.

## 7.0 DATA ACQUISITION

### 7.1 Field Procedures

7.1.1 Planning. Known or assumed geohydrologic features of the site, potential source locations and migration characteristics of hazardous constituents, are used to select specific techniques and equipment to establish appropriate locations and depths for geophysical measurements. The level of detail necessary (data quality

objectives) determines the amount of effort and, in simple terms, the required number and density of data points. The data quality will depend on the method and specific equipment selected, the supporting hardware and software capabilities, and site-specific conditions.

Most of the details can be planned prior to site activities; however, some leeway must be accorded to the field procedures to account for variable site conditions and weather.

7.1.2 Site Layout. One of the most labor-intensive and time-consuming aspects of the field work involves layout of grids, and surveying or careful measurement of locations to allow geophysical surveys to be accomplished is a systematic, documentable manner. Every data point must be uniquely identified by locational coordinates of sufficient resolution to accomplish the objectives of the survey. Where possible, a cartesian (X,Y) grid should be established to provide these coordinates.

7.1.3 Electromagnetic Induction Measurements. At a given site grid location, the specified orientation of the apparatus is established, i.e., with the axis of the coils either parallel or perpendicular to the direction of the survey line. The meter reading is recorded and the apparatus moved to the next site grid location.

For the dual-coil method, both the intercoil spacing and coplanarity of the coils must be established prior to recording the data. Surveys are normally conducted with the coil axes horizontal and at right angles to the survey direction.

EMI profiles can be accomplished in a continuous manner using vehicle-mounted equipment and strip chart or digital recorders. Location information must be appended by tic marks or voice-over and some means provided to reference written field logs in a consistent manner.

## 7.2 Data Format

7.2.1 General. Information obtained during an EMI survey should be presented according to a standard data format, using standardized data sheets with original field entries. As a minimum, this should include the following information:

- Project, task, site, and location identification;
- Company or organization;
- Date (and time, if applicable);
- Operator's name and signature;
- Method/technique identification;
- Instrument make, model, serial number, and calibration date/frequency (if applicable);
- Coil type and configuration;

- Line or site grid location(s);
- Weather and site conditions and temperatures;
- Identification of relevant calibration and QC data; and
- Data for each sounding or profile should be recorded on a single sheet, if possible.
- Presence and location of cultural or topographic features which could affect the data.

7.2.2 Survey data. Survey data should include, in a tabular format, the following information:

- Coil location, per the survey plan;
- Coil spacing and configuration (unless specified in the heading); and
- Meter reading in millimhos per meter (quadrature phase) or parts per thousand (in-phase).

Special precautions to systematize and preserve data will be required for data that are recorded continuously on strip charts, magnetic tape, or in the memory of a recording device (such as an Omnidata Polycorder). Identifying header information must be recorded directly on the chart, tape, or file. Strip charts should be permanently affixed to the field logbook. Magnetic tapes or memory devices should be downloaded at least daily and hardcopy obtained. The original hard copy of output should be similarly secured/stored.

## 8.0 DATA INTERPRETATION

Corrections may be applied to EMI data for accuracy and drift, variation in location from pre-established coordinates, changes in scale, and nonlinearities associated with high conductivity values. In all cases, such corrections must be fully supported by data originally recorded or annotated in the field. Profile data along traverses are obtained as plots of conductivity versus distance. Parallel traverse data may be combined to provide conductivity contour maps of a site. Two or more profiles at different sampling depths, as well as sounding data at a given location, provide information on the relative conductivities of shallow and deeper layers. Contour plots can provide valuable information on the extent and direction of groundwater flow and contaminant transport.

Detailed comparison of EMI sounding measurements with layer models of the site can be made. This type of interpretation has been used at sites with relatively simple, uniform geohydrology to determine overburden—bedrock spatial and depth relationships. In some cases, very detailed interpretations, including aquifer flow properties, location of permeable zones, and interaquifer transfer, are possible.

## 9.0 SURVEY DESIGN

### 9.1 Prerequisites

Appropriate planning of EMI surveys requires at least a basic understanding of general site features and geohydrologic characteristics, including the probable variability in conditions. A statement of work should be generated that describes, in as much detail as possible, the known site conditions that may affect subsurface conductivity and the objectives of proposed survey efforts. The type and degree of data interpretation and the desired format for data presentation should be specified if possible.

### 9.2 Instrument Selection

For a given exploration target, the selection of the proper EMI instrument will primarily depend on the target depth. In general, the sampling depth of an EMI instrument is about 1.5 times the coil spacing. Some EMI instruments have adjustable coil orientations which allow the exploration depth to be somewhat modified by altering the orientation of the coils. For example, the Geonics EM-31 (coil spacing of 12 feet) has an exploration depth of about 0-15 feet in the horizontal dipole orientation and 5-20 feet in the vertical dipole orientation. Since fixed coil instruments do not generally offer coil spacing much greater than 12 feet, exploration targets greater than 20 feet deep require the use of a variable coil instrument.

### 9.3 Profiling vs Sounding

If it is necessary to determine the location and lateral extent of an exploration target (or targets), then the profiling method is indicated. If the survey needs to determine the changes of conductivity with depth (stratigraphic characterization) or the depths of conductivity changes, then EMI soundings are indicated.

### 9.4 Grid Design

The survey grid should be designed such that there are sufficient measurement points to adequately locate and define the distribution and extent of the exploration targets. For convenience, an orthogonal sampling grid, relative to a known location, is usually established over the survey area and the sampling points located by their grid coordinates (x,y). This is a convenient method for recording the data in an organized manner, for graphically displaying the data, and for ease of locating sampling points. Conductivity measurements are then taken at regular intervals along the gridlines, commonly every 5, 10, 20, or 50 feet - depending on the size of the exploration targets.

### 9.5 Work Planning and Scheduling

Conductivity surveys may be performed concurrently with field geotechnical investigations. In this case, on-site interpretation of data may provide real-time guidance for well drilling, sampling, or testing activities. Ideally, however, the geophysical surveys should be conducted in advance, allowing sufficient time for data interpretation and use of the results in planning other field exercises.

The time and effort required by conductivity surveys vary greatly depending on the site-specific objectives and site conditions. Typically, 1,000–10,000 feet of EMI profiling or 20-200 EMI soundings can be accomplished per day by a two-person team. Data reduction and interpretation will require at least an equivalent amount of time to the field

work. Weather conditions, terrain, and obstructive site features cause considerable variability in these estimates.

## 10.0 QUALITY CONTROL

### 10.1 General

Geophysical surveys are subject to misapplication, erroneous interpretations, and use of incomplete or inadequate data. All of these avoidable errors can severely impact both the cost of subsequent site investigations and the validity of the site characterization. This susceptibility to misuse and potential for negative impact demands in assurance that appropriate quality control measures have been implemented. Quality control aspects common to most types of geophysical field programs are as follows:

- Program management personnel with technical expertise in preparing of statements of work, reviewing of proposals, work plans and reports, and technical supervision of subcontracts and field-related programs.
- Insistence on a defined scope of work, clear specifications, and data validation procedures.
- Requirement of a field quality control program.
- Appropriate justification before rejection of data points from a data set. Field data sheets should contain all observed data and the conditions that could impact data validity.
- Field data should be recorded in permanent ink in a bound logbook and each page signed and dated by the operator. Original unaltered logbooks should be retained in the site file.
- Proper calibration of instrumentation. In general, the objectives of geophysical surveys can be met by relative measurements across an area or with depth and , therefore, absolute calibration is of lesser importance than precision of measurements. However, a properly calibrated instrument provides an added measure of data validity. Furthermore, proper calibration permits correlation and comparison of the associated data with site features and geohydrologic characteristics not evident at the time of the field effort.
- An evaluation should be made of noise, interferences, and obstructions at a site. Such measurements, inferences, and explanations should be recorded in the field. These real-time quality control procedures aid field personnel in correction of noise sources over which they have control, in validating suspected external sources, and in early detection of problems that may jeopardize the survey objectives.

### 10.2 Instrument Quality Control

10.2.1 Calibration. EMI instruments are calibrated by the manufacturer over massive rock outcrops of known characteristic that are used as a geologic standard to measure the absolute conductivity over a uniform section of earth. The EMI apparatus should be maintained in calibration by the user, by noting drift in the readings at a stable "secondary standard" site. A secondary standard site is a location established in the field that is used to check the accuracy (calibration of the instrument and the drift (precision of the instrument. A secondary standard site is a location that the user of the instrument uses daily on large projects, much the same way the manufacturer uses massive rock outcrops for precision and accuracy determination.

Unacceptable drift or erratic operation shall be corrected by replacement with an instrument in proper working order. Values that are obtained from measurements over the stable secondary standard site that vary by more than 10%–15% are considered to be unacceptable drift.

10.2.2 Daily Quality Control. All aspects of the daily quality control measures for resistivity measurements apply also to EMI measurements. Repeated periodic measurements (at least three times a day) should be made at one or more locations and orientations at the site to determine the precision of measurements and to detect instrument drift.

## 11.0 HEALTH AND SAFETY CONSIDERATIONS

### 11.1 General

All procedures for hazardous waste site entrance, traverse, and egress that apply to general field operations also apply to conduct of geophysical surveys. Conductivity surveys depend on traverse of the site on foot or in vehicles, and there are extended periods during which personnel are subject to adverse environments at the site. Geophysics survey personnel will adhere to the guidelines of the Site Health and Safety Plan for field activities.

The geophysical methods discussed herein do not require extremely strenuous activity, and exposure to heat or cold is similar to that during other field activities. Extreme weather conditions will have adverse effects on the time required to obtain validated data, there by increasing the duration of personal exposure to the elements and to hazardous site influences.

### 11.2 Explosive Ordnance Disposal (EOD)

For surveys in areas where ordnance disposal or the presence of ordnance is suspected, EOD personnel will clear site access and survey areas prior to survey activities as per the Health and Safety Plan. The EMI methods described here involve the transmission of a weak electromagnetic signal whose characteristics are instrument dependent. Variable coil instruments generally emit an electromagnetic signal of 1-10 watts strength at variable frequencies ranging from 6.5-0.4 kHz (for the Geonics EM-34). Fixed coil instruments emit signals of about 0.5-1.2 watts strength at frequencies from 9-15 kHz (for the Geonics EM-31 and EM-38). These signal strengths are maximum strengths measured at the transmitting coil and fall off rapidly with depth in the subsurface. It is possible that this signal may not be safely broadcast in the vicinity of certain types of (live) ordnance

detonators. The individual EMI instruments proposed for the survey(s) will be cleared for use, for each site where ordnance may be present, by the facility Health and Safety officer.

## 12.0 POTENTIAL PROBLEMS

EMI surveys are geophysical methods that, although standardized and frequently applied, are subject to a wide variety of problems. Problems can be expected to arise in the following areas:

12.1 **Planning and Execution.** Rarely is a survey accomplished exactly according to the original plan. Site features not previously specified and myriad other variations can occur that force changes in the details of the approach. However, the data quality objectives of the survey, the general methodology, the amount of data required, and the degree of data interpretation requested should remain unchanged. Project work scopes should be written with some degree of latitude to allow a change in plans whenever justified.

12.2 **Noise and Interferences.** Measurements can be affected severely both by natural and by man-made sources of electrical and electromagnetic noise. Nearby power lines, stray ground currents, and atmospheric discharges adversely effect both types of surveys. Large masses of buried metal, fences, railroad tracks and underground pipes or cables can strongly distort measurements and reduce instrument sensitivity to features of interest. These problems generally can be accounted for or overcome but must be recognized early in the program so that appropriate avoidance measures can be implemented. Known or suspected sources of interference should be included in the initial planning for a project.

12.3 **Weather Conditions.** It is possible to conduct the surveys under almost any conditions that permit traverse of the site. However, snow cover, standing water, heavy rainfall, or thoroughly saturated surface soils may severely restrict the ability to meet project objectives and schedules. Scheduling contingencies should be included whenever possible, especially during periods when inclement weather is expected.

12.4 **Technical Difficulties.** Preventable difficulties include equipment malfunction or misapplication; poor operator training, and lack of applications experience. Other difficulties may arise because the behavior of the site is not as initially conceptualized. The effect of these problems can be minimized by early recognition of their cause and severity and responsive and responsible technical management. Interim, real-time scrutiny of the data by the site geophysicist and management personnel is essential. The geophysical subcontractor must be responsive regarding equipment replacement, repair, or changes in personnel. The site manager and the site geologist should be cognizant of technical difficulties beyond the control of the field personnel and should recognize the need to change plans, field personnel, or cancel a survey, as appropriate.

## 13.0 REFERENCES

Good discussions of various survey techniques and applications are found in the following references.

Benson, Richard C., Robert A. Glaccum and Michael R. Noel, 1982. Geophysical Techniques for Sensing Buried Wastes and Waste Migration, Technos, Inc., Miami, FL., Contract No. 68-03-3050, USEPA Environmental Monitoring Systems Laboratory, Las Vegas, NV.

Costello, Robert L., 1980. Identification and Description of Geophysical Techniques, Report No. DRXTH-TE-CR-80084, US Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground,MD; Defense Technical Information System No. ADA 123939.

McKown, G.L., G.A. Sandness and G.W. Dawson, 1980. Detection and Identification of Buried Waste and Munitions, Proceedings of the 11th American Defense Preparedness Association Environmental Systems Symposium, Arlington, VA.

Ward, Stanley H., ed., 1990. Volume I - Review and Tutorial, Geotechnical and Environmental Geophysics, Society of Exploration Geophysicists Investigations in Geophysics No. 5.; SEG, Tulsa, OK.

Ward, Stanley H., ed., 1990. Volume II - Environmental and Groundwater, Geotechnical and Environmental Geophysics, Society of Exploration Geophysicists Investigations in Geophysics No. 5.; SEG, Tulsa, OK.

# STANDARD OPERATING PROCEDURE 019
# MONITORING WELL INSTALLATION

## 1.0 Scope and Application

The installation of monitoring wells is contingent upon the existing conditions at the project site. The purpose of this Standard Operating Procedure is to delineate the quality control measures required to ensure the accurate installation of monitoring wells. The applicable Cluster Work Plan or site Sampling Design Plan should be consulted for specific installation instructions. The term "monitoring wells", as used herein is defined to denote any environmental sampling well. Subsections 6.7 *et seq.* and 6.8 *et seq.* of the Field Investigation Plan, as well as sections 1, 2, 3, and 6 of the Field Sampling Plan (Appendix A) are included by reference. Example well log forms are given at the end of this SOP. Alternate, equivalent forms are acceptable.

## 2.0 Material

### 2.1 Drilling Equipment

a. Appropriately sized drill adequately equipped with augers, bits, drill stem, etc.
b. Steam cleaner and water obtained from approved source for decontaminating drilling equipment.
c. PID: Microtip HL-200 (or equivalent)
d. Water level indicator
e. Weighted Steel tape measure
f. LEL-Oxygen monitor
g. Steel drums for intrusion derived wastes (drill cuttings, contaminated PPE, decon solutions, etc.)
h. Source of approved water
i. Heavy plastic sheeting
j. Sorbent pads and/or logs

### 2.2 Well Installation Materials [18]

a. Well screen : [19]

PVC: JOHNSON (or equivalent); PVC Vee Wire Continuous slot, wire wrapped screen; 4-inch diam.; SCH 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer

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[18] Technical information on all installed materials (screens, riser pipe, filter pack, bentonite, cement, etc.) and representative samples of the proposed filter pack, bentonite powder, and bentonite pellets will be supplied to the Contracting Officer's Representative (COR).

[19] Well screen slot size and filter pack gradation will be determined from sieve analysis of aquifer materials. Screen and calling material type will be determined based on field tests of groundwater chemistry and contaminants.

Stainless Steel: JOHNSON (or equivalent); Brainless steel Vee-Wire ContinuOUs BlOt, wire wrapped screen; 304 stainless steel [20]; ASTM F480 flush threads; cleaned, wrapped, and heat sealed by manufacturer.

b. Riser pipe:

PVC: JOHNSON (or equivalent); STD. PVC; 4-inch diam.; SCH 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer

Stainless Steel: JOHNSON (or equivalent); SCH 5; 304 stainless steel; ASTM type A312 material; 4-inch diam.; cleaned, wrapped and heat sealed by manufacturer.

c. Plugs/Caps: JOHNSON (or equivalent); standard PVC or stainless steel
d. Filter pack: MORIE, 100 well gravel (or equivalent) Note: final gradation may vary as a function of the gradation of the formation (see footnote 2)
e. Fine Ottawa sand
f. Bentonite seal: BAROID, bentonite pellets (3/8-inch diam.)
g. Cement: Type II Portland Cement [21]
h. Bentonite powder: BAROID, Aquagel Gold Seal
i. Steel Protective Casing: BRAINARD-KILMAN (or equivalent) zinc-plated steel, lockable, painted.[22]
j. Geotextile: MIRAFI (or equivalent); GTF 130; non-woven; 4 oz.
k. Coarse (blanket) gravel: Crushed stone aggregate
l. Containers for purged water, as required.
m. Submersible pump or bailer of appropriate capacity, and surge block sized to fit well
n. Hach DREL 2000 portable laboratory (or equivalent)
o. Conductivity, pH, ORD, turbidity, dissolved oxygen, and temperature meters
p. Electric well sounder and measuring tape.
q. Portland Type II cement (see footnote)
r. Steel Posts (pickets), Painted (see footnote)

**2.3 Documentation**

a. Copy of appropriate Cluster Work Plan
b. Copy of Section 6 "Field Investigation Plan'
c. Copy of Appendix A 'Field Sampling Plan"
d. Copy of approved Health And Safety Plan

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[20] Unless the sum of $Cl^-$, $F^-$, and $Br^-$ is >1000ppm, in which case type 316 should be used (see also "Field Investigation Plan" Section 6.8.6 and Appendix A, "Field Sampling Plan" § 3.3.2.)

[21] If sulfates are higher than 1500ppm type IV Portland Cement will be used

[22] All painted components (protector casing, steel pickets) will be painted high-visibility orange and allowed to dry completely prior to being brought onsite.

e. Copies of well and excavation permits
f. Copies of SOPs 003, 005, 008-012, 019(this SOP), 023, 024, and 028
g. Boring log forms
h. Well completion diagram form
i. Well development form

**2.4 Geologist's personal equipment**

a. 1OX handlens
b. Unified Soil classification System chart
c. Munsell color chart
d. Sieve set (Keck model SS-81 or equivalent)
e. PPE as required by HASP

## 3.0 Procedure

### 3.1 Materials Approval

**3.1.1** Water sources for drilling, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the COR prior to arrival of the drilling equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, Cost per gallon (if applicable), dates and results associated with all available chemical analyses over the past two years, and the name and address of the analytical laboratory (if applicable).

**3.1.2** Pure sodium bentonite with no additives (bentonite) will be the only drilling fluid additive allowed, and its use must be approved by the COR prior to the arrival of the drilling equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product description, and intended use for the product.

**3.1.3** Granular Filter Pack material must be approved by the COR prior to drilling. A one-pint representative sample must be supplied to the COR. Information required includes: lithology, grain size distribution, brand name, source, processing method, and BlOt size of intended screen.

**3.1.4** Portland Type II cement will be used for grout (see footnote).

### 3.2 Drilling

**3.2.1** The objective of the selected drilling technique is To ensure that the drilling method provides representative data while minimizing subsurface contamination, cross contamination, and drilling costs. The drilling method used will be hollow stem auger or water/mud rotary[23] . No other methods will be considered as

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[23] Due to the potential for aquifer contamination and plugging, mud rotary drilling is not recommended for monitoring wells. If; however, the well is a deep monitoring well and/or is screened in a "running sand" , and the aquifer is expected to have a relatively high flow rate, then mud rotary

available without approval of EPA and MDE. The method used at a specific site will be proposed in the work plan and evaluated by the COR.

3.2.2 A Site Geologist will be present during all well drilling and installation activities and will fully characterize all tasks performed in support of these activities into the monitoring well logbook. The Site Geologist will be responsible at only one operating rig for the logging of samples, monitoring of drilling operations, recording of water losses/gains and groundwater data, preparing the boring logs and well diagrams, and recording the well installation procedures of the rig. The Site Geologist will have onsite sufficient equipment in operable condition to perform efficiently his/her duties as outlined in the Field Investigation Plan (Section 6), and the Field Sampling Plan (Appendix A), and other contractual documents. Items in the possession of each Site Geologist will include, copies of Section 6, Appendix A, the approved HASP, this SOP, a hand lens (10X), a standard color chart, grain-size chart, and a weighted (with steel or iron) steel tape long enough to measure the deepest well, heavy enough to reach that depth, and small enough to fit readily within the annulus between the well and drill casing. The Site Geologist will also have onsite, a water level measuring device, preferably electrical.

3.2.3 No lubricants will be used on downhole drilling equipment. Additives containing either lead or copper will not be allowed. In addition, polychlorinated biphenyls will not be permitted in hydraulic fluids or other fluids used in the drilling rig, pumps, or other field equipment and vehicles.

3.2.4 Surface runoff or other fluids will not be allowed to enter any boring or well during or after drilling/construction.

3.2.5 Antifreeze used to keep equipment from freezing will not contain rust inhibitors and sealants. Antifreeze is prohibited in an areas in contact with drilling fluid. The ground surface at the well site will be protected from possible coolant, fuel, and hydraulic fluid spills and/or leakage by placement of plastic sheeting with raised edges, draining into a lined catch basin large enough to contain spills and/or leakage from motors, radiators, or vehicle tanks. Sorbent pillows will be placed to catch obvious leaks from the drill rig. Sorbent logs may be used instead of, or in conjunction with a lined catch basin to contain spills.

3.2.6 An accurate measurement of the water level will be made upon encountering water in the borehole and later upon stabilization. Levels will be periodically checked throughout the course of drilling. Any unusual change in the water level in the hole such as a sudden rise of a few inches may indicate artesian pressure in a confined aquifer will be the basis for cessation of drilling. The geologist will immediately contact his/her supervisor [24]. Particular attention for such

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may be approved on a case-by-case basis.

[24] The USACE Waterways Experiment Station (WES) may also be contacted for guidance. The WES project manager is Dr. James May (601) 634-3395. He also exercises direct technical oversight of the Lauderick Creek Study Area. Mr. Danny Harrelson (601) 634-2685 has technical oversight of the Westwood Study Area. Dr. Robert Larson (601) 634-3201 has technical oversight of the Bush River Study Area.

water-level changes will be given after penetrating any clay or silt bed, regardless of thickness, which has the potential to act as a confining layer.

Anticipated depths of wells are given in well specific work plans (*e.g.* Appendix I). In case the previously defined criteria have not been met before the depth range for a given hole is reached, the geologist will stop the drilling and confer with her/his supervisor. The current boring conditions (depth, nature of the stratigraphic unit, and water-table depth) will be compared to those of other wells nearby to decide to continue drilling or to terminate and complete the well.

3.2.7 **If the well is to be installed in the surficial aquifer:** Drilling will be terminated before penetrating the basal aquitard. The basal aquitard is defined as the first 2 foot-thick clay below the water table, or below 5 feet in the case of a shallow aquifer (Field Investigation Plan, § 6.7.3)

3.2.8 **If the well is to be installed in a lower, confined aquifer:**

**3.2.8.1** Penetrations of aquifers located lower than the watertable aquifer will be limited to avoid cross-contamination.

**3.2.8.2** Placement of new upper confined aquifer wells will be initially limited to those areas where contamination has been confirmed.

**3.2.8.3** The location of upper confined aquifer wells will be based upon the findings of the water-table aquifer investigation. Areas of known contamination will be targeted for installing upper confined aquifer wells for the purposes of delineating vertical contamination.

**3.2.8.4** Where possible, upper-confined aquifer wells will be located such that they afford triangulation with other wells within the same aquifer to allow for a determination of ground-water flow direction.

**3.2.8.5** Some upper-confined aquifer wells will be installed approximately 10-15 ft from water-table wells to enable the accurate assessment of vertical hydraulic gradients. If the direction of ground-water flow is known, wells within a group will be located sidegradient of each other.

**3.2.8.6** The boring will be advanced until the base of the surficial aquifer is reached (see § 3.2.7).

**3.2.8.7** An outer, surface casing will be set 2 to 5 ft into the confining layer to minimize the potential for cross-contamination from the unconfined aquifer during drilling activities.

**3.2.8.8** The surface casing will be driven into the confining bed and grouted into place. A grout plug at least 2 feet thick will be tremied into the bottom of the surface casing. The grout will be permitted to cure for 24 hours. All drilling fluids within the surface calling will then be removed, and the casing will be flushed with clean potable water.

**3.2.8.9** The drilling equipment will be decontaminated, a smaller bit or auger selected, and the hole will be continued through the grout plug into the confined aquifer.

**3.2.8.10** If deeper aquifers are to be screened, repeat preceding steps until total depth is reached.

**3.2.9** <u>If DNAPL contamination is detected during drilling</u>, the well will be terminated and completed at the base of the aquifer. Drilling will not continue through the confining unit.

**3.2.9.1** Stainless steel screens will be used in DNAPL wells. Screen size selection will be according to criteria set forth in § 3.4.3.2 (below). The formation grain size will be multiplied by the higher factor (6) to determine filterpack grain size. This will ensure that the filterpack is sufficiently coarse to permit DNAPL to pass freely from the formation into the coarser filterpack, then into the open well (Cohen and Mercer, 1993).

**3.2.9.2** DNAPL sampling cups are prohibited. The well screen will be capped, and set 0.3 ft (0.5 ft. max.) into the top of the confining bed and rest on the bottom of the hole or bentonite backfill (if used). No sand will be placed below the screen.

**3.2.9.3** The remainder of the well installation and completion will be accomplished according to section 3.4.

## 3.3 Logging

**3.3.1** All borings for monitoring wells will be logged by a geologist. Logs will be recorded in a field logbook and/or a boring log. If the information is recorded in a logbook, it will be transferred to Boring Log Forms on a daily basis. Field notes are to include, as a minimum:

a. Boring Number
b. Material Description (as discussed below)
c. Weather conditions
d. Evidence of Contamination
e. Water Conditions (including measured water levels)
f. Daily Drilling Footage and Quantities (for billing purposes)
g. Notations on Man-Placed Materials
h. Drilling Method and Bore Hole Diameter
i. Any Deviations from Established Field Plans
i. Blow Counts for Standard Penetration Tests
k. Core and Split-Spoon Recoveries

**3.3.2** Material description for soil samples must include:

a. Classification
b. Unified Soil Classification Symbol
c. Secondary Components and Estimated Percentages
d. Color
e. Plasticity

f. Consistency
g. Density
h. Moisture Content
i. Texture/Fabric/Bedding and Orientation
j. Grain Angularity
k. Depositional Environment and Formation
l. Incidental odors
m. OVA reading(s)
n. Staining

3.3.3 Material description for rock samples must include:

a. Classification
b. Lithologic Characteristics
c. Bedding/Banding Characteristics
d. Color
e. Hardness
f. Degree of Cementation
g. Texture
h. Structure and Orientation
i. Degree of Weathering
i. Solution or Void Conditions
k. Primary and Secondary Permeability
l. Sample Recovery
m. Incidental odors
n. OVA reading(s)
o. Staining

See also SOP003 and SOP016 for details on logbook entries.

**3.4 Well Construction and Installation**

After the hole is drilled and logged, backfill hole as required for proper screen placement. The integrity of the aquitard will be restored by placing a bentonite plug of an appropriate thickness, either to the top of the aquitard (normal well installation) or to within 0.3 ft. of the top of the aqitard (DNAPL well). Aquifer fill will be clean filter pack.

Normal screen placement for the water table (surficial) aquifer will be with 2 ft. of the screen extending above the static water level. The bottom of the screen will rest no more than 6 in. from the bottom of the hole or backfill material, whichever is applicable.

Note: the end cap in DNAPL wells will rest on the bottom of the bottom of the hole, or bentonite backfill if applicable. (see § 3.2.9.2 above)

Screen placement for a confined aquifer well will normally be at the top of the confined aquifer.

3.4.1 Screen lengths will not normally exceed 10 feet. If it appears advantageous in a given situation (e.a. to screen an entire aquifer which is thicker than 10 feet, approval must be sought on a case-by-case basis from MDE and EPA. Otherwise, wells will be screened as follows:

| Thickness of Aquifer: | Action: |
|---|---|
| < 10' | Screen entire aquifer |
| > 10' < 30' | screen top 10' consider vertically nested well cluster |
| > 30' | install vertically nested well cluster |

**3.4.2** The installation of monitoring wells in uncased or partially cased holes will begin within 12 hours of completion of drilling, or if the hole is to be logged, within 12 hours of well logging, and within 48 hours for holes fully cased with temporary drill casings. once installation hall begun, work will continue until the well has been grouted and the drill casing has been removed. Exceptions MUST be requested in writing by the contractor to the Contracting officer's Representative prior to drilling. Unscheduled delays attributable to unforeseeable site occurrences will not require advance approval.

**3.4.3** Well screens, casings, and fittings will conform to National Sanitation Foundation Standard 14 or American Society for Testing and Materials (ASTM) equivalent for potable water usage. These materials will bear the appropriate rating logo. If the logos are not present, a written statement from the manufacturer/supplier stating that the materials contain the appropriate rating must be obtained. Material used will be new and essentially chemically inert to the site environment.

**3.4.3.1** Well screen and casing should be inert with respect to the ground water; therefore, the selection Of screen and casing material will be based on select field tests of aquifer chemistry and potential contaminants. The screen will be capped without sediment trap or DNAPL sampling cup, and lowered into the hole. The well casing will be pre- cut to extend 2 to 2.5 ft above the ground surface. Prior to placement of the last piece of well casing, a notch or other permanent reference point will be cut, filed, or scribed into the top edge of the casing.

**3.4.3.2** Screen slot size will be appropriately sized to retain 90 to 100% of the filter pack material, the size of which will be determined by sieve analysis of formational material (See § 3.4.3.2).

**3.4.3.3** The tops of all well casing will be capped with covers composed of materials compatible with the products used in the well installation. Caps may either be vented, or a telescopic fit, constructed to preclude binding to the well casing caused by tightness of fit, unclean surfaces, or weather conditions. In either case it should be secure enough to preclude the introduction of foreign material into the well, yet allow pressure equalization between the well and the atmosphere.

**3.4.4** Filter pack material will be tremied into place, and lightly tamped and leveled. Filter pack will extend from the bottom of the hole to a height of 1-2 ft above the

top of the screen. The filter pack will be capped with 1 ft of fine (Ottawa) sand to prevent the bentonite seal from infiltrating the filter pack.

**3.4.4.1** Granular filter packs will be chemically and texturally clean, inert, and siliceous.

**3.4.4.2** Filter pack grain size will be based on formation grain-size analysis. The D30 (70% retained) sieve size multiplied by a factor of not less than 3 nor greater than 6 will be used to determine the appropriate grain size.

**3.4.4.3** Calculations regarding filter pack volumes will be entered into the Field Logbook along with any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10% exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Logbook.

**3.4.5** Bentonite seals will be no less than two feet nor more than five feet thick as measured immediately after placement. The normal installation will include a five foot seal. Thinner seals may be used in special cases as defined in Section 3.12 of Appendix A. The final depth to the top of the bentonite seal will be measured and recorded.

**3.4.6 GROUT**

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, type II)(see footnote)
- 0.4 to 1 part (max.)(2-5%) bentonite
- 8-gallons (max.) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

**3.4.6.1** All grout material will be combined in an above-ground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement.

**3.4.6.2** Grout placement will be performed using a commercially available grout pump and a rigid, side discharge tremie pipe.

**3.4.6.3** The following will be noted in the Field Logbook: 1) calculations of predicted grout volumes, 2) exact amounts of cement, bentonite, and water used in mixing grout, c) actual volume of grout placed in the hole, d) any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10% exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Logbook.

**3.4.7** Well protective casings will be installed around all monitoring wells on the same day as the initial grout placement around the well. Any annulus formed between the outside of the protective casing and the borehole will be filled to ground surface with cement.

3.4.8 The construction of each well will be depicted as built in a well construction diagram. The diagram will be attached to the boring log and will graphically denote:

a. Screen location, length
b. Joint location
c. Granular filter pack
d. Seal
e. Grout
f. Cave-in
g. Centralizers
h. Height of riser
i. Protective casing detail

**3.5 Monitoring well completion**

3.5.1 Assemble appropriate decontaminated lengths of pipe and screen. Make sure these are clean and free of grease, soil, and residue.

3.5.2 Lower each section of pipe and screen into the borehole, one at a time, screwing each section securely into the section below it. No grease, lubricant, polytetrafluoroethelyne (PTFE) tape or glue, may be used in joining the pipe and screen sections.

3.5.3 Centralizers will be used every 50 ft below the top 50 ft. (except within screened interval and bentonite seal). Centralizer material will be PVC, PTFE, or stainless steel. Determination of centralizer material will be based on the same criteria as screen and calling selection (see Field Investigation Plan, Section 6.8)

3.5.4 Prior to installation, cut the riser so that it will extend approximately 2-2.5 ft. above grade. Notch, file, or otherwise permanently mark a reference point on the top of the casing. All pipe cuts MUST be square to ensure that the elevation between the highest and lowest point of the well casing is less than or equal to 0.02 ft.

3.5.5 When the well is set to the bottom of the hole, temporarily place a cap on top of the pipe to keep the well interior clean.

3.5.6 Place the appropriate filter pack into using a tremie pipe. Monitor the rise annulus with a weighted tape to assure that bridging is not occurring.

3.5.7 After the pack is in place, wait three to five minutes for the material to settle, tamp and level a capped PVC pipe, and check its depth weighted steel tape.

3.5.8 Add a 1-2 foot cap of fine-grained (Ottawa) sand to prevent infiltration of the filterpack by overlying bentonite seal.

3.5.9 Install the bentonite seal (2 ft to 5 ft thick) by dropping bentonite pellets into the hole gradually. If the well is deeper than 30 feet, a tremie pipe will be used to place the pellets. Tamp and level the pellets with a capped PVC pipe, and check depth with a weighted tape as above.

**3.5.10** Wait a for the pellets to hydrate and swell. Hydration times will be determined by field test or by manufacturer's instructions. Normally this will be 30 to 60 minutes. Document the hydration time in the field notebook. If the pellets are above the water level in the hole, add several buckets of clean water to the boring. Document the amount of water added to the hole.

**3.5.11** Mix an appropriate cement-bentonite slurry (§ 3.4.6). Be sure the mixture is thoroughly mixed and as thick as is practicable.

**3.5.12** Lower a side discharge tremie pipe into the annulus to the level of the pellet seal.

**3.5.13** Pump the grout slurry into the annulus while withdrawing the tremie pipe and temporary casing.

**3.5.14** Stop the grout fill at 5 feet below the ground surface. Allow to cure for not less than 12 hours. If grout settles more than 6-in., add grout to bring level back up to within 5-ft. of ground surface. Place approximately 2 ft. of bentonite pellets (minimum 0.5 ft) in annulus. Seat the protective casing in the bentonite seal, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Fill inner annulus (between well casing and protective casing) with bentonite pellets to the level of the ground surface. Cover bentonite pellets with 1 ft. of clean granular material (coarse sand or pea gravel filter pack). Fill the outer annulus (between the protective casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing. Lock the cap.

-or-

**3.5.15** Continue the grout fill to the ground surface. Seat the protective casing in the grout, allowing no more than 0.2 - ft between the top of the well casing and the bottom of the protective casing cap. Lock the cap.

-and-

**3.5.16** Allow the grout slurry to set overnight.

**3.5.17** Fill the outer annulus (between the casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing.

**3.5.18** Slope the ground surface away from the casing for a distance of two feet, at a rate of no less than 1 inch in two feet. Surface this sloping pad with a geotextile mat covered by 3 in. of coarse gravel.

**3.5.19** Set pre-painted protective steel pickets (3 or 4) evenly around and 4 feet out from well.

## 3.6 Well Development

**3.6.1** Well development is the process by which drilling fluids, solids, and other mobile particulates within the vicinity of the newly installed monitoring well have been removed while restoring the aquifer hydraulic conductivity. Development

corrects any damage to or clogging of the aquifer caused by drilling, increases the porosity of the aquifer in the vicinity of the well, and stabilizes the formation and filter pack sands around the well screen.

**3.6.2** Well development will be initiated after 48 consecutive hours but no longer than 7 calendar days following grouting and.or placement Of surface protection. The record of well development will be submitted to the Contracting Officer's Representative within three working days after well development is completed.

**3.6.3** Two well development techniques - over pumping and surging will be employed in tandem. over pumping is simply pumping the well at a rate higher than recharge. Surging is the operation of a plunger up and down within the well casing similar to a piston in a cylinder.

**3.6.4** Materials Required

a. Well Development Form
b. Boring Log and Well Completion Diagram for the well
c. Submersible pump or bailer of appropriate capacity, and surge block
d. Conductivity, pH, ORD, turbidity, dissolved oxygen, and temperature meters
e. Electric well sounder and measuring tape.
f. Containers for purged water, if required.

**3.6.5 Summary of Procedures and Data Requirements.**

**3.6.5.1** Pump or bail the well to ensure that water flows into it, and to remove some of the fine materials from the well. Removal of a minimum of one equivalent volume (EV) is recommended at this point. The rate of removal should be high enough to stress the well by lowering the water level to approximately 1/2 its original level.

**3.6.5.2** Slowly lower a close-fitting surge block into the well until it rests below the static water level, but above the screened interval. (Note: this latter is not required in the case of an LNAPL well.)

**3.6.5.3** Begin a gentle surging motion which will allow any material blocking the screen to break up, go into suspension, and move into the well. Continue surging for 5-10 minutes, remove surge block, and pump or bail the well, rapidly removing at least one EV.

**3.6.5.4** Repeat previous step at successively lower levels within the well screen until the bottom of the well is reached. Note that development should always begin above, or at the top of, the screen and move progressively downward to prevent the surge block from becoming sand locked in the well casing. As development progresses, successive surging can be more vigorous and of longer duration as long as the amount of sediment in the screen is kept to a minimum.

**3.6.5.5** Development is expected to take at least 2 hours in a small well installed in a clean sand, and may last several days in large wells, or in wells set in silts with low permeabilities.

**3.6.5.6** Development will continue until little or no sediment can be pulled into the well, and target values for parameters listed in 3.6.4.9 (e.) (below) are met.

**3.6.5.7** At a minimum, development will remove 3 to 5 EV, plus 3 to 5 times the amount of fluid lost during drilling, and 3 to 5 times the volume used in filter pack placement.

**3.6.5.8** All water removed must be disposed of as directed by the Sampling Design Plan.

**3.6.5.9** Record all data as required on a Well Development Record Form (see example), which is made a part of the complete Well Record. These data include:

a. Depths and dimensions of the well, the casing, and the screen, obtained from the Well Diagram.

b. Water losses and uses during drilling, obtained from the boring log for the well.

c. Water contained in the well, obtained from calculations using the depth of the water column and the well radius, plus the radius and height of the filter pack and an assumed 30% porosity.

d. Measurements of the following indicator parameters: turbidity, pH, conductivity, oxidation-reduction (ORD, Redox) potential, dissolved oxygen, and temperature before, twice during, and after development.

e. Target values for the indicator parameters listed above are as follows: pH - stabilize, conductivity - stabilize, ORD - stabilize, DO stabilize, temperature - stabilize, turbidity NTU 5 or stabilize

f. Notes on characteristics of the development water.

g. Data on the equipment and technique Used for development.

h. Estimated recharge rate and rate/quantity of water removal during development. (See SOP 013 section 3.2.)

**3.7** Refer to SOP003 (Field Logbook), 005 (Decontamination), 008 - 012 and 036 038 (Instrumentation for Groundwater Parameters).

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during well development activities.

## 6.0 References

Aller, Linda, *et al.*, 1989. Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, National Water Well Association

Cohen, Robert M., and Mercer, James W. 1993. DNAPL Site Evaluation, CRC Press, Inc.

COMAR 26.04.04 Well Construction

EPA Groundwater Handbook 1989

Nielsen, David M., 1993. Correct Well Design Improves Monitoring, in "Environmental Protection", Vol.4, No.7, July, 1993.

USATHAMA. 1987. Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition. and Reports, March 1987.

ASTM D 2487-92 Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)

ASTM D 5092-90 Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers

WELL DESIGNATION: _____ DATE(S) OF INSTALLATION: ____/____/____

SITE GEOLOGIST: _____ DEVELOPMENT DATE(S): ____/____/____

STATIC WATER LEVELS BEFORE AND AFTER DEVELOPMENT * :

BEFORE _____ DATE _____ 24 HR. AFTER _____ DATE _____

DEPTH TO SEDIMENT BEFORE AND AFTER DEVELOPMENT * :

BEFORE _____ DATE _____ 24 HR. AFTER _____ DATE _____

DEPTH TO WELL BOTTOM *: _____ SCREEN LENGTH _____

HEIGHT OF WELL CASING ABOVE GROUND SURFACE: _____

QUANTITY OF MUD/WATER:

| | | |
|---|---|---|
| LOST DURING DRILLING | (+)_____ | gallons |
| REMOVED PRIOR TO WELL INSERTION | (-)_____ | gallons |
| LOST DURING THICK FLUID DISPLACEMENT | (+)_____ | gallons |
| ADDED DURING FILTER PACK PLACEMENT | (+)_____ | gallons |
| TOTAL LOSSES | _____ | gallons |

(a) Water column ht. (ft.) _____ (b) Well radius (in.)_____

(c) Screen length (ft.) _____ (d) Borehole radius (in.)_____

(e) QUANTITY OF FLUID STANDING IN WELL

$(12 * a * \pi * b^2 * 0.0043) =$

_____ gallons
(Show Calculation)

(f) QUANTITY OF FLUID IN ANNULUS

$[12 * c * \pi 8(d^2 - b^2) * 0.0043 * 0.30]$

_____gallons
(Show Calculation)

DEVELOPMENT VOLUME = (5 * TOTAL LOSSES) + [5 * (e + f)] = _____ gallons
(Show Calculation)

* **ALL DEPTHS MEASURED FROM TOP OF WELL CASING**

# EXAMPLE WELL DEVELOPMENT RECORD
(PAGE 2 OF 2)

WELL DESIGNATION _____ DATE(S) OF DEVELOPMENT:
____/____/____

TYPE AND SIZE OF PUMP: _____

TYPE AND SIZE OF BAILER: _____

DESCRIPTION OF SURGE TECHNIQUE:
_____
_____

RECORD OF DEVELOPMENT

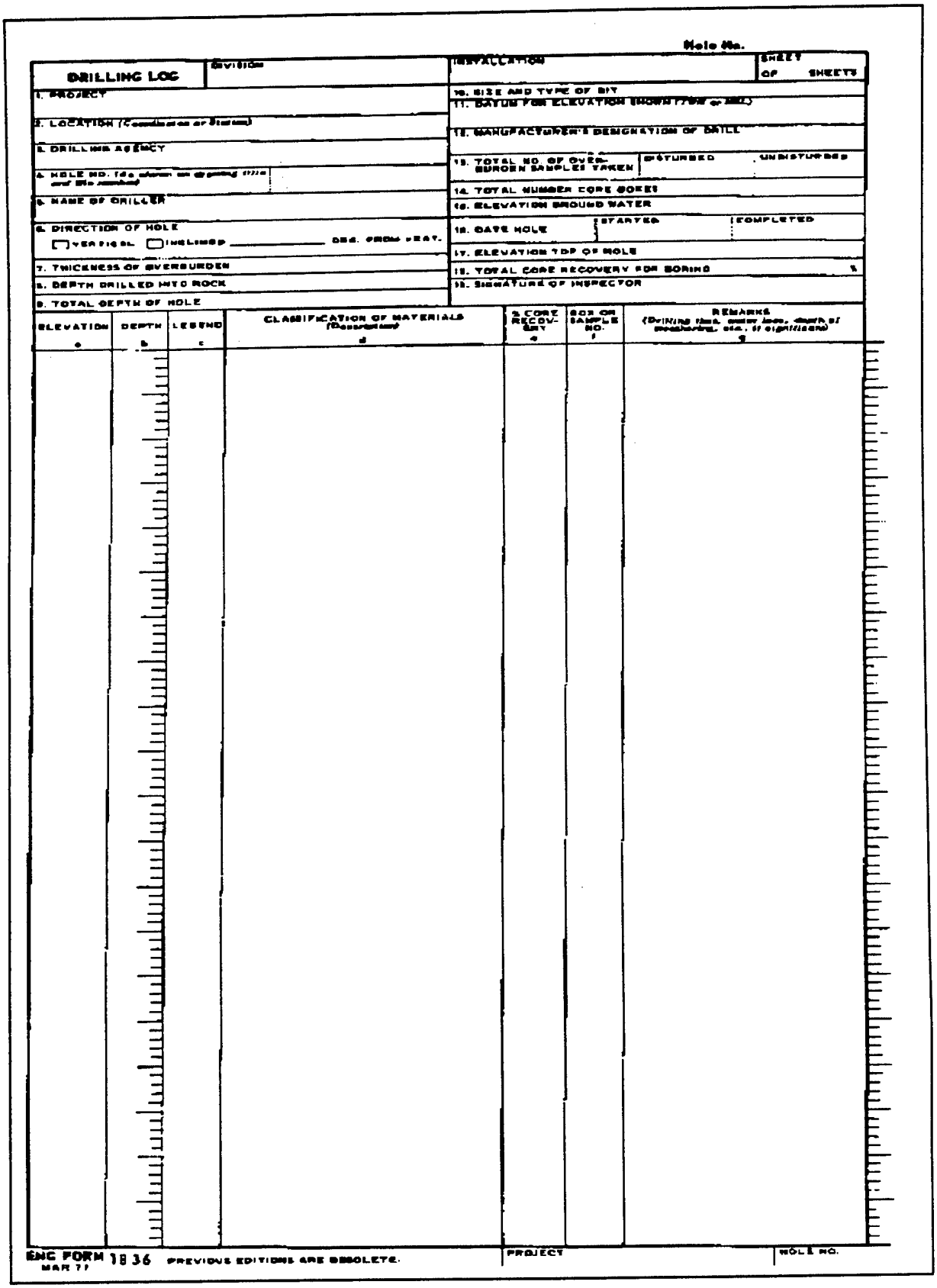
| DATE & TIME | QUANTITY REMOVED | TIME REQ'D | pH | Cond | Temp | ORD | Turb | DO | Character of water (color / clarity / odor / partic.) |
|---|---|---|---|---|---|---|---|---|---|
| (before) | | | | | | | | | |
| (during) | | | | | | | | | |
| (during) | | | | | | | | | |
| (during | | | | | | | | | |
| (after) | | | | | | | | | |

TYPICAL PUMPING RATE _____ GAL./HR. EST. RECHARGE RATE _____

TOTAL QUANTITY OF WATER REMOVED _____ TIME REQUIRED _____

REMARKS _____
_____
_____

SIGNATURE OF SITE GEOLOGIST _____



Hole No.

DRILLING LOG | DIVISION | INSTALLATION | SHEET OF SHEETS

1. PROJECT
2. LOCATION (Coordinates or Station)
3. DRILLING AGENCY
4. HOLE NO. (As shown on drawing title and file number)
5. NAME OF DRILLER
6. DIRECTION OF HOLE ☐ VERTICAL ☐ INCLINED ____ DEG. FROM VERT.
7. THICKNESS OF OVERBURDEN
8. DEPTH DRILLED INTO ROCK
9. TOTAL DEPTH OF HOLE
10. SIZE AND TYPE OF BIT
11. DATUM FOR ELEVATION SHOWN (TBM or MSL)
12. MANUFACTURER'S DESIGNATION OF DRILL
13. TOTAL NO. OF OVER-BURDEN SAMPLES TAKEN | DISTURBED | UNDISTURBED
14. TOTAL NUMBER CORE BOXES
15. ELEVATION GROUND WATER
16. DATE HOLE | STARTED | COMPLETED
17. ELEVATION TOP OF HOLE
18. TOTAL CORE RECOVERY FOR BORING %
19. SIGNATURE OF INSPECTOR

| ELEVATION a | DEPTH b | LEGEND c | CLASSIFICATION OF MATERIALS (Description) d | % CORE RECOV-ERY e | BOX OR SAMPLE NO. f | REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g |
|---|---|---|---|---|---|---|
| | | | | | | |

ENG FORM 1836 MAR 77 PREVIOUS EDITIONS ARE OBSOLETE. | PROJECT | HOLE NO.

# STANDARD OPERATING PROCEDURE 020
# ACTIVE SOIL GAS SAMPLING

## 1.0 Scope and Application

This standard operating procedure is applicable when conducting soil gas sampling. A soil gas survey is an effective screening tool in locating areas contaminated with volatile organic compounds.

## 2.0 Material

a. Probe set, including probe jack
b. Rotary hammer with 1"x36" drill bit
c. Disposable shield points
d. 3/16" o.d. polyethylene tubing
e. Extension cord
f. Portable generator or other power source
g. 1L Tedlar bags [25]
h. Sample labels
i. Vacuum box and vacuum pump
j. Tygon tubing for vacuum box
k. Clean sand
l. Powdered bentonite
m. Two measuring cups
n. Tools: vise grips, 3/4" and ⅝" wrenches, scissors

## 3.0 Procedure

### 3.1 Soil Gas Point Installation

**3.1.1** Assemble clean probe sections to the desired sampling depth.

**3.1.2** Cut polyethylene tubing to at least 1' longer than the depth of the hole.

**3.1.3** Insert one end of the tubing approximately ¼" inside of aluminum shield point. Crimp the shield point tightly around the tubing with vise grips and insert the tube and shield point inside of the clean KV probe.

**3.1.4** Using rotary drill and 36" drill bit, bore down 30" at the location desired for sampling. Be sure to clear the hole well so that soil does not fall back into hole.

**3.1.5** Drive stainless steel probe and attached shield point and polyethylene tubing down the hole with a rotary hammer to about 4', or above the saturation zone. (It is desired to obtain a sample of the soil gas, not the ground water.) If samples are needed from greater than 4', drive the steel probe with a solid tip to the

---

[25] Tedlar bags and on-site analysis is preferred. Glass vials and offsite analysis will be acceptable. An equivalent SOP for glass vials and offsite analysis will be submitted by the contractor prior to sampling. Holding times for either sample container will be kept to a minimum.

desired depth, extract, and insert a probe fitted with a disposable shield point and tubing.

**3.1.6** Extract the probe by hand or with the jack. Be sure that shield point and tubing stays in the ground and attached to the shield point.

**3.1.7** Pour ½ cup of sand down sampling hole. Gently shake the tubing to ensure that the sand settles and no bridged spaces remain.

**3.1.8** Pour ½ cup bentonite down sampling hole, add ¼ cup distilled water, add another ½ cup bentonite down hole and another ¼ cup water. Continue until bentonite seal reaches the surface.

**3.1.9** Allow at least 20 minutes before extracting sample.

**3.1.10** Take sample (see 3.2 below)

**3.1.11** Remove probe and backfill hole with bentonite.

**3.2 Soil Gas Sample Collection Using Tedlar Bags**

**3.2.1** Cut at least 1" off the end of the tubing to ensure a clean sample.

**3.2.2** Attach tubing to the vacuum box and pump.

**3.2.3** Open valve on a clean, dry Tedlar bag and attach inside the vacuum box.

**3.2.4** Close the vacuum box, close stopcock (3-way valve) between vacuum box and pump and then turn the pump on.

**3.2.5** Allow Tedlar bag to fill 90% (do not overfill bag), shut off, crimp Tygon tubing (to prevent release of sample back down hole), open stopcock, and remove Tedlar bag from box.

**3.2.5.1** If the bag is filled with air only, squeeze the air out completely to purge air that was in the tubing and sand and reattach inside the box. Repeat steps 3.2.4 and 3.2.5. Close the valve on the Tedlar bag upon removal, label it accordingly, and put it in a cool, dark area. Note: not so cool as to cause condensation.

**3.2.5.2** If Tedlar bag is filled with water and air, be sure to close valve on Tedlar bag before removing it, label the bag accordingly, and put it in a cool, dark area. Note: not so cool as to cause condensation.

**3.2.5.3** If water is pulled into the Tedlar bag, Tygon tubing inside the vacuum box must be replaced.

**3.2.6** Remove and decon probes.

**3.2.7** Repeat the above procedures for each additional soil gas point.

**3.3** Sampling with glass vials.

## 4.0 Maintenance

None

## 5.0 Field Quality Control Measures

**5.1** To ensure that the equipment is free of volatile contaminants, collect at least two QC samples per day by drawing uncontaminated air through an unused representative sampling apparatus (assembled shield point and tubing). One sample should be taken at the beginning of the day, prior to collecting any samples, the other at the end of the day, after decontaminating the equipment. Ambient air may usually be assumed to be uncontaminated. If site ambient air is assumed to be contaminated, it should be sampled for contaminant levels.

**5.2** To ensure that the analyzed samples are representative of the collected samples, and that the Tedlar bags are not losing volatile samples, spiked samples of known volatile concentration will be prepared. These samples will be stored and handled in the same manner as other field samples. Spiked samples will be the first collected and last analyzed.

Selected low level samples should also be duplicated at a different time and analyzed immediately to verify that analyte loss is not occurring.

Alternatively, samples may be analyzed in the field, using either Tedlar bags or syringe samplers to collect and transport the samples to the gas chromatograph.

**5.3** Note sampling times for each sample in field notebook and on sample bag (if bags are used).

**5.4** No more that 4 hours should elapse between sampling and analysis 15 minutes is preferable.

## 6.0 References

Posner, Judd C., and Woodfin, James; 1986; Sampling With Gas Bags I: Losses of Analyte With Time; Applied Industrial Hygene, November, 1986 pp 163-168

ASTM D 5314-93 Standard fof Soil Gas Monitoring in the Vadose Zone

# STANDARD OPERATING PROCEDURE 021
# SEDIMENT SAMPLING

## 1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure is to delineate protocols for sampling sediments. Sediments include solid matter derived from rocks or biological materials which are suspended in, or settled from, water. This procedure can be applied to the collection of sediment samples from areas of deposition such as streams, rivers, ditches, lakes, ponds and lagoons. Sediment samples indicate the amount of contamination adsorbed on sediment particles and/or the amount of wastes transported from the site. It is therefore important to collect a representative sample.

## 2.0 MATERIAL

a. Stainless steel, Polytetrafluoroethelyne (PTFE), or PTFE-lined sampling tray or bowl
b. Stainless steel or PTFE dip sampler, scoops, trowels, spoons, ladles
c. PVC pipe, 2 in. diameter
d. Hand core sediment sampler, liners (optional) and extensions
e. Pipe dredge sampler
f. Jaw type sampler
g. Sample bottles
h. Rubber boots/waders
i. Plastic sheeting
j. Utility knife
k. Rope
l. Boat
m. PPE
n. Personal flotation devices (PFDs)

## 3.0 PROCEDURE

The water content of the sediment may vary greatly. Likewise, the sediments themselves may range from very soft to dense. It may be necessary to use a variety of equipment to obtain the required samples, even at a single site.

**3.1** Upon arrival at the site, immediately set up and organize the equipment downstream (where applicable) from sampling point.

**3.1.1** Cut a section of plastic sheet approximately 6 ft. x 6 ft. Place plastic on ground to use as a clean staging area for sampling equipment.

**3.1.2** Arrange sample containers, samplers, preservatives, and decon equipment on the plastic sheet. Exercise caution not to step on, or otherwise contaminate this clean working surface.

**3.1.4** Don personal protective equipment in accordance with the site safety and health plan.

**3.1.5** Collect surface water sample.

**3.1.6** Collect sediment sample. The preferred method of collecting sediment samples will be by hand corer (section 3.2), or PVC pipe (section 3.3). If using a scoop, trowel, spoon, or ladle, refer to section 3.5. If using a dredge sampler see section 3.4.

**3.2 If using a hand corer, proceed as follows:**

**3.2.1** Ensure that the corers and (optional) liners are properly cleaned.

**3.2.2** Force the corer into the sediment with a smooth continuous motion to a depth of approximately 9 inches.

**3.2.3** Twist the corer to detach the sample; then withdraw the corer in a single smooth motion.

**3.2.4** Remove top of corer and decant excess water into a 1-L sample bottle. This water sample will be labelled and analyzed as an additional unfiltered surface water sample.

**3.2.5** Remove the nosepiece and deposit the sample onto a stainless steel, PTFE, or PTFE-lined tray.

**3.2.6** Transfer the sample into sample containers (see 3.3.6) using a stainless steel laboratory spoon (or equivalent device). The transfer equipment may be disposable to avoid decontamination costs, and the risk of cross-contamination.

**3.2.7** The top 6 inches of the core will be sampled into 3 separate containers - 2 inches per container - to ensure that an accurate chronology of contamination can be determined.[26]

**3.2.8** Ensure each container will be properly labeled, appropriate preservatives added (see SOP 039), and placed in cooler with ice packs.

**3.2.9** Decontaminate equipment according to SOP 005.

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[26] If specific data quality objectives mandate, the sample may be homogenized in bowl using sampling spoon, then samples will placed in containers, preserved (as required) and packed on ice.

3.3 **A two-inch diameter PVC pipe can be effectively used instead of a hand corer in very soft sediments as follows:**

3.3.1 Force pipe into sediment with a smooth continuous motion to a depth of approximately 9 inches

3.3.2 Cap the pipe, forming an airtight seal, to create a vacuum as it is withdrawn from the water

3.3.3 Decant excess water as in 3.2.4 above.

3.3.4 Deposit the sample onto a stainless steel or PTFE tray, and sample as in steps 3.2.5 through 3.2.7 above.

3.3.5 Decontaminate equipment according to SOP 005.

3.4 In deeper water, sampling will be accomplished from a boat.

3.4.1 **If a pipe dredge is used**, it will be thrown outward, then dragged across the bottom to collect sediment. The sampler will be emptied into a stainless steel or PTFE tray. The composite sample will be placed in the sample bottle by pouring, or through the use of a stainless steel spoon or trowel.

3.4.2 **If a jaw type bottom sampler is used**, refer to SOP 022 for details.

3.4.3 Decontaminate equipment according to SOP 005 § 3.3.2 "Solid materials samplers".

3.5 If using a scoop, trowel, spoon, or ladle, sample as follows:

3.5.1 Insert the sampling device into the material at the selected point and slowly remove the sample. Care should be taken to retain as much of the clay component as possible.

3.5.2 Transfer the sample into the appropriate container, add preservative as required in SOP 039, cap the container, and place in ice chest.

3.5.3 Decontaminate equipment according to SOP 005 § 3.3.2 "Solid materials samplers".

3.6 Refer to SOP 1-5, 16, 31, and 39.

3.7 For all samples, mark the sampling location on a site map. Photograph (optional, recommended) and describe each location, and place a numbered stake above the visible high water mark on the bank closest to the sampling location. The photographs and description must be adequate to allow the sampling station to be relocated at some future date.

3.8 Dispose of all sampling wastes in properly labelled containers.

## 4.0 MAINTENANCE

Not Applicable.

## 5.0 PRECAUTIONS

5.1 Both surface water and sediment samples are to be collected at the same location.

5.2 Take the surface water sample first (SOP 007). Sediment sampling usually results in disturbance of the sediments which may influence the analytical results of the surface water samples.

5.3 Wear gloves when collecting sediment samples. Be sure to consult the health and safety plan for the proper dermal and respiratory protection prior to collecting any samples.

5.4 Higher levels of personal protective equipment may be required by the HASP.

5.5 If sampling from a boat or near water bodies with a depth of four feet or more, the sampling team shall wear personal flotation devices (life jackets).

5.6 Collect samples first from those areas that are suspected of being the least contaminated, thus minimizing the risk of cross contamination.

5.7 Collecting sediment samples directly into sample bottles is not recommended.

## 6.0 REFERENCES

EPA/540/P-87/001, A Compendium of Superfund Field Operations Methods.

# STANDARD OPERATING PROCEDURE 022
# SEDIMENT AND BENTHIC MACROINVERTEBRATE SAMPLING WITH ECKMAN GRAB

## 1.0 Scope and Application

This standard operating procedure covers the protocol for obtaining qualitative or quantitative samples of soft sediments and macroinvertebrates inhabiting soft sediments in lakes, reservoirs, and other water bodies. The Eckman grab sampler is well suited to collecting samples in deeper (up to 100 feet) water bodies.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Materials

a. Eckman grab sampler: a box-shaped device with two scoop-like jaws.
b. Boat
c. Sample containers
d. Sieve - 500 $\mu$ (U.S. std. #30)
e. Stainless steel spoon or trowel
f. Personal protective equipment (PPE)
g. Personal flotation devices (PFDs)

## 3.0 Procedure

**3.1** Cock the sampler by raising each jaw upward into the cocked position using the attached cable and secure the cable to the catch pin located at the top of the sampler.

**3.2** Once cocked, lift the sampler overboard and lower slowly but steadily to the bottom.

**3.3** Once on the bottom, indicated by a slack line, the weighted messenger is sent down the line tripping the catch mechanism, causing the spring loaded jaws to close the bottom of the sampler, containing the sediment.

**3.4** Raise the sample at a slow but steady rate to prevent sample loss or washout.

**3.5** Once the sample is on board, empty the sample into a stainless steel, PTFE, or PTFE-lined bowl or tray for processing.

**3.5.1** If the sediment will be analyzed for VOCs, transfer the sample into the appropriate sample sontainers immediately.

**3.5.2** If the sediment will not be analyzed for VOCs, use Stainless steel spoon to thoroughly homogenize sample, then transfer sample into appropriate containers. Add preservative as directed in SOP 039. Place in ice-filled chest.

**3.5.3** If benthic macroinvertebrates are to be collected, sieve sample and transfer macroinvertebrates into appropriate container. Preserve sample as directed in SOP 039.

**3.6** Thoroughly decontaminate the device as described in SOP 005 § 3.3.2 "Solid Materials Samplers".

## 4.0 Maintenance

Maintain according to manufacturers suggestions.

## 5.0 Precautions

**5.1** Inspect the device for mechanical deficiencies prior to its use.

**5.2** This sampler is inefficient in waters deeper than approximately 75 to 100 feet, under adverse weather conditions, and in waters of moderate to strong currents or wave action.

**5.3** Exercise caution at all times once the grab is loaded or cocked because a safety lock is not part of the standard design.

**5.4** Operate the sampler from a boat with a winch and cable.

**5.5** Wear gloves when collecting sediment samples. Be sure to consult the health and safety plan for the proper dermal and respiratory protection prior to collecting any samples.

**5.6** Higher levels of personal protective equipment may be required by the HASP.

**5.7** While sampling from a boat in water bodies with a depth of five feet or more, the sampling team shall wear personal flotation devices (life jackets).

**5.8** Collect samples first from those areas that are suspected of being the least contaminated, thus minimizing the risk of cross contamination.

## 6.0 References

ASTM Standard 2.1. D4387 Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates

USEPA. 1990. Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters. Office of Research and Development. EPA/600/4-90/030. November, 1990.

# STANDARD OPERATING PROCEDURE 023
# ORGANIC VAPOR ANALYZER (FOXBORO 128 GC)

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for field operations with the organic vapor analyzer (Foxboro Model 128 GC). The organic vapor analyzer (OVA) is an intrinsically safe, flame ionization detector designed to detect and measure organic vapor concentrations by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated. This information is used to determine control measures such as protection and action levels.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. probe/readout assembly
b. sidepack assembly
c. tedlar bag
d. calibration gas (*e.g.* methane 90 - 100ppm)
e. fuel (zero grade hydrogen - 99.999%)
f. tygon tubing
g. regulator

## 3.0 Startup Procedure

**3.1** Connect the Probe/Readout Assembly to the Sidepack Assembly by attaching the sample line and the electronic jack to their respective receptacles located on the side of the Sidepack.

**3.2** Check the battery condition by moving the INSTRUMENT toggle switch to the "BATT." position and ensure that the meter needle is beyond the white BATTERY OK line.

**3.3** Move the INSTRUMENT toggle switch to the ON position, and allow a 5 minute warm-up.

**3.4** Adjust the CALIBRATION ADJUST knob to set the meter needle to the level desired for activating the audible alarm. If the alarm level is other than zero, the CALIBRATION range toggle switch must be set to the appropriate range (i.e., X1, X10, or X100).

**3.5** Turn the ALARM VOLUME knob fully clockwise.

**3.6** Using the ALARM LEVEL ADJUST knob (located on the back of the Probe/Readout Assembly), turn the knob until the audible alarm is activated.

**3.7** Move the CALIBRATION range toggle switch to the X1 position and adjust the meter reading to zero using the CALIBRATION ADJUST knob.

**3.8** Turn the PUMP toggle switch ON. Place the instrument in the vertical position. Observe the SAMPLE FLOW RATE tube. Ensure flow rate is between 1-1/2 - 2-1/2 units.

**3.9** Open the $H_2$ TANK valve and the $H_2$ SUPPLY valve, wait one minute for hydrogen to purge the system.

**3.10** Press the red IGNITER BUTTON (located on the side of the Sidepack Assembly) until the alarm sounds and the needle on the Probe/Readout Assembly jumps upscale. CAUTION: THE IGNITER BUTTON SHOULD NOT BE DEPRESSED FOR MORE THAN 8 SECONDS. IF FLAME DOES NOT LIGHT WITHIN 8 SECONDS, WAIT 1 MINUTE AND TRY AGAIN.

**3.11** The instrument is ready for use. Use the CALIBRATION ADJUST knob to zero out ambient background organics.

## 4.0 SHUT DOWN PROCEDURE

**4.1** Close $H_2$ TANK VALVE

**4.2** Close $H_2$ SUPPLY VALVE

**4.3** Move "INSTRUMENT" BATT./OFF/ON toggle switch to OFF.

**4.4** Wait 5 seconds and move the "PUMP" toggle switch to OFF.

## 5.0 FUEL REFILLING

**WARNING: THERE SHOULD BE NO POTENTIAL IGNITERS OR FLAMES IN THE AREA.**

**5.1** The instrument and the charger should be completely shut down prior to hydrogen tank refilling operations. Refilling should be done in a well ventilated non-hazardous area.

**5.2** If this is the first filling of the instrument or if the filling hose has been allowed to fill with air, the filling hose should be purged with hydrogen prior to filling the instrument tank.

**5.3** The filling hose should be left attached to the hydrogen supply tank when possible. Ensure that the FILL/BLEED valve on the instrument end of the hose is in the OFF position. Connect the hose to the refill connection on the Sidepack Assembly.

**5.4** Open the hydrogen supply bottle valve slightly. Open the $H_2$ REFILL VALVE and the $H_2$ TANK VALVE on the instrument and place the FILL/BLEED valve on the filling hose assembly to the FILL position.

**5.5** After the fuel tank is filled, close the $H_2$ REFILL VALVE on the instrument, close the FILL/BLEED valve on the refill hose, and close the valve on the hydrogen supply bottle.

**5.6** The hydrogen trapped in the refill hose must now be bled off. CAUTION: THE REFILL HOSE WILL CONTAIN A SIGNIFICANT AMOUNT OF HYDROGEN AT HIGH PRESSURE. Turn the FILL/BLEED valve on the filling hose to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED valve should be turned to the FILL position to allow the hydrogen trapped in the connector fittings to move into the hose assembly. Turn the FILL/BLEED valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED valve to the OFF position to keep the remaining hydrogen in the hose at one atmosphere to ensure no air will be trapped in the hose for the next filling.

**5.7** Close the $H_2$ TANK VALVE.

**5.8** Observe the $H_2$ TANK PRESSURE meter and ensure that the pressure reading does not decrease rapidly.

## 6.0 CALIBRATION

Field calibration is accomplished using a single known sample of methane in air in the range of 90ppm to 100ppm. This may not provide the accuracy stated under specifications but is adequate for field survey work.

**6.1** Place instrument in normal operation with the CALIBRATION range toggle switch set to X10 and the GAS SELECT KNOB SET TO 300.

**6.2** Use the "CALIBRATION" ADJUST knob to adjust the meter reading to zero.

**6.3** Fill a tedlar bag with methane sample of known concentration (between 90ppm and 100ppm) and connect to the OVA sample probe.

**6.4** Adjust the CALIBRATION GAS SELECT KNOB until the meter reading is equivalent to the value of the gas standard.

**6.5** Record in a field log book: date, time, location, instrument ID number, calibration gas and concentration, final GAS SELECT setting, and the name of the person calibrating the instrument.

## 7.0 BATTERY CHARGING

WARNING: NEVER CHARGE BATTERY IN A HAZARDOUS ENVIRONMENT

**7.1** Insert battery charger cable into the battery pack RECHARGER receptacle. Plug battery charger into 115 VAC outlet.

**7.2** Turn battery charger on.

**7.3** Approximately one hour of charging is required for each hour of use. However, an overnight charge is recommended. The charger can be left on indefinitely without damage to the batteries. When finished, turn the charger off and disconnect the charger from the battery.

## 8.0 PRECAUTIONS

**8.1** Keep battery on charger when not in use, and recharge battery as soon as possible after use.

**8.2** Avoid intake of boiling vapors and liquids.

**8.3** Avoid over-tightening of valves.

**8.4** Use zero grade $H_2$ (99.999%, certified total hydrocarbons as methane < 0.5 ppm recommended).

**8.5** Calibration gas mixture must be balanced in air.

**8.6** Do not over tighten valves.

## 9.0 REFERENCES

Foxboro OVA 128 Reference Manual, December, 1985.
ICF Field Equipment Manual, November, 1988.

# STANDARD OPERATING PROCEDURE 024
# PHOTOIONIZATION DETECTOR (MICROTIP HL-200)

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for field operations with the photoionization detector (Microtip HL-200). The photoionization detector (PID) uses an ultraviolet emitting lamp designed to detect, measure and display the total concentration of airborne ionizable gases and vapors. This information is used to determine control measures such as protection and action levels.

Use of brand names in this SOP is in no wise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. Microtip
b. Battery Pack
c. Calibration Gas (100ppm Isobutylene)
d. Tedlar Bag
e. Tygon Tubing
f. Regulator

## 3.0 Startup/Calibration Procedure

**3.1** Turn the instrument on by pressing the back of the power switch located on the handle of the Microtip.

**3.2** The message "Warming up now, please wait" will be displayed for up to three minutes. After normal display appears, the Microtip is ready for calibration.

**3.3** Fill a tedlar bag with the desired calibration gas (usually 100ppm Isobutylene).

**3.4** Press SETUP button and select the desired Cal Memory using the arrow keys (normally set to 200ppm). Press EXIT button to leave setup function.

**3.5** Press CAL button and expose Microtip to Zero Gas. (Usually clean outdoor air will be suitable. If any doubt exists as to the cleanliness of the background air a commercial source of zero gas should be used.).

**3.6** The Microtip then asks for the Span Gas concentration. Enter the known span gas concentration and then connect the tedlar bag containing the Span Gas.

**NOTE: THE SPAN GAS CONCENTRATION IS DEPENDENT UPON BOTH THE CONCENTRATION OF THE SPAN GAS USED AND THE RATING OF THE UV LAMP IN THE MICROTIP AT TIME OF CALIBRATION. IF USING 100ppm ISOBUTYLENE AND THE STANDARD 10.6 eV LAMP, THE SPAN GAS CONCENTRATION WILL BE 56ppm.**

3.7 Press enter and the Microtip sets its sensitivity. Once the display reverts to normal the Microtip is calibrated and ready for use. Remove the Span Gas from the inlet probe. The instrument should be calibrated at least once a day.

## 4.0 Battery charging

4.1 Ensure Microtip is off.

4.2 Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage.

4.3 Press the release button on the bottom of the Microtip and remove the battery pack by sliding it backwards.

4.4 Plug charger into the battery pack and then into an AC outlet and allow the battery to charge for at least 8 hours.

4.5 After charging, remove the charger, first from the outlet then from the battery pack, and slide the battery pack back onto the Microtip.

## 5.0 Precautions

5.1 Microtip does not carry an Intrinsic Safety Rating and must not be used in a hazardous location where flammable concentrations of gases or vapors are constantly present.

5.2 All calibration, maintenance and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

5.3 Do not open or mutilate battery cells

5.4 Do not defeat proper polarity orientation between the battery pack and battery charger.

5.5 Substitution of components may affect safety rating.

## 6.0 References

Microtip HL-200 User's Manual, February, 1990.

# STANDARD OPERATING PROCEDURE 025
# SOIL SAMPLING

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for sampling surface and subsurface soils. Soil samples give an indication of the area and depth of site contamination, so a representative sample is very important.

## 2.0 Materials

a. Stainless steel spoon, trowel, knife, spatula, (as needed)
b. Split-spoon, Shelby tube, or core barrel sampler
c. Bucket auger or push tube sampler
d. Drill rig and associated equipment
e. Stainless steel bowl
f. PPE as required by the HASP

## 3.0 Procedure

### 3.1 Subsurface Samples

**3.1.1** Don PPE. Collect split-spoon, core barrel, or Shelby Tube samples during drilling.

**3.1.2** Upon opening sampler, or extruding sample, immediately screen soil for volatile organic compounds using either a PID or FID. If sampling for VOCs, determing the area of highest concentration, use a stainless steel knife, trowel or lab spatula to peel and sample this area.

**3.1.3** Log the sample in field notebook in accordance with SOP 003, while it is still in the sampler.

**3.1.4** Peel and transfer the remaining sample in a decontaminated stainless steel bowl. Mix thoroughly with a decontaminated stainless steel spoon or trowel.

**3.1.5** Place the sample into the required number of sample jars.

**3.1.6** Preserve samples as required in SOP 039.

**3.1.7** Discard any remaining sample into the drums being used for collection of cuttings.

**3.1.8** Decon sampling implements according to SOP 005 § 3.3.2.

**3.1.9** All borings will be abandoned according to procedures in SOP 028.

NOTES: If sample recoveries are poor, it may be necessary to composite samples before placing them in jars. In this case, the procedure will be the same, except that two split-spoon samples will be mixed together. The field logbook should clearly state that the samples have been composited, which samples were composited, and why the compositing was done.

Samples taken for geotechnical analysis will be undisturbed samples, collected using a thin-walled (shelby tube) sampler.

**3.2 Surficial Soil Samples**

**3.2.1** Don PPE. Remove vegetative mat. Collect a sample from under the vegetative mat with a stainless steel trowel, push tube sampler, or bucket auger.

**3.2.2** If a representative sample is desired over the depth of a shallow hole or if several shallow samples are to be taken to represent an area, composite as follows:

**3.2.2.1** As each sample is collected, place a standard volume in a stainless steel bowl.

**3.2.2.2** After all samples from each hole or area are in the bucket, homogenize the sample thoroughly with a decontaminated stainless steel spoon, trowel or spatula.

**3.2.3** If no compositing is to occur place sample directly into the sample jars.

**3.2.4** Place the leftover soil in the auger borings and holes left by sampling. If necessary, add clean sand to bring the subsampling areas back to original grade. Replace the vegetative mat over the disturbed areas.

**3.2.5** Samples for VOCs will not be composited. A separate sample will be taken from a central location of the area being composited and transferred directly from the sampler to the sample container.

**3.2.6** Preserve samples as required in SOP 039.

**3.2.7** Decon sampling implements according to SOP 005 § 3.3.2

**3.3 Refer to SOP 1-5, and 16.**

**4.0 Maintenance**

Not Applicable.

## 5.0 Precautions

5.1 Refer to the Health and Safety Plan.

5.2 Soil samples will not include vegetative matter, rocks, or pebbles, unless the latter are part of the overall soil matrix.

## 6.0 References

ASTM Method D1586-84, Penetration Test and Split-Barrel Sampling of Soils.

ASTM Method D1587-83, Thin Walled Sampling of Soils.

Department of the Army, Office of the Chief of Engineers, Engineer Manual 1110-2-1907 Soil Sampling, 31 March 1972

# STANDARD OPERATING PROCEDURE 026
# ACTIVE SOIL GAS ANALYSIS

## 1.0 Scope and Application

The purpose of this standard operating procedures is to describe protocols for using the Sentex Scentograph Portable Gas Chromatograph (GC) to analyze soil gas samples for volatile organic compounds. The primary method described here for introducing samples into the GC uses an absorbent tube to concentrate samples prior to analysis, although sample loop and direct injection methods may also be used. Operation of the GC should not be attempted without first reading the operation manual.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. Sentex Scentograph Portable GC with argon ionization detector (AID)/electron capture detector (ECD)
b. Toshiba T 1200 laptop personal computer
c. Battery charger for Scentograph GC
d. Battery charger for Toshiba PC
e. Industrial Solvents Calibration Library software and empty data disks
f. Argon (high purity) carrier gas
g. Calibration gases, as needed
h. Regulator for carrier gas
i. Regulator for calibration gas
j. Copper gas transfer tubing and fittings
k. Three 1L Tedlar bags
l. Multimeter
m. Spare analytical column
n. Spare Septa
o. Sample loop assembly and loops
p. Spare preconcentrator assembly
q. Other spare parts (e.g., tubing, swagelock fittings)
r. Syringes (1 $\mu$L, 5 $\mu$L, 10 $\mu$L, 100 $\mu$L, 1 mL, 5 mL, 50 mL
s. Syringe cleaner
t. Vacuum pump for syringe cleaner
u. Tools (Allen wrench, large adjustable wrench, small adjustable wrench, ¼" wrench, small screwdrivers)
v. Paperwork (applicable regulations and NRC license, Scentograph Operators' Manual, GC log book, table of ionization potentials)

## 3.0 Setup

3.1 The GC should preferably be operated in a clean environment, without significant concentrations of volatile organic compounds in the atmosphere. Batteries and internal gas canisters allow self-contained operation of up to six to eight hours, but longer continuous operation requires a 110-v power supply and external source of ultra-high-purity argon carrier gas. After each portable operation, the instrument must be recharged for at least 8 hours, preferably overnight, before resuming operations. When the instrument is not in use, it should be connected to the battery charger.

3.2 Install regulator on the carrier gas cylinder, attach ⅛" copper tubing to the outlet of the regulator and briefly purge the tubing by carefully opening the cylinder valve slightly. Connect the free end of the copper tubing with quick connector to the fitting marked carrier on the back of the GC, being careful to insert the connector straight. Open cylinder valve and carrier gas valve on GC to fill the internal tank. The maximum pressure is 1,800 psi, and severe damage can result from over pressuring the internal tanks. Repeat this process for calibration gas, connecting tubing to fitting marked calibration. Do not operate the GC if the carrier gas pressure is less than 200 psi.

3.3 Set column pressure to desired value (typically 30 psi) by adjusting carrier gas regulator inside the GC with an Allen wrench. Do not use a column pressure of less than 10 psi. After at least five minutes, turn on the PC (with Sentex software disk in A drive and blank data disk in the B drive) and set initial conditions at the operating parameters menu. For the industrial solvents calibration library, the operating conditions should be as follows:

Sample time: Adjust as needed, generally 10–20 sec.
Delay time: 0.5 sec.
Desorption time: 4.0 sec.
Inhibit time: 80 sec.
Oven temperature: 100°C
Chart duration: 20 min.
Analyses/calibration: Adjust as needed (used in automatic mode only)
Column: 12' 10% SP1000
Column pressure: 30 psi
Retention time window: Typically 3%
Noise threshold: Typically 50–100

Other operating conditions should be selected as needed depending on project needs if the Industrial Solvents Calibration Library is not being used.

## 4.0 Calibration

4.1 Using the Industrial Calibration Library software, it is only necessary to calibrate with benzene. Otherwise, calibration gases containing the compounds of interest are needed. A calibration standard near the expected sample concentration should be used.

4.2 At the operating parameters menu, name the calibration mixture, identify the peaks, and specify their concentrations. Recheck operating parameters.

4.3 Calibration gas from the internal cylinder or from an external source may be used. If using the internal tank, turn on the calibration gas valve on the rear of the GC. If an external source is used, turn off the carrier gas valve, run calibrations until the calibration gas pressure is zero, and attach the carrier gas source (at atmospheric pressure) to the calibration port on the side of the GC.

4.4 Initiate the calibration run by pressing 4 [enter] at the main menu. If using the industrial solvents calibration library, the benzene peak must be the first peak and must fall between 230 and 242 seconds. If not, make small adjustments to the temperature or column pressure and repeat.

## 5.0 Operation

5.1 After satisfactory calibration of the instrument, attach a Tedlar bag or similar sample container to the analysis port on the instrument and open the inlet valve on the container. Type 3 followed by a return at the main menu to initiate a sample run. If manual operation is selected after a change in operating parameters before a calibration run, the instrument will automatically start a calibration cycle before the analysis run.

5.2 The instrument will match observed peaks with those in the calibration gas and in the calibration library (if used), identify the peaks, and calculate concentrations based on peak area. Peak data as well as operating conditions are stored on a data disk in the B drive.

## 6.0 Quality Control

At a minimum, a quality control (GC) sample containing constituents of interest should be analyzed at the beginning and end of each batch of samples. The instrument should be calibrated before each sample batch, after any change in operation conditions, and when any changes in instrument response is noted. Duplicate samples should be analyzed every 10 to 20 samples.

## 7.0 Recall and Display of Results

To recall results, type 5 followed by a return at the operations menu. The analysis summary, listing trace number, analysis date, analysis time, peak identification, concentration, retention time, peak area, and sample (or calibrant) name, will be displayed. To view a particular trace, type 6 followed by a return at the operation menu, then specify the trace number.

## 8.0 Shutdown

After the last analysis, turn off the PC. Allow the column and detector to cool before shutting off the carrier gas supply. Turn off carrier and calibration gas valves on the back of the GC and turn off cylinder valves. Bleed excess pressure from the copper tubing and disconnect from the GC.

## 9.0 Precautions

The argon ionization detector uses a 150-mCi tritium source on a thin metal foil. The source is sealed in a steel cylinder. Any repairs of the detector must be made by the manufacturer. The detector housing and the sealed source must never be opened.

To prevent damage to the instrument, the following precautions must be observed:

- Do not operate the GC with less Than 200 psi carrier gas pressure.
- Do not fill the internal tanks to over 1,800 psi.
- Allow at least 5 minutes of carrier gas flow through the column before turning off the instrument.
- Do not replace the preconcentrator assembly without instructions from the manufacturer.
- Do not select a column pressure less than 10 psi or greater than 30 psi.
- Use only carrier grade argon as a carrier gas (at least 99.995%, preferably 99.999% pure). Industrial grade argon (e.g., from a welding shop) will cause contamination.
- Use only high purity regulators for the gas supply.
- If problems occur, call Sentex at (201) 945-3694.

# STANDARD OPERATING PROCEDURE 027
# PASSIVE SOIL GAS SURVEYS

## 1.0 Scope and Application

The steps and information herein are the "Standard Procedures" for carrying out a Petrex environmental survey. Possible deviations from standard procedures may occasionally be implemented on-site by our field staff to adjust for unique survey conditions. The Petrex Technique is also frequently used for oil and gas, geothermal, and mineral exploration which force slight variations on these "Standard Operating Procedures." Also, surveys performed in winter in frozen ground offer a unique situation and slightly different field practices.

The fact that the standard procedures may occasionally be altered is done to maintain quality service while using the Petrex Technique. It must also be understood that the ion flux data from one survey at a given site and a given time interval should not be compared to the flux numbers from another survey. Since the data is semi-quantitative, only the flux patterns of a survey or the relative difference between flux values of two samples from the same survey should be considered during interpretation.

If any questions arise upon review of this document, please address your questions to NERI technical staff at:

Northeast Research Institute, Inc.
309 Farmington Avenue, Suite A-100 Farmington, Connecticut
06032
(203) 677-9666

-or-

Northeast Research Institute, Inc. 605 Parfet Street, Suite
100, Lakewood, Colorado 80215
(303) 238-0090

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Sample Production and Preparation

### 2.1 Charcoal Sieving

The static VOC (Volatile Organic Compound) collector is prepared by applying pre-sieved activated charcoal to the end of a ferromagnetic wire.

## 2.2 Charcoal Bonding

The details of the procedure for preparing the activated charcoal is proprietary information. The procedure results in the production of a collector consisting of size-sorted activated charcoal bonded to the area within 1 cm of the end of a ferromagnetic wire with a Curie point of 358°C.

## 2.3 Collector Containers

Culture tubes, measuring 25 mm x 125 mm and having a screw cap closure, are washed in a biodegradable detergent, rinsed in methanol, and baked at 180 °C for one hour.

## 2.4 Wire Cleaning

The previously constructed wires are cleaned by heating in a special apparatus at 358 °C a total of 35 times under high vacuum. The wires are cleaned in lots of 32 wires. From each lot, two wires are removed for immediate analysis to verify the cleanliness of the lot. The remaining 30 wires are then sealed in one clean culture tube under an inert atmosphere and placed in inventory.

## 2.5 Packaging for Client

Immediately prior to shipping the wires to the field, the tubes containing 30 wires are removed from inventory and the wires are repackaged under an inert atmosphere in individual tubes. All of the repackaged tubes contain two wires. Ten percent of these have three wires. The collectors are packaged by bagging in zip-seal plastic bags in an inert atmosphere. These bags are then placed in inventory in a temperature-controlled room. The basis for having two wires in each tube is that it allows NERI to analyze one wire by standard Thermal Desorption-Mass Spectrometry (TD-MS) while the second sample is available for TD-GC/MS or as a backup to the TD-MS. The third wire in selected samples from each survey is used to establish optimum instrument parameters.

## 2.6 Quality Control and Quality Assurance

Prior to releasing stocked wires for a field survey, two single wires from each lot are checked for cleanliness and collecting potential. This QA/QC phase measures and documents collector preparedness when leaving the laboratory. One of these wires is analyzed without exposure in order to demonstrate that the lot is clean, and the other wire is exposed to hexane vapor for two seconds and then analyzed in order to verify that the charcoal is highly adsorptive. The triplicate wires are used when the wires return from the field. These wires help determine the required machine sensitivity and act as a measure of reproducibility.

## 2.7 Custody Document

A "custody document" accompanies each group of collectors leaving the laboratory and remains with the group until the collectors have been exposed, analyzed, and disposed of.

## 3.0 Field Operations

### 3.1 Locating Sample Sites

Sample placement sites, usually predetermined on an accepted survey proposal, are located from a nearby, surveyable landmark using a compass and pacing or some other measuring device (e.g., pacing wheel, hip chain, or tape measure). A transit may be used for more accurate placement, but such accuracy is seldom required.

### 3.2 Soil Coring

Once a sample site has been established, a hole is cored to a predetermined depth (sample placement depth is held constant for a given survey). This is accomplished using a variety of tools depending on the nature of the material to be cored. The holes should be vertical and as free from debris as possible. When the sampling is performed in areas covered by asphalt or concrete, a generator-powered rotary hammer drill with a carbide-tipped bit is used to drill a 1½ inch diameter hole in the cover. A hand auger is used to remove the cuttings and road base from the hole.

### 3.3 Collector Placement

Immediately after the hole is cored, a collector tube is removed from the zip-seal plastic bag and the bag is resealed. The cap is then removed from the tube, and the tube is placed vertically, open end down, into the hole. The hole is then back-filled with the soil core which was removed. The cap is placed in a clean zip-seal plastic bag and stored until collector retrieval. Collectors placed under asphalt or concrete are treated the same as those in uncovered soil, except for modifications to permit easy retrieval and to avoid potential down-hole contamination from surface cuttings. To allow retrieval of these collectors, a piece of galvanized wire is twisted around the neck of the tube and run to the surface so that the sample may be recovered by pulling the retrieval wire. An aluminum plug is then placed near the top of the hole, and the remainder of the hole is plugged with quick setting hydraulic cement.

### 3.4 Site Identification

Each site is flagged using pin flags, spray paint or ribbon flagging, and the site location is marked and numbered on a base map. A field notebook is used to record the date, collector number, site location description, soil type, and general observations.

### 3.5 Exposure Time

Time calibration collectors are included as part of every survey. These are QA collectors used to monitor sample loading during the survey. These collectors are placed in an area of known or suspected contamination, and sets are retrieved and analyzed at intervals to indicate the appropriate residence time for survey samples. Separate "travel blank" collectors are also included as a QC measure in every survey. These collectors are transported along with the survey collectors but the tubes are never opened. These control collectors monitor for potential contamination during transport or placement.

### 3.6 Collector Retrieval

The collectors are retrieved when the time calibration collectors reveal that there has been sufficient loading of gases on the charcoal absorbent. In the field, the soil is removed until the tube is exposed. A cap is taken from the sealed zip-seal plastic bag. The Viton seal is checked to make sure it is seated inside the cap. The culture tube is removed from the hole and any dirt that is on the threads of the tube is wiped off with a clean cloth. In the event the tube is broken or cracked, the collector wire is transferred to a new tube using forceps. The tube is capped and sealed. All flagging material is retrieved.

### 3.7 Collector Numbering

Each tube is immediately numbered according to the scheme established in the field notes and on the base map. The collector number is written on adhesive labels which are applied to the tube cap. No two sites may have the same number.

### 3.8 Collector Shipment

Once the collectors have been retrieved, they are sealed in zip-seal plastic bags and then wrapped with bubble packing. Material such as styrofoam peanuts or newsprint can introduce possible contaminants to the collectors and should not be used for packaging. The collectors, field notes, base map, and chain-of-custody document are either hand carried back to NERI's analytical laboratories, or are shipped by overnight carrier service.

### 3.9 Decontamination

All down-hole equipment and tool parts which contact excavated soil are constructed of heavy gauge steel and have no natural or synthetic components which could absorb and retain most soil-borne organic contaminants. These tools are decontaminated between use at each sampling location by rotation through a four step cleaning process. These steps are:

a. Immersion and vigorous scrubbing in a mild solution of laboratory grade detergent until all visual accumulations of soil are removed.

b. Thorough rinsing with potable water.

c. Spray rinsing with methyl alcohol.

d. Air Dry.

All derived liquids (and sediment) are contained in dedicated disposable vessels.

## 4.0 Collector Analysis

### 4.1 Numbering Check

Upon receipt of the collectors, the number on each tube is recorded and any missing or duplicated numbers are noted. A missing number generally indicates that the collector could not be retrieved. Samples with identical numbers generally cannot be used unless their true site location can be established.

### 4.2 Sample Holding

A Petrex soil gas sample consists of a minute quantity of various volatile organic compounds sorbed onto a charcoal element and enclosed in a protective container with a near impervious Viton seal.

Maximum sample holding time is a function of both the chemical stability of the sorbed compounds and the integrity of the seal of the container.

It has been the experience of Northeast Research Institute, Inc. (NERI) that Petrex soil gas samples that are properly repackaged after retrieval from the field and stored under environmentally controlled conditions typically remain compositionally and quantitatively unchanged through periods of greater than four months.

All samples scheduled for analysis via Curie-point pyrolysis/mass spectrometry are analyzed within three weeks of retrieval from the field.

### 4.3 Instrumentation

Thermal desorption is accomplished using a Fisher radio frequency power supply and a Curie point pyrolizer designed by NERI and Extrel. The mass spectrometer used is an Extrel Spectrel quadrapole mass spectrometer. The analysis is controlled and recorded by a DEC PDP 11/23 microcomputer. Following the analysis, all data are collected and archived on a PDP 11/73 microcomputer. Data for all active jobs are stored on both of the PDP 11 computers, as well as on magnetic tape. Data for all completed jobs are stored on magnetic tape in perpetuity.

### 4.4 Calibration

An Extranuclear Quadrupole Spectrometer equipped with a Curie-point pyrolysis/thermal desorption inlet is used for collector analysis. Mass assignment and resolution are

manually adjusted using a Perfluorotributylamine (PFTBA) standard. A linear correction, based on the known spectrum of PFTBA, is calculated. This correction is applied to a second PFTBA spectrum. If correct mass (M/Z) values are obtained, the operator proceeds to the next turning step. If not, Step 1 is repeated until correct masses are obtained.

Peak intensity ratios are set from the major peaks in the PFTBA spectrum using the following values:

| Mass (M/Z) | | Spectrum Intensities |
|---|---|---|
| 69 | - | 100% |
| 131 | - | 25%±5% |
| 219 | - | 35%±5% |
| 502 | - | 5%±2% |

At the standard mass (M/Z = 69), PFTBA is measured at a preset sample pressure and detector voltage and compared to previous values at the same setting.

Electron energy is set to 70 electron volts and emission is set at 12 milliseconds. All other operating parameters, such as scans, scan range, mass offset are established in the computer program. These values may only be changed by the laboratory manager.

Tuning is performed at the beginning of a run, so that an individual survey is analyzed at the same set of instrument conditions. The samples are analyzed in random order.

**4.5 Instrument Parameters**

The instrument is operated with the following parameters.

Vacuum - $<3 \times 10^{-6}$ torr
Ionization Energy - 70.0 eV
Ionization Current - 12.0 mA
Desorption Time - 5.0 sec
Desorption Temperature - 358 °C
Number of Scans/Sample - 30
Scan Rate - 1,250 amu/sec

**4.6 Mass Spectrometer Analysis and QA/QC**

Each collector wire is analyzed in random order. The entire group of survey collectors are analyzed as one run without interruption from other surveys.

The organic gases adsorbed on the carbon are thermally desorbed from the carbon, separated according to ion mass, counted, and a mass spectrum of masses from 29 to 240 is obtained.

Periodic (approximately every 20 samples) machine background analyses are performed as a QC measure to assure minimal influence from internal communication. If there are peaks that are not related to atmospheric gases, the supervisor is notified and the mass spectrometer is shut down and cleaned as necessary.

A written sample number record is kept during the analysis to prevent accidental cross numbering.

The mass spectrometer control program prompts the operator with a warning if a sample number is entered that has already been used. The operator then checks the current number, along with the disk storage location of the previously entered number, to resolve the true numbering situation.

### 4.7 Data Filing

The raw data file generated by the sample analysis is labeled and stored under a unique file name.

### 4.8 Schedule of Maintenance

1,000 Samples: Cleaning of sample introduction area, ion source, and expansion chamber by in-house technicians.

4,000 Samples: Above noted procedures plus cleaning of lenses and quadrapoles

Annually: Preventative maintenance program conducted by manufacture's service representative.

## 5.0 Data Interpretation and Presentation

### 5.1 Map Generation

The sample location maps are created by placing the field base map on a digitizing board and entering each site as an X-Y coordinate relative to an origin. The relative ion counts for each compound can then be plotted at the sample locations. Cultural and topographic features can also be digitized onto the map as reference points.

### 5.2 Compound Identification

The mass spectrum that is drawn for each sample is compared to a library of mass spectra derived from known volatile organic compounds. Several thousand pure compound spectra have been developed by the Bureau of Standards and are available for spectra comparison. NERI has also developed its own library of spectra through headspace analysis of pure compounds using the Petrex wires. Once a compound has been identified in this manner, the ion current or "flux" for this compound is defined as the total ion current for the "parent peak" or least interfered peak of that compound.

### 5.3 Relative Flux Determination

The process of determining ion currents (relative intensities) of indicator peaks is computerized. All ion current data are extracted from the original data file and are processed for identification.

The relative ion current intensity (relative intensities) of the gases that are desorbed from the collectors are matched with sample locations on a map of the survey area. These relative intensities are useful for inferring the areal extent of contamination and relative differences in the concentrations of the compounds in the soil or groundwater. This can aid in determining the location of source areas or direction of movement of contamination.

These surface collections and analyses cannot be used to determine the depth to the source contaminants or the precise concentration at depth.

Because compounds can be differentiated by their spectra, analyses from the carbon collectors can be used to help differentiate multiple compounds and multiple source areas within a single survey.

### 5.4 Data Interpretation

Once the relative intensities for a compound are mapped, the data can be contoured to reveal those areas with "hot spots" and the orientation of plume migration. All other available data, such as geologic setting, soil types, groundwater conditions, type of contaminant, site history, and other factors are taken into account as the interpreter draws his conclusions.

### 5.5 Additional Uses of Petrex Collectors

Some of the other uses of the Petrex Technique that are utilized in surveys are headspacing of soil and water samples and depth profiling.

#### 5.5.1 Headspace

A headspace soil sample is analyzed by collecting approximately 25 grams of soil, which are transferred to a thermochemically cleaned headspace container. Several

adsorption wires are added and the headspace container is sealed and allowed to equilibrate for up to 24 hours, depending on the level of contamination. The wires are then removed and prepared for desorption mass spectrometric analysis as described earlier. An identical process is performed for screening water samples.

### 5.5.2 Depth Profiling

In order to determine if the source of the soil gas signal is near surface or in a deeper vadose/saturated zone, depth profiling can be used.

At each selected location, shallow bore holes are drilling a few feet apart to depths such as 1, 2, 4, and 6 feet deep. After all the loose cuttings and carvings have been removed from the bottom of the hole, a core of soil may be taken for headspace analysis. Next, a Petrex collector is lowered into the hole and back-filled. The collectors remain in place for the same length of time as the survey wires.

Each of the sampling methods addresses a different aspect that will help indicate the nature of the VOC source. In the case of composite soil sampling, detection of VOCs during analysis implies that the VOCs are actually contained within the soil matrix. When the VOC is anthropogenic in nature, the VOC presence is indicative of soil contamination of that depth interval.

When performing an in situ time-integrated sampling program with Petrex collectors, the collector serves as both an extended headspace sampler relative to the soil matrix in its immediate vicinity, as well as measuring the soil gas flux though that zone during the exposure period.

Soil gas movement through the vadose zone is theorized to be a diffusion process. If the headspace data indicate that the VOC is not present in the soil matrix, then the in situ depth profiling collectors should show a relative increase of ion counts as the depth increases. By combining both pieces of data, the nature of the VOC source (near surface or deep vadose/saturated) can be inferred.

## 5.6 Data Presentation

Once the data have been compiled, interpreted, and mapped, a report is produced for the client's use. Also, the maps are printed which display the relative intensity of the compounds of the client's specifications. These reports and maps are for the client's use only, and no report or map is released to anyone else without prior written consent of the client. This confidentiality policy is never breached.

# 6.0 Interpretation of Petrex Maps

The policies outlined in this Standard Operating Procedure are strictly followed on each survey. It should be noted that the relative intensities for any compound at one sample location can only be

compared to another location within the same survey for the same compound. Relative intensities of different compounds cannot be compared to each other. Also, the relative intensities of one survey cannot be compared to the relative intensities of any other survey, even between two surveys at different times of the year over the same site. However, the same "hot spots" and plumes should contour in the same place over multiple surveys at a given site, allowing for migration.

# STANDARD OPERATING PROCEDURE 028
# WELL AND BORING ABANDONMENT

## 1.0 Scope and Application

The purpose of this standard operating procedure is to establish the protocols by which all borings and wells will be safely abandoned. The primary objective of well abandonment is to ensure that the abandoned well or boring does not provide a conduit for the vertical migration of contamination between aquifers.

## 2.0 Materials

a. Drill Rig
b. Filter Pack Material
c. Pure Sodium Bentonite With no Additives (bentonite) Powder (grout)
d. Bentonite Pellets (seal)
e. Cement (Portland Type II)
f. Approved Water

## 3.0 Procedure

The procedures used in boring abandonment will ideally accomplish two objectives: 1) protect aquifers from cross-contamination by sealing the borehole 2) restore the strata in the borehole to nearly original conditions by selective placement of fill material.

Any casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 feet of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD", for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site or cluster.

If the hole is within 15 ft. of a monitoring well in the same aquifer, or a replacement well is to be installed within 15 ft. of the well, any temporary casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be back-filled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2-feet or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, two feet of bentonite will be placed above the sand and a 3-foot concrete plug will be placed at the surface. Otherwise, backfill materials will be placed from the bottom of the hole to within 3 feet of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD", for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples,

completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 15 ft. of another monitoring well, or if there are no substantial, continuous sand bodies, and no replacement well is planned within 15 feet of the original well location then the hole may be grouted from the bottom to the top.

## 3.1 Grout

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, type II or V);
- 0.4 to 1 part (max.)(2-5%) bentonite; and
- 8-gallons (max.) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an above-ground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement.

Grout placement will be performed using a commercially available grout pump and a rigid tremie pipe removal and grouting will be accomplished in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This will be accomplished by placing a grout pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the top-most section, until grout flows from the boring at ground surface. Efforts will be made to grout incrementally as the temporary casing is removed.

After 24 hours, the abandoned drilling site will be checked for grout settlement. On that day, any settlement depression will be filled with grout and rechecked 24 hours later. This process will be repeated until firm grout remains at the ground surface.

## 3.2 Borings

The term "Borings" as used in this SOP applies to any drilled hole made during the course of RI which is not completed as a well. This includes soil test borings, soil sampling borings, and deep stratigraphic borings. Whether completed to the planned depth or aborted for any reason prior to reaching that depth, borings will be grouted and will normally be closed within 4 hours, or within 4 hours or completion of logging of completion of logging.

### 3.2.1 Shallow Borings NOT Penetrating Water Table

Shallow borings made for the collection of subsurface soil samples will be abandoned by backfilling the hole with cuttings from the hole, **if and only if the boring does not penetrate the water table.** Clean sand will be used to make up any volume not filled by the cuttings.

### 3.2.2 Borings Penetrating the Water Table

Shallow borings made for the collection of subsurface soil samples **which penetrate the water table** will be abandoned by grouting the hole from the bottom to the top.

### 3.2.3 Deep Stratigraphic Borings

Deep stratigraphic borings will normally be located in areas which, by virtue of the historical record, are presumed relatively uncontaminated. Therefore, these borings are usually over 100 feet from any sampling well locations. Any boring located within 15 feet of a proposed well location, or located directly upgradient or downgradient (on anticipated flow line) of a proposed well location will be abandoned by placing clean sand in the aquifer intervals and bentonite or grout in aquitard intervals as described above. If the boring is over 15 feet from and/or not upgradient of a proposed well location, the boring will be completely filled with grout.

## 3.3 Wells

The following procedure applies to wells aborted prior to completion and existing wells determined to be ineffective or otherwise in need of closure.

Prior to abandoning any developed well, MDE will be provided written notification along with an abandonment plan for that well.

If the well is within 15 ft. of another monitoring well in the same aquifer, or a replacement well is to be installed within 15 ft. of the well, casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be back-filled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2-feet or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, two feet of bentonite will be placed above the sand and below the concrete plug near the surface. Backfill materials will be placed from the bottom of the hole to within 3 feet of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD", for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 15 ft. of another monitoring well, and is not to be replaced by another well within 15 ft. of the original location, casing will be pulled, drilled out, or

thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 feet of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD", for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

## 5.0 Replacement Wells

Replacement wells (if any) will normally be offset at least 15 feet from any abandoned well in a presumed up- or cross-gradient ground-water direction. Site-specific conditions may necessitate variation to this placement.

## 6.0 Precautions

None.

## 7.0 References

COMAR 26.04.04 Regulation of Water Supply, Sewage Disposal, and Solid Waste § .11 Abandonment Standards

# STANDARD OPERATING PROCEDURE 029
# EXTRACTION WELLS

## 1.0 Purpose

The purpose of this standard operating procedure is to delineate the protocol to be used in installing wells which will be used for ground-water extraction rather than monitoring. Examples of such uses are pump tests, water supply wells, and wells used in pump-and-treat remediation systems.

## 2.0 Materials

### 2.1 Drilling Equipment

a. Appropriately sized drill adequately equipped with augers, bits, drill stem, etc.
b. Steam cleaner and water obtained from approved source for decontaminating drilling equipment.
c. PID: Microtip HL-200 (or equivalent)
d. Water level indicator
e. Weighted steel tape measure
f. LEL-Oxygen monitor
g. Steel drums for intrusion derived wastes (drill cuttings, contaminated PPE, decon solutions, etc.)
h. Source of approved water
i. Heavy plastic sheeting
j. Sorbent pads and/or logs

### 2.2 Well Installation[27]

a. Well screen:[28]

PVC: JOHNSON (or equivalent); PVC Vee Wire Continuous slot, wire wrapped screen; SCH 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer

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[27] Technical information on all installed materials (screens, riser pipe, filter pack, bentonite, cement, etc.) and representative samples of the proposed filter pack, bentonite powder, and bentonite pellets will be supplied to the Contracting Officer's Representative (COR).

[28] Well screen slot size and filter pack gradation will be determined on case by case basis. Material type will be determined based on field tests of groundwater chemistry and contaminants, as well as planned rehabilitation technique(s) (*e.g.* acid wash or oxidizer to eliminate Fe slimes, etc.).

Stainless Steel: JOHNSON (or equivalent); stainless steel Vee-Wire Continuous slot, wire wrapped screen; 304 stainless steel; ASTM F480 flush threads; cleaned, wrapped, and heat sealed by manufacturer.

b. Riser pipe:

PVC: JOHNSON (or equivalent); std. PVC; SCH 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and pre-packaged by manufacturer

Stainless Steel: JOHNSON (or equivalent); SCH 5; 304 stainless steel; ASTM type A312 material; 4-inch diam.; cleaned, wrapped and heat sealed by manufacturer.

c. Plugs/Caps: JOHNSON (or equivalent); standard PVC or stainless steel
d. Filter pack: MORIE, #00 well gravel (or equivalent) Note: final gradation may vary as a function of the gradation of the formation (see footnote 2)
e. Fine Ottawa sand
f. Bentonite seal: BAROID, bentonite pellets (3/8-inch diam.)
g. Cement: Type II Portland Cement [29]
h. Bentonite powder: BAROID, Aquagel Gold Seal
i. Steel Protective Casing: BRAINARD-KILMAN (or equivalent) zinc-plated steel, lockable
j. Geotextile: MIRAFI (or equivalent); GTF 130; non-woven; 4 oz.
k. Coarse (blanket) gravel: Crushed stone aggregate
l. Containers for purged water, as required.
m. Submersible pump or bailer of appropriate capacity, and surge block sized to fit well
n. Hach DREL 2000 portable laboratory (or equivalent)
o. Conductivity, pH, ORD, turbidity, dissolved oxygen, and temperature meters
p. Electric well sounder and measuring tape.
q. Portland Type II cement

**2.3 Documentation**

a. Copy of appropriate Cluster Work Plan
b. Copy of Appendix A of the Generic Work Plan
c. Copy of approved Health And Safety Plan
d. Copies of well and excavation permits
e. Copies of SOPs 3, 5, 8-12, 19(this SOP), 23, 24, and 28
f. Boring log forms

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[29] If ground-water sulfate content is greater than 1500ppm, Type V cement is required for greater sulfate resistance.

g. Well completion diagram form
h. Well development form

**2.4 Geologist's personal equipment**

a. 10X handlens
b. Unified Soil Classification System chart
c. Munsell color chart
d. Sieve set (Keck model SS-81 or equivalent)
e. PPE as required by HASP

## 3.0 Procedure

### 3.1 Materials Approval

**3.1.1** Water sources for drilling, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the COR prior to arrival of the drilling equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analyses over the past two years, and the name and address of the analytical laboratory (if applicable).

**3.1.2** Pure sodium bentonite with no additives (bentonite) will be the only drilling fluid additive allowed, and its use must be approved by the COR prior to the arrival of the drilling equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product description, and intended use for the product.

**3.1.3** Granular Filter Pack material must be approved by the COR prior to drilling. A one-pint representative sample must be supplied to the COR. Information required includes: lithology, grain size distribution, brand name, source, processing method, and slot size of intended screen.

**3.1.4** Portland Type II cement will be used for grout (see previous footnote).

### 3.2 Drilling

**3.2.1** The objective of the selected drilling technique is to ensure that the drilling method provides representative data while minimizing subsurface contamination, cross contamination, and drilling costs. The drilling method used will be hollow stem auger or water/mud rotary. No other methods will be considered as available without approval of EPA. The method used at a specific site will be proposed in the work plan and evaluated by the COR.

3.2.2 A Site Geologist will be present during all well drilling and installation activities and will fully characterize all tasks performed in support of these activities into the monitoring well logbook. The Site Geologist will be responsible at only one operating rig for the logging of samples, monitoring of drilling operations, recording of water losses/gains and groundwater data, preparing the boring logs and well diagrams, and recording the well installation procedures of the rig. The Site Geologist will have onsite sufficient equipment in operable condition to perform efficiently his/her duties as outlined in the Generic Work Plan (GWP) and other contractual documents. Items in the possession of each Site Geologist will include, copies of Appendix A of the GWP, the approved HASP, this SOP, a hand lens (10X), a standard color chart, grain-size chart, and a weighted (with steel or iron) steel tape long enough to measure the deepest well, heavy enough to reach that depth, and small enough to fit readily within the annulus between the well and drill casing. The Site Geologist will also have onsite, a water level measuring device, preferably electrical.

3.2.3 No lubricants will be used on downhole drilling equipment. Additives containing either lead or copper will not be allowed. In addition, polychlorinated biphenyls will not be contained in hydraulic fluids or other fluids used in the drilling rig, pumps, or other field equipment and vehicles.

3.2.4 Surface runoff or other fluids will not be allowed to enter any boring or well during or after drilling/construction.

3.2.5 Antifreeze used to keep equipment from freezing will not contain rust inhibitors and sealants. Antifreeze is prohibited in an areas in contact with drilling fluid. The ground surface at the wellsite will be protected from possible coolant, fuel, and hydraulic fluid spills and/or leakage by placement of plastic sheeting with raised edges, draining into a lined catch basin large enough to contain spills and/or leakage from motors, radiators, or vehicle tanks. Sorbent pillows will be placed to catch obvious leaks from the drill rig. Sorbent logs may be used instead of, or in conjunction with a lined catch basin to contain spills.

3.2.6 An accurate measurement of the water level will be made upon encountering water in the borehole and later upon stabilization. Levels will be periodically checked throughout the course of drilling. Any unusual change in the water level in the hole such as a sudden rise of a few inches indicating artesian pressure in a confined aquifer will be noted. Appropriate action will be taken as required in the work plan for that well. Particular attention for such water-level changes will be given after penetrating any clay or silt bed, regardless of thickness, which has the potential to act as a confining layer.

**3.3 Well Construction and Installation**

After the hole is drilled and logged, backfill hole as required for proper screen placement.

**3.3.1** Well screen and casing should be inert with respect to the ground water; therefore, the selection of screen and casing material will be based on select field tests of aquifer chemistry and potential contaminants. Screen slot size will be determined by sieve analysis of formational material. The screen will be capped without sediment trap or DNAPL sampling cup, and lowered into the hole. The well casing will be pre-cut to extend 2 to 2.5 ft above the ground surface. Prior to placement of the last piece of well casing, a notch or other permanent reference point will be cut, filed, or scribed into the top edge of the casing.

**3.3.2** Filter pack material will be tremied into place, lightly tamped, and leveled. Filter pack will extend from the bottom of the hole to a height of 1 ft above the top of the screen. The filter pack will be capped with 1 ft of fine (Ottawa) sand to prevent the bentonite seal from infiltrating the filter pack.

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

Not Applicable.

## 6.0 References

COMAR 26.04.04 Regulation of Water Supply, Sewage Disposal, and Solid Waste § .11 Abandonment Standards

# STANDARD OPERATING PROCEDURE 030
# RADIOACTIVITY SURVEYS

## 1.0 Scope and Application

This protocol is developed to serve as guidance to personnel performing radiological environmental surveys of surface soil, water bodies, or other environmental media which may be potentially contaminated with alpha, beta and beta- gamma radioisotopes.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

The following assumptions will be made:

1. Radiological contamination is present at the site.

2. All radioisotopes are present, unless historical documentation is available to help identify the specific radioisotope(s) present.

3. A potential health hazard exists from external and internal radiation exposure until instrumentation survey data and appropriate environmental samples indicate otherwise.

## 2.0 Material

a. Eberline Model PAC-1SAG survey meter mated with an alpha scintillation detector (or equivalent)

b. Eberline Model E-520 survey meter (or equivalent), mated with a HP-210 hand held detector (or equivalent)

c. Eberline Smart Portable (ESP-2) survey meter (or equivalent) mated with a SPA-3 low energy gamma scintillation detector (or equivalent)

## 3.0 Survey Procedures

### 3.1 Survey Site Preparation.

**3.1.1** All survey instrumentation and sampling equipment will remain outside the potentially contaminated area until the boundaries of the contaminated site can be established. Upon establishing the contaminated boundaries, entry and exit routes will be designated for ingress and egress into the area. Cold and hot lines should be established to control the spread of potential radiological contamination from hot to cold areas.

3.1.2 A cartesian grid (X-Y) of the survey site will be developed. The Project Officer in charge of the survey will develop a specific methodology to accomplish this framework. The grid pattern should also be usable in locating sampling points for clean-up and in reproducing the sampling data. The grid of the survey site should be used in planning for the collection of other environmental samples from the site. UTM coordinates are preferred but not mandated.

3.1.3 The Project Officer in charge should note the following observations:

- Any standing water on the survey site

- Water run-off areas and where the run-off water is leading to i.e., streams, lakes, marshes,etc. these areas must be considered during the pathway analysis

- A pathway analysis will be performed to assist in determining the number and type of environmental samples needed to assess the potential health hazard.

**3.2 Instrumentation survey.**

**3.2.1 Calibration and Operational Checks.**

(a) All portable survey meters will be calibrated at quarterly intervals. All Instruments will be properly labeled with the calibration date posted on the label.

(b) All portable survey meters will be checked for operability prior to packing and shipping the instruments to the survey site. The operability check will consist of checking the operation of the survey meter with an appropriate radiation check source at a known distance from the instrument detector. The reading will be documented on the quality control form for each instrument and will be included in the instrumentation shipping kit.

(c) The operability check will be repeated at the survey site prior to starting the instrumentation survey, and periodically during the survey. The instrument readings will be recorded on the quality control form for each instrument.

3.2.2 Background radiation levels will be determined prior to entering the survey site.

3.2.3 An alpha instrumentation survey will be performed with an Eberline Model PAC-1SAG survey meter mated with an alpha scintillation detector (or equivalent). All alpha instrumentation readings will be taken at approximately 1 centimeter from the surface of the test media. All results will be recorded in disintegrations per minute (dpm).

3.2.4 A beta instrumentation survey will be performed with an Eberline Model E-520 survey meter (or equivalent), mated with a HP-210 hand held detector (or equivalent). All results will be recorded in millirad per hour (mrad/hr). All

readings will be taken approximately 1 centimeter from the surface of the test media.

3.2.5 A beta-gamma instrumentation survey will be performed with an Eberline Smart Portable (ESP-2) survey meter (or equivalent) mated with a SPA-3 low energy gamma scintillation detector (or equivalent). All beta-gamma measurements will be taken at approximately 1 meter from the surface of the test media. The results will be recorded in microrads per hour and the distance at which the measurement was taken will be documented.

**3.3 Surface Soil Sample Survey.**

3.3.1 The Project Officer will evaluate the need to collect soil samples. Soil samples may be needed to assess projected airborne contamination during remedial clean-up or when vehicles and personnel transverse the contaminated site. The Project Officer will design a soil sampling plan to adequately assess potential health risks from low level contamination in soil. The grid developed for the instrumentation survey could be used to assist in determining the soil sample collection points.

3.3.2 Background soil samples should be collected from areas outside of the potentially contaminated area. Background sample data could be used to compare natural occurring radioisotopes in the natural surroundings versus what is present in the contaminated site.

3.3.3 Soil samples will be collected, labelled and preserved as detailed in SOP 025 "Soil Sampling". Soil sample numbers will incorporate grid coordinates so that each can be readily identified and tracked back to the collection point.

**3.4 Water Samples Surveys.**

3.4.1 The Project Officer will evaluate the need to assess the potential contamination in the water bodies surrounding the contaminated survey site.

3.4.2 A water sampling plan will be designed to accomplish this task. Sampling will be accomplished according to protocols established in SOP 007 "Surface Water Sampling Procedures"

3.4.3 Background water samples should be collected from tap water sources in the near-by areas, and any other water bodies that could provide background data comparison to the potentially contaminated water site.

3.4.4 Water samples may be treated with Nitric Acid to prevent the plating of radiological materials to walls of the sample containers. Use of preservative is dictated specifically by the method and/or laboratory used for analysis (refer to work plan or QAPjP).

3.4.5 All sample containers should be labelled and packaged to assist in tracking management and to prevent leaking and spills.

**3.5 Other Environmental Surveys.**

3.5.1 The Project Officer will evaluate the need to assess the potential contamination in other environmental media such as air samples, vegetation samples, animal samples, etc.

3.5.2 A sampling plan and assessment methodologies will be developed for site specific environmental assessment.

**3.6 Refer to SOP 3 and 16.**

## 4.0 Maintenance

Refer to manufacturer's manuals for calibration and maintenance of instruments.

## 5.0 Precautions

NOTE: For purpose of this protocol, the soil surface is defined as the top 1-15 centimeter of soil.

## 6.0 References

Krey, Phillip W. (Acting Director) and Beck, Harold L. (Acting Deputy Director). 1990. EML Procedures Manual 27th Edition, Volume 1 (HASL-300), Chieco, Nancy A. *et al.* eds, Environmental Measurements Laboratory, U.S. Department of Energy

# STANDARD OPERATING PROCEDURE 031
# SAMPLE CONTAINER CLEANING

## 1.0 Scope and Application

The purpose of this standard operating procedure is to define laboratory protocols to be used in cleaning and preparing containers used to collect environmental samples.[30]

## 2.0 Materials

a. Polyethylene bottles
b. Amber glass bottles
c. 40 ml vials
d. Bottle caps
e. Polytetrafluoroethelyne (PTFE) liners
f. 5% NaOH
g. 5% Ultrex $HNO_3$
h. Deionized water
i. Alconox detergent
j. Hexane (Nanograde or equivalent)
k. Acetone
l. Methelyne Chloride

## 3.0 Procedures

### 3.1 Polyethylene bottles

3.1.1 Rinse bottles and lids sequentially with 5% NaOH, with deionized water, with 5% Ultrex nitric acid, and with deionized water.

3.1.2 Drain and allow to air dry.

### 3.2 Amber glass bottles and 40 ml vials

3.2.1 Wash bottles in detergent and rinse with copious amounts of distilled water.

3.2.2 Rinse with acetone

3.2.3 Rinse with methelyne chloride

3.2.4 Rinse with hexane

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[30] This SOP is included for completeness only. It is anticipated that sample containers will either be provided by the laboratory, or that the sampling contractor will purchase new, certified clean sample containers.

3.2.5 Allow bottles to air dry.

3.2.6 Place bottles in a drying oven and heat to 200°C.

3.2.7 Allow bottles to cool prior to sealing with clean caps and PTFE liners.

**3.3 Bottle Caps**

3.3.1 If applicable, remove paper liners from caps.

3.3.2 Wash caps with detergent, followed by a distilled water rinse.

3.3.3 Dry caps in drying oven at 40°C.

**3.4 PTFE liners**

3.4.1 Always handle liners with forceps or tweezers, never use fingers.

3.4.2 Wash liners with detergent, followed by distilled water rinse.

3.4.3 Rinse the liners with acetone, followed by hexane (Nanograde or equivalent).

3.4.4 Allow liners to air dry prior to placing in clean caps, then heat liner and caps in drying oven at 40°C for 2 hours.

3.4.5 Allow caps and liners to cool prior to placing on clean bottles.

**3.5** A statistically representative number of randomly selected clean sample containers shall be analyzed for TAL/TCL analytes (GWP Tables 8.2 - 8.5). Results of these analyses shall be provided to the contracting officer's representative (COR).

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

None.

## 6.0 References

None

# STANDARD OPERATING PROCEDURE 032
# DRIVE-POINT PIEZOMETER INSTALLATION

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SOP being drafted

# STANDARD OPERATING PROCEDURE 033
# SLUG TESTS

## 1.0 Scope and Application

A slug test is conducted to determine the characteristics of a confined aquifer in soil materials where the conductivity is too small to conduct a pumping test.

## 2.0 Materials

Test Conducted with Inert Cylinder

a. Transducer or other water level indicator
b. Logbook
c. Semilogarithmic paper-arithmetic vertical scale and logarithmic horizontal scale
d. Inert, negatively buoyant cylinder of known volume

Test Conducted with Input/Output of Water

a. Teflon bailer with teflon-coated stainless steel leader and rope or pump.
b. Logbook
c. Semilogarithmic paper
d. Transducer or other water level indicator

Note: All well intrusive equipment must be decontaminated prior to and after use, as is indicated in SOP 005.

## 3.0 Procedure

The slug test is conducted by measuring the response of a well to either a raising or a lowering of the water table. Two methods are commonly employed, insertion of an inert object or the addition/removal of water. The insertion of an object gives an instantaneous water level change, thereby providing a more accurate test than is obtained by adding or bailing water.

### 3.1 OPTION 1 - INERT OBJECT INSERTION

**3.1.1** Select an appropriate transducer for the range of water level change anticipated in the slug test.

**3.1.2** Submerge the transducer in the well to a suffcient depth to provide effective performance. The range of the transducer must be considered in the

determination of the submersion depth. Well bottom sediment plugging of the transducer must be avoided as well as transducer interference by the inert object.

**3.1.3** Measure the water level (background) for 24 hours.

**3.1.4** Lower an inert object into the well to displace the water and thereby raise the water level. The inert object may be a retrievable sealed PVC cylinder filled with sand or similar material. All intrusive equipment must be decontaminated as is described in SOP 005.

**3.1.5** Record the water level response during cylinder emplacement and record the water level response (falling) with the cylinder in place. This is called the falling head slug test.

**3.1.6** Once the water level has stabilized, the cylinder is removed and the rising head slug test is conducted by measuring the response of the water level to the removal of the cylinder.

### 3.2 OPTION 2 - ADDING OR REMOVING WATER

**3.2.1** Select an appropriate transducer for the range of water level change anticipated in the slug test.

**3.2.2** Submerge the transducer in the well to a sufficient depth to provide effective performance. The range of the transducer must be considered in selection of the submersion depth. Well bottom sediment plugging of the transducer must be avoided.

**3.2.3** Measure the water level background for 24 hours before initiating the water addition/extraction test.

**3.2.4** Water levels and water volumes extracted/added are recorded during the entire test.

**3.2.5** Water addition/extraction is continued until a condition of water table level equilibrium is reached. At equilibrium the rate of addition/extraction equals the well recharge rate and the recharge/drawdown remains constant.

**3.2.6** Water addition/withdrawal is then stopped and the response of the water level is continuously monitored and readings recorded.

NOTE: If removing water from a well, the water must be containerized for testing and if necessary, properly disposed of. If adding water to the well, the water should be from a non-chlorinated approved water source.

## 4.0 Interpretation

4.1 The field data are used in a mathematical analysis to determine the hydraulic condition in the well. Hydraulic conductivity, transmissivity, and the storage coefficient of the well can be determined from either the falling or rising head test. The coefficient of storage is the volume of water released from storage, per unit of aquifer storage area per unit change in head. The slug test may also be used to determine specific capacity; yield per unit of drawdown, expressed as gallons of water per minute per foot of drawdown (metric: cubic meters per day per meter).

## 5.0 Field Data Records

5.1 Logbook

5.1.1 Only one site or installation per logbook, and only one slug test per data table (see below).

5.1.2 The first page must include the well number, location, date of test, persons conducting the test, and reference plane for drawdown measurements.

5.1.3 Next page(s) must be table format with the columns designating time/date, water volume withdrawn/added or displaced by inert cylinder, water levels, etc.

5.1.4 Test data must be entered in a table as data are acquired. Data must include sufficient information to indicate that the water level was stable before the test, during equilibrium and after the test(s).

5.1.5 Further information on the logbook can be obtained from SOP 003.

## 6.0 Well Parameters, Data Logging and Plotting Procedures

6.1 Figure 1 (H. H. Cooper, et al., 1967) gives the definition and relationships of a confined aquifer well in which a volume of water has been displaced or added. The parameters need to be known to determine all formational characteristics listed in section 3.1 (above).

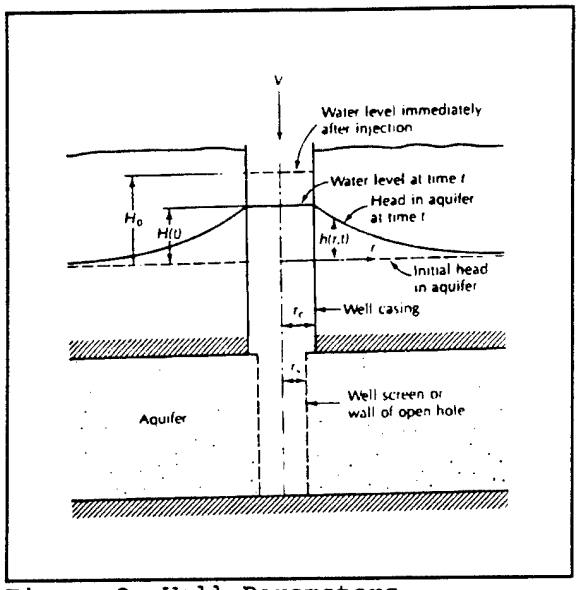


Figure 2 Well Parameters

**6.2 Data logging and plotting procedures**

6.2.1 Record the water level in the well immediately after the inert object emplacement/withdrawal (option 1) or at the equilibrium of the water table (option 2). This is the initial water level reading.

6.2.2 Following the initial water level reading, the water level in the well is continuously recorded along with the time of the level measurement.

6.2.3 The ratio of the initial water level to the change in head are plotted with respect to time.

6.2.4 The ratio is plotted on the arithmetic scale and time is plotted along the logarithmic scale.

6.2.5 The relationships of the initial water level to changes in the water level are a function of parameters shown in Figure 1 and the formation transmissivity. The values of the function relationship are plotted for a series of transmissivities and are depicted in Figure 2.

6.2.6 The resulting field data plot (curve) is compared to a series of type curves (Figure 2). The field-data curve is placed over the type curves with the arithmetic axis coincident. The field data curve is matched to the type curve that has the same curvature.

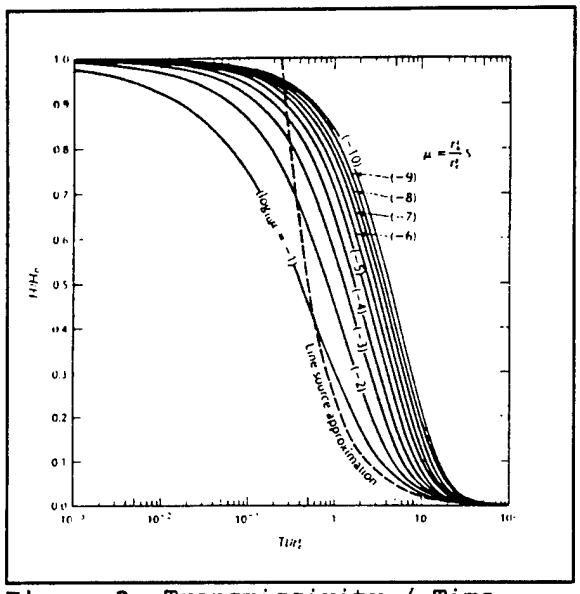


**Figure 3** Transmissivity / Time Function Curves

6.2.7 The formation transmissivity is determined.

6.2.8 The value of storativity is calculated.

## 7.0 Maintenance

7.1 The transducers must be kept clean, operable, and thoroughly tested before emplacement in the well. A plugged or malfunctioning piezometer will give erroneous responses or fail to give any response.

## 8.0 Precautions

8.1 The well must be cased above the aquifer.

8.2 The well must penetrate the entire aquifer.

8.3 The well must be an open hole or be screened.

## 9.0 References

Cooper, H. H., Jr., J. D. Bredehoeft, and I. S. Papadopulos, 1967. Response of a Finite Diameter Well to an Instantaneous Charge of Water, Water Resources Research, 3, pages 263-269.

Driscoll, F. G., 1986. Groundwater and Wells, Johnson Division, St. Paul, Minnesota 55112.

Fetter, C. W. Jr. 1980. Applied Hydrogeology, C. E. Merrill Publishing Company, Columbus, OH 43216.

# STANDARD OPERATING PROCEDURE 034
# "ORPHAN OR UNCLAIMED " WELLS

## 1.0 Scope and Application

The purpose of this standard operating procedure is to provide a plan of action for protecting, evaluating, and redeveloping, or abandoning "orphan or unclaimed" wells discovered during RI field investigations.

## 2.0 Material

a. Field Notebook
b. Padlock
c. Duct tape

## 3.0 Procedure

Upon discovering an "unclaimed" or "orphaned" well, the following steps should be taken to preserve the integrity of the well, prevent contamination to the aquifer, and determine the history of the well.

**3.1** Note any markings, or features of the "well" such as type of casing, amount of stickup, presence or absence of protective casing, presence or absence of cap, etc. in the field notebook (see also SOP 003).

**3.2** Accurately locate the well on map, air photo, or in field notebook by direction and distance from landmarks. This will ensure that the well can be located again (see also SOP 003).

**3.3** The well will be immediately secured from easy intrusion as follows:

**3.3.1** If protective casing is in place, make sure it is locked.

**3.3.2** If a lock is in place, no further immediate action is necessary.

**3.3.3** If no lock is in place, and the hasp is in working order, procure a lock and secure the well as soon as possible.

**3.3.4** If the hasp is missing or broken, or if the protective casing is missing, secure the well by taping it shut.

**3.4** Notify within 24 hours orally or by facsimile:

a - Project manager
b - Appropriate APG project manager(s)
c - Maryland Department of the Environment - appropriate study area manager
d - EPA Remedial Project Manager

**3.5** Begin to research all available documentation to attempt to determine the history of the well no later than the next working day.

**3.6** No later than 1 week later. Return to the well with a water level indicator or weighted tape measure and appropriate decon materials (see SOPs 003, 005, 010, and 016) to determine the depth of the well, whether the well does or does not have water in it, and the water level.

**3.7** If the well is appropriately located, (*e.g.* use of that well would contribute to attaining site data objectives either as a sampling well or as a piezometer) and its surface casings appear intact, initiate procedures to evaluate whether the well construction is adequate for environmental sampling. This evaluation should include a thorough records search to determine well construction and downhole geophysics to determine grout, screen, and casing integrity. This evaluation may also include a downhole video camera survey to verify screen and casing type and condition.

**3.8** If it is desirable to use the well, **and** the well is appropriately constructed, **and** downhole geophysics indicate that the casing, screen, and grout are intact, then re-development proceedings pursuant to Section 6.12 of the GWP may be initiated. Otherwise initiate abandonment procedures (appendix A 1.12.2 of the GWP and SOP 028)

## 4.0 Maintenance

Not Applicable.

## 5.0 Precautions

Not Applicable.

## 6.0 References

COMAR 26.04.04.11 "Abandonment Standards"

# STANDARD OPERATING PROCEDURE 035
# AGENT SCREENING

*** SOP currently being drafted ***

# STANDARD OPERATING PROCEDURE 036
# TURBIDITY MEASUREMENTS (DRT 100)

## 1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure is to delineate protocols for measuring the turbidity of all types of aqueous solutions, including drinking water, saline water, and industrial and domestic wastes. Turbidity is an indication of the optical properties that cause light to be scattered or absorbed through an aqueous sample. Turbidity is largely a function of the refractive index and the size and shape of the particles suspended or dissolved in the solution. Turbidity meters do not produce an "absolute" measurement, but one that is "relative" to the optical nature of the solids in solution.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 MATERIAL

a. Turbidity meter (DRT 100 or equivalent)
b. Lint free laboratory wipes (Kimwipes or equivalent)
c. Formazin standards (from manufacturer)
d. Sample bottle
e. Cuvettes

## 3.0 PROCEDURE

**3.1** Calibration of the turbidity meter will be checked on a daily basis as follows.

**3.1.1** Set the range switch to 1000 range before turning the turbidity meter on and whenever the light shield is not in place over the sample well.

**3.1.2** Allow the turbidity meter 15 to 60 minutes to warm-up.

**3.1.3** Clean the reference standard with kimwipes.

**3.1.4** Place the formazin suspension or reference standard in the turbidity meter sample well.

**3.1.5** Place the light shield over the reference standard.

**3.1.6** Rotate the front panel range switch counterclockwise to the appropriate NTU range.

**3.1.7** Adjust the reference adjust knob counterclockwise to read the same value as the reference standard value. This value is stamped on top of the reference standard.

**3.1.8** The turbidity meter is now standardized on all ranges to the factory formazin calibration and unknown samples may be read directly in NTU, FTU, or JTU.

**3.1.9** Rotate the range switch clockwise to the 1000 range before removing the reference standard.

**3.1.10** Record reading in Field Logbook. (Refer to SOPs 003, and 016.)

**3.1.11** Do not leave the reference standard in the sample well for long periods.

**3.2** Turbidity will be measured as follows.

**3.2.1** Pour aqueous sample into a new cuvette assuring no air bubbles.

**3.2.2** Place the cuvette into the sample well.

**3.2.3** Place the light shield over the sample.

**3.2.4** Rotate the range switch counterclockwise to the range which provides best readability and sensitivity for the sample being measured.

**3.2.5** Allow the turbidity meter to stabilize before recording the NTU value.

**3.2.6** Turn the range switch clockwise to the 1000 range and then remove the sample.

**3.2.7** Do not leave the filled cuvette in the sample well for long periods.

**3.2.8** Repeat steps 3.2.1 - 3.2.7 for additional samples.

**3.3** Cuvette cleaning procedure is as follows.

**3.3.1** Cuvette must be clean and free of rubs or scratches.

**3.3.2** Wash the cuvette in a detergent solution.

**3.3.3** Rinse thoroughly 8-10 times, preferably with distilled water to remove all streaks.

**3.3.4** Polish with kimwipes.

**3.3.5** Cuvettes must be stored in a clean dust-free environment.

## 4.0 MAINTENANCE

**4.1** Source Lamp may be replaced as follows.

**4.1.1** Remove the instrument case per manufacturer instruction.

**4.1.2** Remove the bulb by loosening a screw and removing the electrical leads.

**4.1.3** Insert the new bulb and reconnect the electrical leads.

**4.1.4** Before tightening the screw, be sure to position the filament so that it will be parallel to the axis of the sample well.

**4.1.5** Insert the lamp alignment tool in the sample well to focus the new bulb.

**4.1.6** Move the lamp bracket assembly in or out until a focused image of the filament is within the rectangular box on the lamp alignment tool.

**4.1.7** Once the filament image has been aligned and focused within the rectangular box on the lamp adjustment tool, tighten all screws snugly.

**4.1.8** Replace the instrument case

## 5.0 PRECAUTION

**5.1** Handle the reference standard or sample cuvettes by the top to prevent surface scratches or finger smudges which will cause analysis errors.

**5.2** Check the mechanical meter zero when the instrument is in a vertical position and the power switch is off. Adjust to zero only if necessary by means of the black screw on the meter face.

**5.3** The turbidity meter should be left on for the entire work shift to minimize warm-up and recalibration delays.

**5.4** Do not leave the reference standard or filled cuvette in the sample well for long periods.

**5.5** Leave the light shield in place on the instrument when it is not in use in order to protect the sample well for long periods.

**5.6** Always set the range switch to 1000 range before turning the instrument on and whenever the light shield is not in place over the well.

## 6.0 REFERENCES

Manufacturer's Manual

# STANDARD OPERATING PROCEDURE 037
# DISSOLVED OXYGEN MEASUREMENTS (YSI Model 57)

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for measuring the dissolved oxygen (DO) of all types of aqueous solutions, including drinking water, saline water, and industrial and domestic wastes. DO is a measurement of the amount of soluble oxygen in an aqueous solution. It is a general indication of an aerobic/anaerobic condition of a water sample.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. DO meter (YSI model 57 or equivalent)
b. Self-stirring BOD bottle probe
c. Membrane standards
d. BOD bottle

## 3.0 Procedure

### 3.1 Setup

It is important that before the meter is prepared for use and calibrated, it should be placed in the intended operating position, i.e., vertical, tilted, or horizonal. The instrument may need readjustment if the operating position is altered. The setup procedures should be as follows:

**3.1.1** With the switch set to **OFF**, adjust the meter pointer to zero with the screw in the center of the meter panel.

**3.1.2** Switch to **RED LINE** and adjust the **RED LINE** knob until the meter needle aligns with the red mark if necessary.

**3.1.3** Switch to **ZERO** and adjust to 0 mg/L scale with the **ZERO** control knob.

**3.1.4** Attach the prepared probe to the **PROBE** connector of the instrument and adjust the retaining ring finger tight.

**3.1.5** Before calibrating, allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been **OFF** or the probe has been disconnected.

3.2 Calibration

Calibration is accomplished by exposing the probe to a known oxygen concentration, such as water-saturated air (%), or water of a known oxygen content (mg/L), and then adjusting the calibration controls so the display shows a reading matching the oxygen concentration of the known sample. Calibration can be disturbed by physical shock, touching the membrane, and fouling of the membrane or drying out of the electrolyte. Calibration will be checked after each series of measurements. Calibration of the DO meter will be performed on a daily basis as follows.

**3.2.1** Place the probe in moist air. BOD probes can be placed in partially filled (50 ml) BOD bottles. Wait 10 minutes for temperature stabilize ± 2°C.

**3.2.2** Switch to **TEMPERATURE** and read. Refer to Table 1 for solubility of oxygen in fresh water (calibration value).

**3.2.3** Determine altitude or atmospheric correction factor from Table 2.

**3.2.4** Multiply the calibration value from Table 1 by the correction factor from Table 2 to obtain the correct calibration value.

**EXAMPLE:** Assume a temperature of 20°C and an altitude of 1100 ft. From Table 1, the calibration value of 20°C is 9.09 mg/L. From Table 2, the correction factor for 1100 ft is 0.96. Therefore, the corrected calibration value is 9.09 mg/L x 0.96 = 8.73 mg/L.

**3.2.5** Switch to the appropriate mg/L range, set the **SALINITY** knob to zero and adjust the **CALIBRATE** knob until the meter reads the calibration value from Step 4. Wait two minutes to verify calibration stability. Readjust if necessary.

3.3 DO Measurement

**3.3.1** With the instrument prepared for use and the probe calibrated, place the probe in the sample.

**3.3.2** Turn the **STIRRER** knob **ON**.

**3.3.3** Adjust the **SALINITY** knob to the salinity of the sample if appropriate.

**3.3.4** Allow sufficient time for the probe to equilibrate to the sample temperature and dissolved oxygen.

**3.3.5** Read dissolved oxygen on appropriate scale.

**3.3.6** Before measuring the DO of the next sample, rinse probe and sample bottle with distilled water and then with next water sample.

**3.3.7** Follow steps 3.3.1 - 3.3.6 for the next sample(s).

**3.3.8** The DO meter should normally be left on during the working day to avoid the delay of waiting for probe repolarization.

## 4.0 MAINTENANCE

The following steps will be taken to maintain the DO meter.

**4.1** Replace the batteries when the **RED LINE** knob is at its extreme adjustment or at least annually.

**4.2** In the **BATT CHECK** position on the **STIRRER** knob, the voltage of the stirrer batteries is displayed on the red **0-10** scale. Do not permit them to discharge below 6 volts.

**4.3** Replace membrane every 2 to 4 weeks depending on application. Probes will be stored in a humid environment to prevent drying out.

## 5.0 PRECAUTION

The DO meter case is water resistant when properly closed. As a precaution against damaged gaskets or loose fittings, the instrument case will be opened and inspected for moisture whenever the instrument has been subjected to immersion or heavy spray. The case is opened by removing the screws on the rear cover an lifting the cover off.

## 6.0 REFERENCES

Manufacturer's handbook.

**TABLE 1. SOLUBILITY OF OXYGEN IN WATER EXPOSED TO WATER SATURATED AIR AT 760 mm Hg PRESSURE**

| Temp. °C | Solubility mg/L | Temp. °C | Solubility mg/L | Temp. °C | Solubility mg/L |
|---|---|---|---|---|---|
| 0 | 14.62 | 17 | 9.67 | 34 | 7.07 |
| 1 | 14.22 | 18 | 9.47 | 35 | 7.95 |
| 2 | 13.83 | 19 | 9.28 | 36 | 7.84 |
| 3 | 13.46 | 20 | 9.09 | 37 | 6.73 |
| 4 | 13.11 | 21 | 8.92 | 38 | 6.62 |
| 5 | 12.77 | 22 | 8.74 | 39 | 6.52 |
| 6 | 12.45 | 23 | 8.58 | 40 | 6.41 |
| 7 | 12.14 | 24 | 8.42 | 41 | 6.31 |
| 8 | 11.84 | 25 | 8.26 | 42 | 6.21 |
| 9 | 11.56 | 26 | 8.11 | 43 | 6.12 |
| 10 | 11.29 | 27 | 7.97 | 44 | 6.02 |
| 11 | 11.03 | 28 | 7.83 | 45 | 5.95 |
| 12 | 10.78 | 29 | 7.69 | 46 | 5.84 |
| 13 | 10.54 | 30 | 7.56 | 47 | 5.74 |
| 14 | 10.31 | 31 | 7.43 | 48 | 5.65 |
| 15 | 10.08 | 32 | 7.31 | 49 | 5.56 |
| 16 | 9.87 | 33 | 7.18 | 50 | 5.47 |

Derived from 17th Edition, Standard Methods for the Examination of Water and Wastewater.

**TABLE 2. CALIBRATION VALUES FOR VARIOUS ATMOSPHERIC PRESSURES AND ALTITUDES**

| Pressure | | | Altitude in | | Calibration Value(%) |
|---|---|---|---|---|---|
| inches Hg | mm Hg | kPa | feet | meter | |
| 30.23 | 768 | 102.3 | -276 | -84 | 101 |
| 29.92 | 760 | 101.3 | 0 | 0 | 100 |
| 29.61 | 752 | 100.3 | 278 | 85 | 99 |
| 29.33 | 745 | 99.3 | 558 | 170 | 98 |
| 29.02 | 737 | 98.3 | 841 | 256 | 97 |
| 28.74 | 730 | 97.3 | 1126 | 343 | 96 |
| 28.43 | 722 | 96.3 | 1413 | 431 | 95 |
| 28.11 | 714 | 95.3 | 1703 | 519 | 94 |
| 27.83 | 707 | 94.2 | 1995 | 608 | 93 |
| 27.52 | 699 | 93.2 | 2290 | 698 | 92 |
| 27.24 | 692 | 92.2 | 2587 | 789 | 91 |
| 26.93 | 684 | 91.2 | 2887 | 880 | 90 |
| 26.61 | 676 | 90.2 | 3190 | 972 | 89 |
| 26.34 | 669 | 89.2 | 3496 | 1066 | 88 |
| 26.02 | 661 | 88.2 | 3804 | 1160 | 87 |
| 25.75 | 654 | 87.1 | 4115 | 1254 | 86 |
| 25.43 | 646 | 86.1 | 4430 | 1350 | 85 |
| 25.12 | 638 | 85.1 | 4747 | 1447 | 84 |
| 24.84 | 631 | 84.1 | 5067 | 1544 | 83 |
| 24.53 | 623 | 83.1 | 5391 | 1643 | 82 |
| 24.25 | 616 | 82.1 | 5717 | 1743 | 81 |
| 23.94 | 608 | 81.1 | 6047 | 1843 | 80 |
| 23.62 | 600 | 80.0 | 6381 | 1945 | 79 |
| 23.35 | 593 | 79.0 | 6717 | 2047 | 78 |
| 23.03 | 585 | 78.0 | 7058 | 2151 | 77 |
| 22.76 | 578 | 77.0 | 7401 | 2256 | 76 |
| 22.44 | 570 | 76.0 | 7749 | 2362 | 75 |
| 22.13 | 562 | 75.0 | 8100 | 2469 | 74 |
| 21.85 | 555 | 74.0 | 8455 | 2577 | 73 |
| 21.54 | 547 | 73.0 | 8815 | 2687 | 72 |
| 21.26 | 540 | 71.9 | 9178 | 2797 | 71 |
| 20.94 | 532 | 70.9 | 9545 | 2909 | 70 |
| 20.63 | 524 | 69.9 | 9917 | 3023 | 69 |
| 20.35 | 517 | 68.9 | 10293 | 3137 | 68 |
| 20.04 | 509 | 67.9 | 10673 | 3253 | 67 |
| 19.76 | 502 | 66.9 | 11058 | 3371 | 66 |

Derived from 17th Edition, Standard Methods for the Examination of Water and Wastewater.

# STANDARD OPERATING PROCEDURE 038
# REDOX POTENTIAL MEASUREMENTS

## 1.0 Scope And Application

The purpose of this standard operating procedure is to delineate protocols for measuring the redox of flooded sediment and soil. Redox is an indication of the reduction of oxidation intensity of an anaerobic system. Redox will be expressed in mV. A complete anaerobic system redox potential is -150 Mv. A positive value indicates an aerobic system.

Redox measurement in a natural system is difficult to obtain due to the absence of true equilibria, heterogeneity of media, and pH effects deprived redox measurements in natural media of precise thermodynamic significance (Ponnamperuma 1972). Despite the difficulties involved in the redox measurements in natural media, it is widely accepted that redox readings in natural anaerobic systems can provide valuable environmental information. Whitfield found redox useful as a semiquantative indicator of the degree of stagnation of a particular aquatic environment as did Ponnamperuma for flooded soils and sediments. The redox measurements of natural waters are not representative of that median, since natural waters are in a highly dynamic state rather than in or near equilibrium, according to Stumm and Morgan. It is generally recognized that redox measurements in oxygenated natural waters are invalid.

## 2.0 Material

a. pH Meter
b. Combination pH electrode
c. Lint free laboratory wipes (Kimwipes, or equivalent)
d. Distilled water
e. Sample bottle
f. Standard solution (pH 4 and 7 buffers saturated with quinhydrone).
g. Wash bottle

## 3.0 Procedure

**3.1** Calibration of the pH meter will be calibrated on a daily basis as follows.

**3.1.1** Prepare beaker of standard solution with known voltage(s).

**3.1.2** Connect electrodes to instrument.

**3.1.3** Turn on and clear

**3.1.4** Rinse electrode with distilled water blot excess with laboratory wipes.

3.1.5 Immerse probe in beaker of standard solution.

3.1.6 Press mV key.

3.1.7 After the reading stabilizes, the absolute mV of solution is displayed.

3.1.8 Rinse electrode and blot excess.

3.2 Redox will be measured after calibration as follows.

3.2.1 Prepare sample in a beaker.

3.2.2 Rinse electrode and blot excess water.

3.2.3 Immerse electrode in sample and stir briefly.

3.2.4 Press mV switch.

3.2.5 Record the reading after it stabilizes.

3.2.6 For next sample(s), follow step 3.2.1 - 3.2.5.

**4.0 Maintenance**

4.1 Check the batteries each time the meter is used.

4.2 Keep the probe stored in a 0.1 M KCL solution when not in use. Alternatively, the electrode may be rinsed with deionized water and trapping any residual water inside the protective cap.

**5.0 Precaution**

5.1 Remove coatings of oil material or particulate matter that can impair electrode response by gentle wiping or detergent washing, followed by distilled water rinsing.

**6.0 References**

Beckman Instruments, Inc., User Manual for Φ 10, 11, and 12 pH/ISE meter.

Ponnamperuma, F. N., "The Chemistry of Submerged Soils," Advances in Agronomy, Vol 24, 1972.

Stumm, W. and Morgan, J. J., Aquatic Chemistry, Wiley, New York, 1970.

Whitfield, M., "Eh as an Operational Parameter in Estuarine Studies," Limnol. Oceanogr., Vol 14, 1969.

# STANDARD OPERATING PROCEDURE 039
# SAMPLE PRESERVATION AND CONTAINER REQUIREMENTS

## 1.0 Purpose and Scope

The purpose of this standard operating procedure is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

## 2.0 Material

a. Containers (see § 3.0 below for description)
b. $HNO_3$
c. $H_2SO_4$
d. NaOH
f. Ice chests
g. Ice

## 3.0 Definition of Container Types

**Type A Container**: 80 oz amber glass, ring handle bottle/jug, 38-mm neck finish.
**Closure**: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm Polytetrafluoroethelyne (PTFE) liner.

**Type B Container**: 40-mL glass vial, 24 mm neck finish
**Closure**: White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size.
**Septum**: 24-mm disc of 0.005-in PTFE bonded to 0.120-in silicon for total thickness of 0.125-in.

**Type C Container**: 1-L high density polyethylene, cylinder-round bottle, 28-mm neck finish.
**Closure**: White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner.

**Type D Container**: 120-mL wide mouth glass vial, 48-mm neck finish.
**Closure**: White polyethylene cap, 40-480 size; 0.015-mm PTFE liner.

**Type E Container**: 250-mL boston round glass bottle
**Closure**: White polypropylene or black phenolic, open top, screw cap.
**Septum**: Disc of 0.005-in PTFE bonded to 0.120-in silicon for total thickness of 0.125-in.

**Type F Container**: 8-oz short, wide mouth, straight -sided, flint glass jar, 70-mm neck finish.
**Closure**: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner.

**Type G Container**: 4-oz tall, wide mouth, straight -sided, flint glass jar, 48-mm neck finish.
**Closure**: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner.

**Type H Container**: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish.
**Closure**: White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner.

**Type K Container**: 4-L amber glass ring handle bottle/jug, 38-mm neck finish.
**Closure**: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner.

**Type L Container**: 500-mL high-density polyethylene, cylinder bottle, 28-mm neck finish.
**Closure**: White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner.

## 4.0 Procedure

4.1 All containers must be certified clean, with copies of laboratory certification furnished to the contracting officer's representative (COR).

4.2 Water samples will be collected according to procedures detailed in SOPs 007, 013, and 014 into containers appropriate to the intended analyte as given in Table 039-1.

4.2.1 Samples taken for metals analysis will be acidified in the field to a pH < 2 by the addition of $HNO_3$. Filtered samples will be acidified after filtration. After acidifying the sample, the container should be lightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated.

4.2.2 Samples taken for total phosphorous content will be acidified in the field to a pH < 2 by the addition of $H_2SO_4$. After acidifying the sample, the container should be lightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated.

4.2.3 Samples taken for cyanide will be alkalized to a pH > 12 by the addition of NaOH.

4.2.4 No preservatives will be added to any other water samples. These samples will be immediately placed on ice and cooled to 4°C.

4.3 Soil, sediment, and sludge samples will be collected according to procedures detailed in SOPs 021, 025, and 041 into containers appropriate to the intended analyte as given in Table 039-2.

4.3.1 Samples taken for metals analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C.

4.3.2 Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C.

4.3.3 Samples taken for cyanide will be alkalized to a pH > 12 by the addition of NaOH.

4.3.4 No preservatives will be added to any other soil samples. These samples will be immediately placed on ice and cooled to 4°C.

4.3.5 Where field screening indicates the presence of mustard, methylene chloride or chloroform will be added to the sample container to cover and preserve the sample. ** awaiting confirmation of this procedure from CRDEC **

## 5.0 Maintenance

Not Applicable.

## 6.0 Precautions

HCl shall not be used to acidify samples. HCl will react with thiodiglycol to produce mustard agent by reverse hydrolysis.

Note that acidifying a sample containing cyanide may liberate HCN gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces such as a vehicle.
- Hold suspected HCN - generating sample away from body and downwind while manipulating it.
- See the HASP for other safety measures

## 7.0 References

Test Methods for Evaluating Solid Waste, SW-845, (EPA 1986)
A Compendium of Superfund Field Operations Methods, EPA 540-P87-001
A Compendium of ERT Soil Sampling and Surface Geophysics Procedures, (EPA 1991)

Table 039-1
Preservation Requirements for Water Samples

| Analyte | Bottle Requirement, Volume | Required Headspace | Preservative | Holding Time |
|---|---|---|---|---|
| Volatile Organic Compounds | (2)Type B, 80-mL total | 0% | Cool to 4°C | 7 days |
| Semivolatile Organic Compounds | Type A, K, or (2)H, 2-L total | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Pesticides/ Arochlors | Type A, K, or (2)H, 2-L total | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Total Metals | Type C, H, or (2)L, 1-L total | 10% | $HNO_3$ to pH < 2<br>Cool to 4°C | 6 months except Mercury (28 days) |
| Dissolved Metals | Type C, H, or (2)L, 1-L total | 10% | $HNO_3$ to pH < 2<br>Cool to 4°C | 6 months except Mercury (28 days) |
| Cyanide | Type C, H, or (2)L, 1-L total | 10% | NAOH to pH > 12<br>Cool to 4°C | 14 days |
| Total Phosphorous | (1) Type C, 1-L | 10% | $H_2SO_4$ to pH < 2<br>Cool to 4°C | 28 days |
| Explosives | (2) Type H, 1-L ea. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Thyodiglycol | (2) Type H, 1-L ea. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Dioxins/ Furans | (2) Type H, 1-L ea. | 10% | Cool to 4°C | 30 days to extraction (45 days to analysis) |
| Org-P and Org-s Compounds | (2) Type H, 1-L ea. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| IMPA and MPA | (1) Type E, 250-mL | 0% | Cool to 4°C | 40 days |
| Herbicides | (2) Type H, 1-L ea. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |

Table 039-2
Preservation Requirements for Soil and Sediment Samples

| Analyte | Bottle Requirement, Volume | Required Headspace | Preservative | Holding Time |
|---|---|---|---|---|
| Volatile Organic Compounds | (2) Type D, 240-mL total | 0% | Cool to 4°C | 14 days |
| Semivolatile Organic Compounds | Type F or G, 3 oz. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Pesticides/ Arochlors | Type F or G, 3 oz. | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Total Metals | Type F or G, 3 oz. | 10% | Cool to 4°C | 6 months except Mercury (28 days) |
| Cyanide | Type I, 1-L | 10% | Cool to 4°C | 14 days |
| Total Phosphorous | 1 1-L Polyethylene bottle | 10% | Cool to 4°C | 28 days |
| Explosives | (2) 1-L amber glass bottles | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Thyodiglycol | (2) 1-L amber glass bottles | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| Dioxins/ Furans | (2) 1-L amber glass bottles | 10% | Cool to 4°C | 30 days to extraction (45 days to analysis) |
| Org-P and Org-s Compounds | (2) 1-L amber glass bottles | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |
| IMPA and MPA | 1 250-mL glass bottle, septum top | 0% | Cool to 4°C | 40 days |
| Herbicides | (2) 1-L amber glass bottles | 10% | Cool to 4°C | 7 days to extraction, 40 days after extraction |

# STANDARD OPERATING PROCEDURE 040
# CONFINED SPACE ENTRY

## 1.0 Purpose and Scope

The following standard operating procedure provides the instructions necessary for entering a confined space. Always consult with Safety Program Management prior to conducting a job that involves entering a confined space.

A "confined space" means any space having a limited means of access or egress or so enclosed that adequate dilution ventilation is not obtained by natural air movement, or mechanically induced movement; and is subject to the accumulation of toxic or combustible agents or an oxygen deficiency. Confined spaces include but are not limited to any of the following areas: a storage tank, tank car, process vessel, bin, tank trailer, or any other tank-like compartment usually having one or more manholes for entry; an open-topped space more than four feet deep, such as a bin, silo, pit, vat, vault, vessel, or floating roof storage tank; a ventilation or exhaust duct, manhole, sewer, tunnel, pipeline, and similar structure; and an oven, furnace, kiln, or similar structure.

Confined spaces are herein classified into three categories based on hazards and the potential for exposure to the identified hazards. The classes and definitions include:

> Class A = A class A confined space includes those spaces that are designed to contain, are known to contain, or have contained hazardous or toxic materials (e.g., underground storage tanks, tank cars, process vessels, pipelines, etc.).
>
> Class B = A class B confined space includes those spaces that are subject to the infiltration of toxic or hazardous vapors or materials or to the reduction of oxygen content through displacement or chemical reaction (e.g.,bins, silos, pits, vats, manholes, sewers, tunnels, etc.).
>
> Class C = A class C confined space includes those spaces that meet the definition of a confined space, but have a relatively low probability for the accumulation of hazardous or toxic materials or for oxygen deficiency (e.g., open-topped space, construction site trench, etc.).

Whenever personnel are to enter a confined space, due consideration must be given to the safety of the person entering the space. The Project Manager, Field Operations Manager, and worker shall consider all possible options to ensure worker safety. At a minimum, consideration shall be given to using lifelines, the buddy system, and standby safety personnel.

## 2.0 Responsibilities

**2.1** Safety Program Management will:

a. Provide guidance and assistance to field personnel in the preparation of procedures and the identification and classification of confined spaces

b. Approve or disapprove entry procedures and SOPs for entry into confined spaces

c. Recommend engineering controls necessary to ensure a safe working environment

**2.2** Project Manager/Field Operations Manager will:

a. Complete all necessary confined space permits

b. Ensure all field personnel have read and understand the confined space entry procedure

c. Ensure team composition is adequate to safely perform tasks

d. Ensure personnel entering confined spaces and the personnel serving as safety spotters are properly trained in rescue and cardiopulmonary resuscitation procedures

e. Provide for constant communication between employees inside the confined space and employees outside the confined space

**2.3** Safety Spotter will:

a. Ensure proper rescue equipment is on site prior to confined space entry

b. Maintain continuous contact, either visual or verbal, with entrant

## 3.0 Required Equipment

a. Oxygen Analyzer
b. Explosimeter
c. Organic Vapor Monitor
d. Personal Protective Equipment

## 4.0 Prerequisites

**4.1** All personnel working in confined spaces are properly trained in safe entry and rescue procedures.

**4.2** A safety spotter is available to remain outside the confined space to give assistance as needed. The safety spotter shall be equipped with a self-contained breathing apparatus if conditions warrant.

## 5.0 Precautions and Limitations

**5.1** If for any reason a hazardous or unsafe condition presents itself for which there is no clear procedure or guideline, work shall cease until Program Safety Management can be notified and the condition rectified.

**5.2** No person shall enter a confined space without an emergency response spotter outside the confined space.

**5.3** A ladder shall be provide for any trench which is deeper than 4 ft. or when appropriate in other confined spaces.

## 6.0 Performance Steps

**6.1** Evaluate the work area against the definition of a confined space.

**6.2** Notify the Project Manager or Program Safety Management of any work spaces not previously identified that are suspected of meeting the definition of a confined space.

**6.3** Contact the APG designated confined space attendant prior to entry and verify APG requirements for entry.

**6.4** Obtain positive identification of the materials that are (or have been) present in the space.

a. Evaluate the hazards presented by the materials and byproducts.

b. Determine if any atmospheric monitoring will be required.

**6.5** Determine the necessary safety precautions and protective clothing required for the particular space, based on the classification of the confined space.

a. If the space meets the definition of a class A confined space, then proceed to step 6.6.

b. If the space meets the definition of a class B confined space, then proceed to step 6.7.

c. If the space meets the definition of a class C confined space, then proceed to step 6.8.

**6.6 Enter class A confined space.**

a. Prepare a confined space entry permit. Include the following as a minimum:

1) Identity of the space
2) Purpose of the entry
3) Date of authorized entry
4) Authorized entrants
5) Eligible attendants
6) Personnel eligible to be in charge of entry
7) Substances stored in the confined space
8) Potential hazards
9) Permitted work
10) PPE requirements
11) Safety equipment requirements
12) Signature and printed name of authorizing person

b. Ensure that any line, except for a fire suppressant or extinguishing system, that enters the space and carries a harmful agent is physically disconnected from the space or blocked by a device capable of ensuring complete closure.

c. Render inoperable by disconnection any fixed mechanical device or equipment which, if operated, might endanger personnel.

d. Except for lighting, padlock or tag out-of-service electrical service equipment.

e. Select a suitable entrance point that will be safe for the entrant to pass through. Open the entrance.

f. Survey the entrance to the confined space for oxygen levels, combustible vapors, and other hazards.

g. Verify, based on levels obtained from step f. above, that all precautions are sufficient to permit entry. Ensure entrant is properly suited in required personal protective equipment. Allow entrant to enter the confined space.

h. Conduct atmospheric monitoring inside the confined space to determine the presence of combustible, toxic gases, or an oxygen deficient atmosphere.

1) Determine oxygen levels prior to any other testing.

2) Draw test air from lowest to highest elevations of the confined space: 12-18 inches off of floor, mid-levels, and within 12-18 inches of the top (if possible).

3) For spaces greater than 500 cubic feet, draw test air from additional sample points in sufficient number to categorize the atmosphere in the confined space.

i. Once inside, verify any communication equipment used is properly working.

j Monitor for oxygen levels, combustible vapors, and any other identified hazards on a continuous basis while working in class A confined spaces. Continuous monitoring shall include all instruments on with the alarms functioning and set, or a second attendant whose only purpose is to monitor the work environment for the identified hazards.

k. Proceed to Step 6.9.

**6.7 Enter class B confined space.**

a. Ensure that any line, except for a fire suppressant or extinguishing system, that enters the space and carries a harmful agent is physically disconnected from the space or blocked by a device capable of ensuring complete closure.

b. Render inoperable by disconnection any fixed mechanical device or equipment which, if operated, might endanger personnel.

c. Except for lighting, padlock or tag out-of-service electrical service equipment.

d. Select a suitable entrance point that will be safe for the entrant to pass through. Open the entrance.

e. Survey the entrance to the confined space for oxygen levels, combustible vapors, and other hazards.

f. Verify, based on levels obtained from step e. above, that all precautions are sufficient to permit entry. Ensure entrant is properly suited in the required personal protective equipment. Allow entrant to enter the confined space.

g. Conduct atmospheric monitoring inside the confined space to determine the presence of combustible, toxic gases, or an oxygen deficient atmosphere.

1) Determine oxygen levels prior to any other testing.

2) Draw test air from lowest to highest elevations of the confined space: 12-18 inches off of floor, mid-levels, and within 12-18 inches of the top (if possible).

3) For spaces greater than 500 cubic feet, draw test air from additional sample points in sufficient number to categorize the atmosphere in the confined space.

h. Once inside, verify any communication equipment used is properly working.

i. Monitor for oxygen levels, combustible vapors, and any other identified hazards on a continuous basis while working in class B confined spaces. Continuous monitoring shall include all instruments on with the alarms functioning and set or a second attendant whose only purpose is to monitor the work environment for the identified hazards.

j. Proceed to Step 6.9.

**6.8 Enter class C confined space.**

a. Select a suitable entrance point that will be safe for the entrant to pass through. Open the entrance.

b. Survey the entrance to the confined space for oxygen levels, combustible vapors, and other hazards.

c. Verify based on levels obtained from step f. above that all precautions are sufficient to permit entry. Allow entrant to enter the confined space.

d. Conduct atmospheric monitoring inside the confined space to determine the presence of combustible, toxic gases, or an oxygen deficient atmosphere.

1) Determine oxygen levels prior to any other testing.

2) Draw test air from lowest to highest elevations of the confined space: 12-18 inches off of floor, mid-levels, and within 12-18 inches of the top (if possible).

3) For spaces greater than 500 cubic feet, draw test air from additional sample points in sufficient number to categorize the atmosphere in the confined space.

e. Monitor for oxygen levels, combustible vapors, and any other identified hazards on an intermittent basis while working in class C confined spaces. Intermittent monitoring shall include a complete set of readings for all initial parameters on a specified basis. The frequency of the monitoring shall be determined prior to entrance by Safety Program Management.

**6.9** Exit confined space.

**6.10** Close off confined space to prevent unauthorized access.

**6.11** Cancel confined space permit (if appropriate).

## 7.0 Documentation

Document the entry and results of all atmospheric monitoring in the field logbook. Maintain a copy of the confined space entry permit (if appropriate) in the project files.

## 8.0 References

COMAR 09.12.35,Maryland Occupational Safety and Health Standards for Confined Spaces

# STANDARD OPERATING PROCEDURE 041
# SLUDGE SAMPLING PROCEDURES

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for sampling sludges. Sludges include solid matter derived from waste materials that are suspended In or settled from a liquid. This procedure can be applied to the collection Of sludge samples from areas of deposition such as: tanks, sumps, landfills, ditches, ponds and lagoons. It Is Important to collect a representative sample of the waste material.

## 2.0 Material

a. Stainless steel or Teflon tray
b. Stainless steel hand core sludge sampler and extensions
c. Stainless steel dip sampler, spoons, trowels, spoons, and ladles
d. Sample bottles
e. Plastic sheeting
f. Utility knife
g. Polypropylene rope

## 3.0 Procedure

The liquid content of the sludge sample may vary from nearly all liquid to a dense, nearly liquid-free material. It may be necessary to use a variety of equipment to obtain the required samples, even at a single site.

### 3.1 General Procedure:

**3.1.1** Upon arrival at the site, immediately set up and organize the equipment.

**3.1.2** Establish background levels of airborne organic compounds using a photoionization detector (PID)or a flame ionization detector (FID).

**3.1.3** Cut a section of 6 mi[ plastic sheeting of approximately 6 ft x 6 ft. Place the sheeting on the upgradient side of the sample area.

**3.1.4** Arrange the sample containers. sampler(s), and decontamination equipment on the plastic sheeting.

**3.1.5** Don PPE in accordance with the site health and safety plan.

**3.1.6** Collect the sample(s). The preferred method of collecting sludge samples will be by hand corer, refer to Section 3.2. If using a scoop, trowel, spoon, or ladle, refer to Section 3.3.

## 3.2 Hand Corer

**3.2.1** Ensure that the corers and liners are properly decontaminated prior to use.

**3.2.2** Force the corer Into the sludge with a smooth continuous motion to a depth of 9 to 12 inches.

**3.2.3** Twist the corer to detach the sample; then withdraw the corer in a single smooth motion.

**3.2.4** Remove the top of the corer and, if excess liquid Is present, decant the liquid Into a sample bottle. This liquid will be labelled and analyzed.

**3.2.5** Remove the nosepiece of the corer and deposit the sample into a stainless steel or Teflon tray.

**3.2.6** Transfer the sample Into sample bottles using a stainless steel laboratory spoon or equivalent object.

**3.2.7** If possible, the top 6 Inches of the core will be sampled Into 3 separate sample bottles. 2 inches per bottle, to ensure that an accurate chronology of contamination can be determined.

**3.2.8** Ensure that each sample bottle Is properly labeled, noted on the chain-of-custody form, and placed In the sample cooler with Ice packs.

**3.2.9** Decontaminate sampling equipment according to SOP 005.

**3.2.10** Dispose of all sampling wastes In properly labelled containers

## 3.3 Scoop, Trowel, Spoon, or Ladle

**3.3.1** Ensure that the sampling equipment Is properly decontaminated prior to use.

**3.3.2** Insert the sampling device Into the material at the selected point and slowly remove the sample. Care should be taken to retain as much of the solid component as possible.

**3.3.3** Transfer the sample Into the appropriate sample bottles.

**3.3.4** Ensure that each sample bottle Is property labeled, noted on the chain-of-custody form, and placed In the sample cooler with Ice packs.

**3.3.5** Decontaminate sampling equipment according to SOP 005

**3.3.6** Dispose of all sampling wastes in properly labelled containers.

### 3.4 Sampling Location

For all samples mark the sampling location on a site map. Photograph (optional, recommended) the sampling site. Describe each sampling location in the field logbook. Establish the sampling coordinates using the Global Positioning System and record the coordinates for each sample In the field logbook.

## 4.0 Maintenance

Not applicable.

## 5.0 Precautions

Sludges may contain high levels of contaminants.

It Is extremely Important to continually monitor the levels of contaminants, using the appropriate survey instruments (e.g., PID, indicator tubes) in the breathing zone of the sampler(s) and other field team members.

Refer to the HASP for appropriate PPE.

Field team members should consult with the Site Health and Safety Coordinator for all health and safety questions or concerns relating to sampling activities.

## 6.0 References

EPA/54/P-87/001, A Compendium of Superfund Field Operations Methods.

# STANDARD OPERATING PROCEDURE 042
# DISPOSAL OF ENVIRONMENTAL WELL DEVELOPMENT/PURGE WATER

***SOP being drafted***

July, 1993

# STANDARD OPERATING PROCEDURE 043
# HYDROLAB MULTIPARAMETER WATER QUALITY MONITORING INSTRUMENT

## 1.0 Scope and Application

The purpose of this standard operating procedure is to delineate protocols for field operation with the multiparameter water quality logging system (H20 data son and SCOUT 2 receiver). The system can monitor up to eight basic parameters including dissolved Oxygen, % saturation, temperature,pH, specific conductance, resistivity, salinity, total dissolved solids, redox level, and depth.

Use of brand names in this SOP is in nowise intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for the maintenance and calibration of same.

## 2.0 Material

a. SCOUT 2 display
b. H20 Water Quality Data Transmitter
c. Underwater cables
d. Accessories

## 3.0 Procedure

These procedures are to be followed when using the Hydrolab in the field.

### 3.1 Start Up Procedures

**3.1.1** Attach the Cable to the Transmitter

**3.1.2** Connect the other end of the cable to the display

**3.1.3** Press the On/Off key on the Display's panel. Allow a few seconds for the Transmitter to start sending data to the Display screen.

**3.1.4** Calibrate the Transmitter as shown in Section 1.5

**3.1.5** Deploy the sensor over the side of the vessel in a minimum of 4" of water.

**3.1.6** Write data values displayed on the screen in the appropriate logbook.

**3.1.7** Retrieve sensor.

**3.1.8** Move to the next sampling location. If travel time is great, turn off display by pressing On/Off key. Check condition of probes after each deployment.

**3.1.9** When finished sampling for the day, disconnect the Transmitter.

### 3.2 Field Calibration Procedure

**3.2.1** Follow the start up procedure in section 3.1

**3.2.2** Fill the calibration cup with the appropriate standard as follows:

a. temperature - none required
b. specific conductance - KCI or seawater standards
c. pH - pH 7 buffer plus a slope buffer
d. dissolved oxygen - saturated air or saturated water
e. redox - quinhydrone or tranosfer
f. depth - set zero in air
g. level - set zero in air
h. salinity - uses calibration for specific conductance

**3.2.3** Press the blue calibrate button.

**3.2.4** Enter the value of the standard.

**3.2.5** Accept the new calibration number.

## 4.0 Precautions

**4.1** Check condition of the probes frequently between sampling.

**4.2** Don't force pins into connectors, note the keying sequence.

## 6.0 References

Hydrolab Scout 2 Reference Manual, December 1991.

# STANDARD OPERATING PROCEDURE 044
# ASSESSMENT OF EXISTING WELLS USING DOWNHOLE GEOPHYSICS

***SOP being drafted***

July, 1993

# STANDARD OPERATING PROCEDURE 045
# ASSESSMENT OF TIDAL EFFECTS ON GROUND-WATER

***SOP being drafted***

July, 1993

# APPENDIX B

# APPENDIX B
# ICF KE PERSONNEL QUALIFICATIONS

**Michael C. Elias** joined ICF Kaiser Engineers in 1991. He holds an M.S.P.H. in Environmental Chemistry and Biology from the University of North Carolina, Chapel Hill and an M.A. in Aquatic Biology from the University of California, Santa Barbara. His background encompasses environmental toxicology, aquatic biology, ecology, and public health. While at ICF Kaiser Engineers, Mr. Elias has focused on conducting ecological and human health risk assessments of hazardous waste sites, developing site-specific sampling plans, designing bioassessment plans, and performing environmental impact assessments for proposed activities. Prior to joining ICF Kaiser Engineers, Mr. Elias worked at the University of North Carolina Wastewater Research Center. His research consisted of evaluating the effect of environmental variables on the validity of aquatic toxicity tests. Mr. Elias also worked at the University of California Marine Science Institute where he conducted research in the Antarctic to determine the effect of selected environmental parameters on the survival, growth, and development of the Antarctic krill.

**Dr. Gary McKown** has more than 25 years of management and multidisciplinary technical experience in projects dealing with environmental contamination, RCRA compliance activities, CERCLA site investigations, chemical and physical analysis of hazardous materials, hazardous materials research, and quality assurance of environmental programs. He is a Vice President in ICF Kaiser Engineers, and has been with the company since 1985. For the past 11 years, Dr. McKown has focused on hazardous waste management studies at federal facilities and industrial plants. He has managed large-scale remedial investigations and feasibility study projects, remedial design and cleanup tasks, and other work dealing with hydrogeologic site characterization, environmental fate and transport of hazardous materials, exposure and endangerment assessments, sampling and chemical analysis for toxic and hazardous constituents in wastes and environmental media, contaminant transport and atmospheric/soil/water partitioning modeling, and evaluation of remedial technologies and corrective strategies. He also has extensive experience in providing technical support to development of regulations and guidance documentation.

**Andy Peters** has more than 15 years of experience in the investigation, handling, transport, storage, and disposal of radioactive and hazardous materials and waste in both the military and private industry. Mr. Peters currently serves as the full-time project health and safety officer for ICF KE's contract with EPA to provide Environmental Service Assistance Teams (ESAT) for Regions 4, 6, 7, 8, 9, and 10. With collateral duties as the Health and Safety Officer for the Southeast Region of ICF KE's Environment Group (EG), he is responsible for all aspects of health and safety support and program execution for six offices in four states. Mr. Peters' responsibilities include developing or approving site-specific health and safety plans, conducting health and safety training, coordinating employee medical examinations, conducting project health and safety inspections, providing general health and safety technical support, and monitoring compliance with all applicable State and Federal occupational safety and health regulations. He administers ICF KE's health and safety program to approximately 200 employees engaged in laboratory analysis, environmental audits, risk assessment, and hazardous waste site work. Mr. Peters has experience as a site health and safety officer dating to 1981 and has written, reviewed, or approved approximately 150 site-specific health and safety plans (HSPs) for hazardous waste site remediation projects throughout the U.S. in addition to managing or participating in over 22 different field studies. He is experienced in recognizing, evaluating, and controlling workplace and environmental hazards; selecting, evaluating, using, and decontaminating personal protective and monitoring equipment; responding to accidents and other emergencies; and decontaminating personnel. He is also trained and experienced in health physics to include radiation protection, control measures, measurement and detection, surveys and monitoring, dose and exposure limits, and emergency procedures.

**Larry Thebeau** is a Senior Project Manager in the Abingdon, Maryland office. For the past two years he has been managing four major biological/ecological risk assessment projects under the Army Total Environmental Project Support (TEPS) contract at Aberdeen Proving Ground, Maryland. Mr. Thebeau is one of ICF KE's more accomplished field biologists. He has fourteen years of experience in

the environmental field, with twelve of these in environmental consulting. In 1989, Mr. Thebeau was the field team leader, oil spill expert and the senior biological investigator on a project relating to the EXXON VALDEZ oil spill. For the past eleven years, his experience has primarily been in the area of hazardous waste site cleanup and assessment, oil and hazardous material spill response, response contingency planning, and incident damage assessment. He joined ICF Kaiser Engineers in 1987 as a Senior Associate and was the Assistant Field Investigation Team Office Manager (AFITOM) for the Field Investigation Team (FIT) contract. Mr. Thebeau was also the Deputy Program Manager for the ARCS VI, VII, VIII contract and the Subcontractor Program Manager for the TES X contract; both multi-year, multi-million dollar contracts. As the ICF KE Contract Manager of the FIT contract, Mr. Thebeau coordinated all field activities, tracked the status of each project, conducted onsite QA/QC evaluation, and reviewed sampling plans. In addition, he was involved in conducting Preliminary Assessments, Site Investigations and preparing Hazardous Ranking System packages. Mr. Thebeau was the assistant site manager on a RI/FS at a Texas NPL site. Mr. Thebeau has been managing projects and professional personnel for the past nine years. For two years prior to joining ICF Technology he was the assistant regional manager of the Technical Assistance Team (TAT) contract to the EPA. He directly supervised 12 multidisciplinary field personnel and coordinated the activities of two satellite offices. Mr. Thebeau was the TAT project manager on several hazardous waste site immediate removal actions in Region VI. In addition, he has been involved in over 150 hazardous material and oil spill responses.

**R. J. Neubauer** Mr. Neubauer is a Scientist III with three years of experience in estuarine and environmental science, particularly in the areas of estuarine and coastal invertebrate ecology, hazardous waste site assessment and estuarine management. He holds a B.S. with honors in Biology from Towson State University, MD and an M.A. in Marine Science from the Virginia Institute of Marine Science, College of William and Mary, VA. Mr. Neubauer is currently working under a U.S. Army contract involving the human health and ecological/biological risk assessment of several terrestrial and aquatic study areas on the military installation. Mr. Neubauer's primary responsibilities are designing and conducting ecological investigations for the Risk Assessment of several creeks and sub-estuaries of the Chesapeake Bay that are on a U.S. Army Installation. Before joining ICF Kaiser Engineers Mr. Neubauer worked for the Chesapeake Bay Estuary Program of the U.S. Fish and Wildlife Service. Other pertinent experience includes a number a field surveys of the marine and estuarine benthos through a variety of methods as well as the technical review of several Chesapeake Bay Program documents. Mr. Neubauer has participated in numerous field and laboratory studies concerning marine and estuarine environments, and is skilled in the collection of sediments, surface water, soil, and ground water. Mr. Neubauer has professional training in the delineation of jurisdictional wetlands based on methods consistent with the U.S. Army Corps of Engineers. Mr. Neubauer is also experienced in the identification of marine and freshwater plants, invertebrates, and vertebrates. Mr. Neubauer is certified as a PADI Advanced Open Water Diver and has over 200 dives for research of coastal environments. Mr. Neubauer is also NOAA Nitrox (mixed gas) certified.

**Diane D. Wisbeck** B.S., Chemistry, University of California, Davis, 1992. Since Ms. Wisbeck joined ICF KE 3 years ago, she has been responsible for the development and implementation of Quality Assurance Project Plans (QAPP) under USAEC, USEPA and relevant state requirements. Experienced in CLP data validation under the *National Functional Guidelines* with Region III modifications, Ms. Wisbeck oversees the validation effort for groundwater, surface soil, sediment, and surface water data collected and analyzed under USEPA CLP methodology.

# APPENDIX C

## APPENDIX C
## ESE LABORATORY'S SOP for the Analysis of TCPU

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

**TITLE: DETERMINATION OF TRICHLOROPHENYL UREA (TCPU) IN SEDIMENTS**

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Effective Date: 4/28/94

Prepared by: Bradley A. Weichert — Bradley A Weichert 3/28/94

Reviewed by: Preston F. Dumas — 3-28-94

Reviewed by: Michael G. Winslow — 3/28/94

Approved by: John M. Mousa (Gainesville Laboratory Director) — 3/28/94

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**TITLE: DETERMINATION OF TRICHLOROPHENYL UREA (TCPU) IN SEDIMENTS**

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedure used to determine Trichlorophenyl Urea sediments by High Performance Liquid Chromatography (HPLC).

## 2.0 SCOPE AND APPLICATION

This SOP is applicable to the determination of TCPU in environmental soil and sediment samples.

## 3.0 SUMMARY OF METHOD

Low Level Procedure: A 5 g dry weight of the sample is placed in a scintillation vial and extracted by sonication with 10 mL of 5% dimethylformamide (DMF) in acetonitrile (v/v) for 60 minutes.

High Level Procedure: A 1 g dry weight of the sample is placed in a 40 mL amber vial and extracted by sonication with 20 mL of 5% dimethylformamide (DMF) in acetonitrile (v/v) for 60 minutes.

After extraction sample extract is filtered through a 0.45 uM Acrodisc filter. The eluate is collected in a 6 mL shorty vial and stored at ambient temperature until analysis by HPLC.

## 4.0 APPARATUS AND MATERIALS

4.1 Vial: 20 ml scintillation (Kimble or equivalent).

4.2 Pipet: 10 mL Class A volumetric.

4.3 Pipet: micro, 25, 50, 100, 200, 250 uL.

4.4 Vial: "shorty" 6 mL (Wheaton or equivalent).



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4.5 Vial: amber glass, 1.6 mL capacity with open cap and Teflon septum (Natl. Scientific Co. C4013-2 or equivalent).

4.6 Vial: amber glass, 40 mL capacity with Teflon-lined cap.

4.7 Transfer pipets: Pasteur 2 mL.

4.8 Filter: 0.45 uM pore size PTFE membrane (Gelman Acrodisc or equivalent).

4.9 Syringe: 10 mL gas-tight, Luer-lock fitting (Hamilton 1010LT or equivalent).

4.10 Cleanup cartridge: C18 (ODS) cartridge to fit gas-tight syringe (Waters Sep-Pak or equivalent).

4.11 Balance: analytical, calibrated daily with NIST certified weights and capable of accurately weighing 0.0001 grams.

4.12 Bottles: amber glass, screw top closure with Teflon-lined caps, 12 and 30 mL volumes (Kimble 60815 and Wheaton 220093 or equivalents).

4.13 Mixer: Vortex-Genie (Scientific Products Model S8223 or equivalent).

4.14 Ultrasonic bath: Bransson model 3200 or equivalent.

4.15 Wash bottle: PTFE Teflon, 500 mL.

4.16 Labels: adhesive, waterproof for sample and standard bottles.

4.17 HPLC equipment

4.17.1 Autosampler: capable of multiple injections at 25 uL with high reproducibility, event out signal for start of data collection and detector autozero. Shimadzu SIL-6A or equivalent.

4.17.2 Pump system controller: capable of executing control of the HPLC pump and electronically connected to the autosampler (Shimadzu SCL-6A or equivalent).



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4.17.3 HPLC pump: unit capable of delivering precise flow at 1.0 mL/minute and equipped with an inline filter of 0.2 micron pore size (Shimadzu LC-6A or equivalent).

4.17.4 Detector: variable wavelength UV, set at 245 nM (Shimadzu SPD-6A or equivalent).

4.17.5 HPLC column: 4.6 x 150 mm ODS, 5 micron particle size.

4.18 Reagents

4.18.1 Acetonitrile(AcN): B&J brand UV or equivalent.

4.18.2 N,N-Dimethylformamide(DMF): EM Science OmniSolv or equivalent.

4.18.3 Water: HPLC grade, B&J brand or equivalent.

4.18.4 5% DMF in AcN: 5 mL of DMF added to 95 mL of AcN, v/v.

## 5.0 METHOD INTERFERENCES

5.1 To reduce interference the glassware should be thoroughly cleaned prior to use with hot water and detergent, followed by thorough rinsing with deionized water and solvent.

5.2 Matrix effects and interferences cannot be predicted due to the variety of sample sources encountered and the associated contaminants contained therein. Additional cleanup procedures may be deemed necessary on a case by case basis.



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## 6.0 SAFETY PRACTICES

TCPU should be treated as a potential health hazard. TCPU has not been tested for mammalian toxicity but the oral LD50 in rats is greater than 100 mg/kg (see reference 12.1). Acetonitrile is a flammable liquid with toxicity associated with ingestion by inhalation, orally, and dermal contact. N,N-Dimethylformamide is a skin, eye, and respiratory organ irritant and can cause adverse health effects from acute exposure. Appropriate eye, skin, and respiratory protection is required when working with these materials.

## 7.0 SAMPLE PRESERVATION AND HOLDING TIMES

No chemical preservation is necessary. Samples are preserved for shipping by cooling to 10° C, and frozen once received by the laboratory. Holding times are 14 days from collection until extraction, 40 days from extraction until analysis (default holding times for soil/sediments since no degradation/stability studies have been documented).

## 8.0 STANDARD PREPARATION

8.1 The solubility of TCPU in DMF is approximately 800 ug/mL. Separate stock standards are prepared for calibration, spiking, and calibration verification (CCV). 20 mg of TCPU is weighed into a tared 25 mL volumetric flask and dissolved in DMF. The stock solution for the CCV may be of lesser concentration to conserve the neat standard material.

8.2 The calibration levels are prepared in 90/10 AcN/DMF (v/v) as follows:

0.5 mL of stock, 800 ug/mL, to 10 mL = 40 ug/ml ("C5" level)

0.2 mL of stock, 800 ug/mL, to 10 mL = 16 ug/mL ("C2" level)

0.5 mL of C5 level, 40 ug/mL, to 5.0 mL = 4.0 ug/ml ("D5" level)

0.5 mL of C2 level, 16 ug/mL, to 5.0 mL = 1.6 ug/mL ("D2" level)

0.5 mL of C2 level, 16 ug/mL, to 10 mL = 0.80 ug/mL ("D" level)

0.5 mL of D5 level, 4.0 ug/mL, to 5.0 mL = 0.40 ug/mL ("E5" level)



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## 9.0 PROCEDURE

### 9.1 LOW LEVEL EXTRACTION PROCEDURE

9.1.1 Spread out a sufficient portion of the sample (enough to obtain a 20 g dry weight) on a sheet of aluminum foil and allow to air dry 24-48 hours under a hood. If water is overlaying the sample decant and discard the water layer before removal of the sediment subsample.

9.1.2 Remove any foreign material (sticks, stones, roots, etc.) and crush and homogenize the air-dried sample. Weigh out a representative amount of the sample and place in a convection oven at 105 degrees C for moisture determination. Weigh 5.0 grams of air-dried sample into a scintillation vial.

9.1.3 Add 10 mL of 5% dimethylformamide (DMF) in acetonitrile (AcN) to the sample and vortex for 30 seconds. Place the sample in a chilled sonicator for 60 minutes.

9.1.4 Allow the sample to settle and centrifuge the sample at a RPM setting appropriate for the scintillation vial.

9.1.5 For highly colored extracts there is a high probability of matrix interference. If the extract is highly colored, proceed to step 9.1.7.

9.1.6 Filter 5.0 mL of the extract through a 0.45 uM Acrodisc filter and collect the eluate in a 6 mL shorty vial. Extracts are stored at ambient temperature due to the low solubility of the analyte in the extract solvent and the very high stability to chemical hydrolysis.

9.1.7 **OPTIONAL CLEANUP PROCEDURE:** 1) prepare a C18 Sep-Pak cartridge by passing 5 mL of the 5% DMF solution through it, discarding the eluate, 2) filter 5.0 mL of sample extract through the prepped cartridge, collecting the eluate in 6 mL shorty vial, 3) sample extract is now ready for HPLC analysis.



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9.2 HIGH LEVEL PROCEDURE

A 1 gram portion of the air-dried sample is weighed into a 40 mL amber vial and extracted by sonication with 20 mL of 5% DMF in AcN for 60 minutes using the same procedure as the low level extraction (steps 9.1.3-9.1.7).

9.3 HPLC ANALYSIS

9.3.1 Conditions and apparatus: 4.6 x 150 mm ODS column, 60/40 acetonitrile/water (v/v) at 1.0 mL/minute, UV at 245 nM, 25 uL injection volume, retention time of approximately 4 minutes.

9.3.2 Calibrate system with a standard curve range of 0.4-40 ug/mL (six standard levels) and linear fit curve. Verify the curve with an independently prepared standard. Run the highest level standard from the curve as a continuing calibration. NOTE: the 40 ug/mL standard requires greater than 5% DMF in AcN to prevent the TCPU from precipitating.

9.3.3 All samples with levels higher than 20 ug/g are to be reextracted using the high level procedure and appropriate QC.

## 10.0 CALCULATIONS

10.1 Summary

The target responses are transferred to the Laboratory Data Management System, CLASS (Chemical Laboratory Analytical and Scheduling System), along with any relevant sample information. The concentration is calculated using the regression equation calculated by CLASS. Final sample results are corrected for sample volume, extract volume, dilution factors and any applicable conversion factors.



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10.2 Peak Identification

Analyte response that lies within the established retention time windows will be considered to be tentatively identified. Analyst must use their judgement as to whether the peak may represent a target compound by examining such factors as peak shape, resolution from interferences and matrix "noise".

10.3 Analyte Quantitation

Peak areas or heights are used to calculate analyte concentrations.

## 11.0 QUALITY CONTROL

11.1 Low level procedure: One spike at approximately 5X the DL and replicate spikes near the upper limit (40 ug/g) will be performed for the low level procedure. The TCPU spike solution stock, nominal concentration of 800 ug/mL, is diluted 1:4 with 50/50 DMF/AcN v/v for a 200 ug/mL solution. 5 grams of USAEC standard soil is spiked with 0.1 mL of the 200 ug/mL TCPU solution for a target of 4 ug/g. The replicate high spikes are 0.1 mL of the 800 ug/mL TCPU spike stock solution into 5 grams of USAEC standard soil for a target of 16 ug/g. Matrix spike replicates will be done at the high control spike level using the same spiking procedure. One set of matrix spike replicates will be done for every 20 samples.

11.2 High level procedure: Control spikes for the high level procedure are one spike at the low level (0.1 mL of the 200 ug/mL TCPU solution into 1 gram of USAEC standard soil for a target of 20 ug/g) and replicate high spikes at 160 ug/g (0.2 mL of the 800 ug/mL TCPU spike stock solution into 1 gram of USAEC standard soil). Matrix spike replicates will be done at the high control spike level using the same spiking procedure.

11.3 For each lot of samples, extract and analyze the following QC required unless specified differently by the client:

5% Method Blank
5% Standard Matrix Spike
5% Sample Matrix Spike
5% Sample Matrix Spike Duplicate



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Note: The number of QC required is the actual number of QC samples rounded up to the nearest whole number, i.e, 5% = 1 QC for 1-20 samples; 2 QC for 21-40 samples, etc.

## 12.0 REFERENCES

12.1 Technical report 8211 - USAMRADC, Ft. Detrick, MD 21701 "An Investigation Of The Presence Of N,N'-bis(2,4,6-trichlorophenyl) Urea In Estuarine Sediments Of Aberdeen Proving Ground, MD (Edgewood Area)"; Dennis, William H. Jr.; Sc. D.; U.S. Army Medical Bioengineering Research and Development Laboratory.



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# APPENDIX D

# APPENDIX D
# ESE LABORATORY'S USAEC CERTIFIED METHODOLOGIES

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AAA9 Analysis of Isopropylmethylphosphonic acid, Methyl phosphonic acid, and Fluoroacetic acid in soils and sediments

TT9 Diisopropylmethylphosphonic acid and Dimethylmethylphosphonate in Environmental Soil

LW12 Explosives in Soil by High Pressure Liquid Chromatography

LW18 Determination of Thiodiglycol and Choracetic Acid in Soil by Gas Chromatography

UT02 Isopropylmethylphosphonic acid, Methylphosphonic acid and Fluoroacetic acid in water

T8 Diisopropylmethylphosphonic acid and Dimethylmethylphosphonate in Environmental Water Samples

UW32 Determination of Explosives in Water by High Pressure Liquid Chromatography

UW22 Analysis of Thiodiglycol and Thiodiglycolic acid in Environmental Water Samples

LL03 Determination of Organosulfur Compounds in Soil by Gas Chromatography

UL04 Determination of Organosulfur Compounds in Water by Gas Chromatography

USATHAMA METHOD AAA9

# ANALYSIS OF ISOPROPYLMETHYL PHOSPHONIC ACID, METHYL PHOSPHONIC ACID, AND FLUOROACETIC ACID IN SOILS AND SEDIMENTS

# USATHAMA METHOD AAA9
# ANALYSIS OF ISOPROPYLMETHYL PHOSPHONIC ACID, METHYL PHOSPHONIC ACID, AND FLUOROACETIC ACID IN SOILS AND SEDIMENTS

## 1. APPLICATION

This method was developed for the quantitative determination of the following compounds in soils and sediments:

- ✓Isopropylmethyl Phosphonic Acid (IMPA)
- Fluoroacetic Acid (FC2A)
- Methyl Phosphonic Acid (MPA)

### A. TESTED CONCENTRATION RANGES

The tested concentration ranges of the compounds examined in standard soil are as follows:

| Analyte | Tested Concentration Range (ug/g) |
|---|---|
| IMPA | 2.0 to 40 |
| FC2A | 2.0 to 40 |
| MPA | 2.0 to 40 |

Note: ug/g = micrograms per gram.

### B. SENSITIVITY

The approximate peak areas in microvolt-minutes (uV-min) [with the conductivity detector on 10 microSiemens per volt (uS/V) scale] for 2.0-ug/g standard Rocky Mountain Arsenal (RMA) soil spikes are:

| Analyte | Peak Area (uV-min) | Approximate Retention Times (min) |
|---|---|---|
| IMPA | 50,000 | 5.8 |
| FC2A | 70,000 | 6.6 |
| MPA | 50,000 | 9.7 |

Note: min = minutes.

C. CERTIFIED REPORTING LIMITS

The certified reporting limits in standard soil, calculated according to the United States Army Toxic and Hazardous Materials Agency (USATHAMA) reporting limit program with 90-percent confidence limits (USATHAMA, 1985), are:

| Analyte | Reporting Limit (ug/g) | Upper Certification Range (ug/g) |
|---|---|---|
| IMPA | 2.1 | 40 |
| FC2A | 2.0 | 40 |
| MPA | 2.0 | 40 |

D. INTERFERENCES

A compound that coelutes with an analyte is an interference. Formate (formic acid) shares a similar retention time with FC2A. Currently, no other commonly occurring substance is known to coelute with any of the three analytes. High concentrations of transition metals can cause low recovery of IMPA and MPA.

E. One analyst can extract and analyze approximately 12 samples in an 8-hour day.

2. CHEMISTRY

A. CHEMICAL ABSTRACT SERVICE (CAS) NUMBERS

The CAS registry numbers for the compounds are:

| Analyte | CAS Registry No. |
|---|---|
| IMPA | NA* |
| FC2A | 62-74-8 |
| MPA | NA |

*Not currently available.

B. CHEMICAL REACTION

A measured weight of soil/sediment is extracted with deionized water (DIW) using a mechanical shaker. The water extract is centrifuged and filtered through a 0.45-micrometer (um) filter. The extract is then analyzed by gradient ion chromatography. Chromatographic conditions are described which permit the separation and measurement of the parameters in the extract. Qualitative identification is performed using retention time, and quantitative analysis is performed using a standard curve of concentration versus area counts.

3. APPARATUS

A. INSTRUMENTATION

1. Ion chromatograph (Dionex Model 4000, with gradient analytical pump capabilities),
2. Anion separator column (Dionex AS4-A),
3. Anion guard column (Dionex AG4-A),
4. Anion membrane suppressor (Dionex AMMS),
5. Integrator (Maxima® integration system or equivalent), and
6. Anion trap column (Dionex ATC).

B. HARDWARE/GLASSWARE

1. Mechanical shaker;
2. 50-milliliter (mL) disposable plastic centrifuge tubes;
3. Assorted Class A volumetric flasks and pipettes; and
4. 25-millimeter (mm), syringe-mounted, disposable filter (0.45-um pore size, polyester filter).

C. CHEMICALS/REAGENTS

1. Sodium hydroxide [5 normal (N), prepared from carbonate-free, Dilut-It® analytical concentrate (Baker)].
2. Sulfuric acid [Baker, American Chemical Society (ACS), reagent-grade].

3. Standards [Standard Analytical Reference Materials (SARMs), when available]: IMPA (SARM Compound 1264); FC2A (no SARM currently available); MPA (SARM Compound 1390).
4. DIW (18-megaohm purity)..

4. STANDARDS

A. INSTRUMENT CALIBRATION STANDARDS

1. Stock Calibration Standards

Prepare 4,000-microgram-per-milliliter (ug/mL) stock standards by weighing 200 milligrams (mg) of each standard material into 50-mL volumetric flasks and diluting to volume with DIW. Prepare fresh at least semiannually and store at 4 degrees Celsius (°C).

2. Combined Intermediate Standard

Prepare a 100-ug/mL combined intermediate standard by adding 2.5 mL of each stock to a 100-mL volumetric flask and diluting to volume with DIW. Prepare fresh at least quarterly, and store at 4°C.

3. Working Calibrations Standards

Working calibration standards are prepared fresh for each lot as follows:

| Standard | Concentration (ug/L) | Volume (mL) of Combined Standard Diluted to 100 mL with DIW |
|---|---|---|
| Blank | 0 | 0 |
| A | 200 | 0.2 |
| B | 400 | 0.4 |
| C | 1,000 | 1.0 |
| D | 2,000 | 2.0 |
| E | 4,000 | 4.0 |
| F | 6,000 | 6.0 |

Note: ug/L = micrograms per liter.

B. PREPARATION OF CONTROL SPIKES FOR METHOD CERTIFICATION

1. Stock Control Spike Solutions

Prepare 4,000-ug/mL stock standards (independent of stock calibration standards) as documented in Sec. 4.A.1. Prepare fresh at least semiannually and store at 4°C.

2. Combined Intermediate Control Spike Solution

Prepare a 100-ug/mL combined intermediate control spike solution by adding 2.5 mL of each control spike stock to a 100-mL volumetric flask and diluting to volume with DIW. Prepare fresh at least monthly and store at 4°C.

3. Working Control Spike Solutions

Working control spike solutions are prepared as follows:

| Control Spike | Concentration (ug/mL) | Volume (mL) of Combined Intermediate Control Spike Solution Diluted to 50 mL with DIW |
|---|---|---|
| A | 0 | 0 |
| B | 4 | 2 |
| C | 8 | 4 |
| D | 16 | 8 |
| E | 40 | 20 |
| F | 80 | 40 |

C. DAILY INSTRUMENT CALIBRATION

A series of six calibration standards (A through F, as prepared in Sec. 4.A.3) is analyzed at the beginning of each day or analytical run using the procedure described in Sec. 5. Calibration Standard E is reanalyzed at the end of the day or analytical run. The response of Calibration Standard E at the end of the day must agree with the previous response within ±15 percent; if this criterion is not met, either the lot is rerun or an adequate explanation is provided to support the reason that this situation does not affect the quality of the data reported for this lot.

D. DAILY CONTROL SPIKE SAMPLES

With each daily lot of environmental samples, prepare and analyze the daily control spike samples as follows (prepare as documented in Sec. 5.B).

| Daily Control Spike | Spike Concentration (ug/g) | Working Control Spike Solution (Sec. 4.B.3) |
|---|---|---|
| Blank | 0 | A |
| Low Spike | 4 | C |
| High Spike 1 | 20 | E |
| High Spike 2 | 20 | E |

5. PROCEDURE

A. EXTRACTION PROCEDURE

Weigh a 2.00-gram (g) sample into a 50-mL disposable plastic centrifuge tube. Add 20.0 mL DIW and shake on a mechanical shaker for 30 min. Centrifuge for 15 min and filter through a 0.45-um filter.

B. CONTROL SPIKES FOR METHOD CERTIFICATION

Weigh 2.00 g of RMA standard soil into separate 50-mL disposable plastic centrifuge tubes. Add 1.0 mL of each working control spike solution (A through F) (Sec. 4.B.3) to each 2.0-g standard soil sample as shown in this section. Let set 1 hour before adding 20.0 mL of DIW. Shake for 30 min; centrifuge for 15 min and filter through a 0.45-um filter.

| Spike Level | Spike Concentration (ug/g) | Concentration of Working Control Spike Solution Added to 2.0 g Standard Soil (ug/mL) |
|---|---|---|
| 0X | 0 | 0 (A) |
| 0.5X | 2 | 4 (B) |
| X | 4 | 8 (C) |
| 2X | 8 | 16 (D) |
| 5X | 20 | 40 (E) |
| 10X | 40 | 80 (F) |

C. ELUENT PREPARATION

Eluents must be prepared with a minimum amount of carbonate contamination. Carbonate has a higher eluting strength than hydroxide and will give poor and nonreproducible results if allowed to contaminate eluents. A carbonate-free eluent stock must be used. Eluent water must be degassed with helium prior to use and prepared eluents kept under helium to prevent adsorption of carbon dioxide from air.

1. a. A 0.5-millimolar (mM) sodium hydroxide eluent (eluent No. 1) is prepared by adding 200 uL of 5-molar (M) sodium hydroxide (Dilut-It®, carbonate-free) to 2 liters (L) of DIW which has been degassed previously with helium for 5 min.

   b. A 98-mM NaOH eluent (eluent No. 2) is prepared by adding 20 mL of 5-M NaOH to 1 L of degassed DIW.

2. Regenerant [approximately 40-millinormal (mN) sulfuric acid] is prepared by adding 4.5 mL of concentrated sulfuric acid to 4 L of DIW.

D. INSTRUMENT PARAMETERS

1. Columns: Noted in Sec. 3.A.
2. Eluents: 0.5-mM and 100-mM sodium hydroxide.
3. Suppressor regenerant: 40-mM sulfuric acid.
4. Flow rate and pressure: 1.5 mL/min at 750 pounds per square inch (psi) (typical).
5. Detector range: 10 uS full-scale.
6. Injection loop: 100 uL.
7. Gradient program: Enter the following program into the gradient pump microprocessor.

| Time | Eluent | Percent | Flow (mL/min) |
|---|---|---|---|
| 0.0 | 1 | 100 | 1.5 |
| 0.1 | 5* | 1 (Inject) | 1.5 |
| 0.2 | 5 | 0 (Inject off) | 1.5 |
| 2.0 | 1 | 100 | 1.5 |
| 8.0 | 2 | 65 | 1.5 |
| 10.0 | 2 | 65 | 1.5 |
| 10.0 | 1 | 100 | 1.5 |

*Note: Port 5 on the gradient pump module becomes the injection value when operating a system without an advanced chromatography module.

8. Total run time (time between injections) should be at least 15 min to allow column to equilibrate with the 0.5-mM sodium hydroxide eluent.
9. Anion trap column (ATC): The function of the ATC is to remove trace contaminants from the eluents which would otherwise collect on the separator column until a buildup is reached in the gradient run and they are pushed off in broad peaks.

   Prepare the ATC according to manufacturer's instructions, using 1-M NaOH to convert the resin from its chloride form to a hydroxide form. The ATC is placed in line between between the pump and injection valve.

E. STARTUP PROCEDURE FOR DIONEX MODEL 4000i ION CHROMATOGRAPH

1. Establish a stable baseline with eluent No. 1 (0.5-mM sodium hydroxide) at 100 percent (20 to 30 min).
2. With integrator ready to receive trigger signal from ion chromatograph (due to narrow peak windows, manual start of integrator is not recommended), inject high standard (Standard A).

3. Upon completion of Standard A run, test integration parameters for correct baseline placement and proper peak starts and peak ends.
4. Proceed with calibration standard run, from high to low, and a blank. Allow 15 min between injections.
5. Analyze method blank and daily control spike samples (Note: analyze certification spike sample, Sec. 5.B., if performing certification).
6. Proceed with analysis of extracted samples; dilute samples that exceed the upper certified range of the method with DIW and reanalyze.
7. Reanalyze Standard E at end of analytical run.

6. CALCULATIONS

Calculate the linear regression equation for response (peak area) versus concentration using the least-squares method. Verify correlation coefficients are >0.995 for the regression equations. Using the linear regression equation, compute concentration for extract analytes.

Preliminary results (ug/g) =

$$\frac{\text{Solution Conc (ug/L) x Extract Vol (L) x Dilution Factor}}{\text{Sample Weight (g) x (100-Percent Moisture/100)}}$$

Preliminary results will be adjusted for percent moisture and for accuracy obtained during method certification to obtain final results.

7. REFERENCE

Dionex Model 4000i Instrument Operation Manual. The Practice of Ion Chromatography, Frank Smith, Jr., and Richard C. Chang.

8. CERTIFICATION DATA

See Att. 1.

9. CALIBRATION DATA

See Att 2.

10. CHROMATOGRAM

See Att. 3.

Environmental Science & Engineering, Inc.
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TITLE: DETERMINATION OF DIISOPROPYLEMETHYLPHOSPHONATE AND DIMETHYLEMETHYLPHOSPHONATE IN ENVIRONMENTAL SOIL SAMPLES (METHOD TT9)

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TITLE: DETERMINATION OF DIISOPROPYLEMETHYLPHOSPHONATE AND DIMETHYLEMETHYLPHOSPHONATE IN ENVIRONMENTAL SOIL SAMPLES (METHOD TT9)

## I. APPLICATION

This method is applicable to the quantitative determination of the following compounds in environmental soil samples:

Diisopropylmethylphosphonate (DIMP)
Dimethylmethylphosphonate (DMMP)

### A. TESTED CONCENTRATION RANGE

The tested concentration ranges in "standard soil" samples are:

| Analyte | Tested Concentration Range (µg/g)* |
|---|---|
| DIMP | 0.114 to 4.57 |
| DMMP | 0.105 to 4.18 |

*µg/g = micrograms per gram.

### B. SENSITIVITY

The normalized responses (integrator counts corrected for attenuation) at the standard soil detection limits (Section I.C) are:

| Analyte | Area Count |
|---|---|
| DIMP | 10,900 |
| DMMP | 12,000 |

### C. DETECTION LIMITS

The "standard soil" detection limits, calculated according to the U.S. Army Environmental Center (USAEC) detection limit program are:

| Analyte | Detection Limit (µg/g) | Range (µg/g) |
|---|---|---|
| DIMP | 0.114 | 4.57 |
| DMMP | 0.133 | 4.18 |

### D. INTERFERENCES

Reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be free from interferences which have retention times equal to the retention times of the compounds of interest.

### E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 samples in an 8-hour day.

## II. CHEMISTRY

### A. CHEMICAL ABSTRACT SERVICE (CAS) NUMBERS

The CAS registry numbers for the compounds are:

| Analyte | CAS Registry Number |
|---|---|
| DIMP | 1445-75-6 |
| DMMP | 756-79-6 |

### B. CHEMICAL REACTIONS

In an amber glass vial, a measured weight of sample is extracted with distilled water. Chromatographic conditions are described which permit the separation and measurement of DIMP and DMMP in "standard" or environmental soil. Qualitative identification is performed using retention times, and quantitative analysis is performed using standard curves.

## III. APPARATUS

### A. INSTRUMENTATION

A HP 5890 gas chromatograph (GC) with a flame photometric detector (FPD) equipped with a HP 7672 automatic sampler and interfaced to a PE Nelson 2700 Turbochrom Data System (or equivalent).

### B. PARAMETERS

1. Instrument: HP 5890A equipped with an autosampler (Model 7672)

2. Detector: FPD with selective spectral detection of phosphorus [530-nanometer (nm) filter]

3. Column: 5-percent SP-1000 on 100/120 supelcoport glass column [2-meter (m) x 2-millimeter (mm) inside diameter (ID)]

4. Gas flow:
   Helium--50 milliliters per minute (mL/min)
   Hydrogen--100 mL/min
   Air 1--120 mL/min

5. Temperature:
   Injector--200 degrees Celsium (°C)
   Detector--245°C
   Oven--60°C (no hold), then programmed at 10 degrees Celsius per minute (°C/min) to 100°C, then 50°C/min to 240°C, hold for 5.2 minutes.

6. Injection volume: 5.0 microliters (µL)

7. Retention times:

| Analyte | Retention Time (min) |
|---|---|
| DIMP | 5.5 ± 0.2 |
| DMMP | 6.1 ± 0.2 |

Daily retention times will be determined based on initial analysis of Standard B.

### C. HARDWARE/GLASSWARE

1. Volumetric flasks [10-, 50-, and 100-milliliter (mL)];
2. Volumetric pipettes (1.0- and 2.0-mL);
3. Microsyringes (100- and 1,000-uL);
4. Pasteur pipettes (disposable);
5. Amber glass vials (8-mL with Teflon-lined screw caps);
6. Glass vials (2-mL with Teflon-lined, crimp-seal caps for use with an automatic sampler);
7. Amber bottles (60 mL with Teflon-lined screw caps);
8. Stainless steel spatulas; and
9. Analytical balance [Mettler AE160 or equivalent, with 0.0001-gram (g) sensitivity].

### D. CHEMICALS

1. Distilled water;
2. "Standard soil" (an uncontaminated natural soil received from Rocky Mountain Arsenal);
3. DIMP [Standard Analytical Reference Material (SARM), identification No. PA2334, obtained from USATHAMA]; and
4. DMMP (SARM, identification No. PA1827, obtained from USATHAMA).

## IV. STANDARDS

### A. INITIAL INSTRUMENT CALIBRATION STANDARDS

1. Individual stock calibration standards are prepared by weighing approximately 50 milligrams (mg) each of DIMP and DMMP into separate 50-mL volumetric flasks, then diluting to volume with distilled water. The actual concentrations in the stock calibration standards prepared in the above manner were:

| Analyte | Concentration of Individual Calibration Standard (ug/mL) |
|---|---|
| DIMP | 1,134 |
| DMMP | 1,030 |

2. A composite secondary stock calibration standard is prepared by adding 1 mL of each individual stock calibration standard to deionized water in a 10-mL volumetric flask and then diluting to volume with distilled water. The actual concentrations in the composite secondary stock calibration standard prepared in the above manner were:

| Analyte | Concentration of Composite Secondary Stock Calibration Standard (ug/mL) |
|---|---|
| DIMP | 113 |
| DMMP | 103 |

3. Prepare composite working calibration standards using the composite secondary stock calibration standard, microsyringes, and volumetric pipettes as shown in Table 1. Each composite working calibration standard is prepared by adding the specified volume of the composite secondary stock calibration standard to distilled water contained in a 100-mL volumetric flask and then diluting to volume with deionized.

4. For initial instrument calibration, inject 5.0 uL of each of the working calibration standards prepared in Table 1. Plot the linear regression of the normalized response versus the concentration of standard injected for each standard to obtain a working curve. This curve must have a correlation coefficient of 0.995 or greater.

## B. DAILY INSTRUMENT CALIBRATION STANDARDS

1. A minimum of three working calibration standards and one blank are analyzed daily for instrument calibration.

   a. At a minimum, 5.0 uL of each of the following working calibration standards (from Table 1) are analyzed daily:
      (1) Blank,
      (2) Working calibration standard .5A,
      (3) Working calibration standard C, and
      (4) Working calibration standard E.

b. Plot the linear regression of the normalized response versus the concentration of standard injected for each standard to obtain a working curve. This curve must have a correlation coefficient of 0.995 or greater.

c. At the end of the daily instrumental analysis, inject 5.0 uL of working calibration standard E. The response of this end-of-day analysis should be ±25 percent of the response obtained from the analysis of working calibration standard E analyzed earlier in the day. If not, the instrument should be recalibrated and the sample extracts reanalyzed.

Table 1. Preparation of Composite Working Calibration Standards

| Volume of Composite Secondary Stock Calibration Standard Used (mL) | Final Volume (mL) | Composite Working Calibration Standard Prepared | Concentration of Prepared Standard (ug/mL) DIMP | DMMP |
|---|---|---|---|---|
| 0 | 100 | Blank | 0 | 0 |
| 0.050 | 100 | .5A | 0.0567 | 0.0515 |
| 0.100 | 100 | A | 0.113 | 0.103 |
| 0.200 | 100 | B | 0.227 | 0.206 |
| 0.400 | 100 | C | 0.454 | 0.412 |
| 1.00 | 100 | D | 1.13 | 1.03 |
| 2.00 | 100 | E | 2.27 | 2.06 |
| 2.50 | 100 | F | 2.86 | 2.58 |

Source: ESE

2. Retention Time Windows

Retention time windows for a method are established based on historical data of daily performance of the method or by using retention windows from a reference method. If a reference method with established windows is not available the windows are calculated by calculating the relative standard deviation of the retention variation throughout the run for all standards, control samples and continuing

calibration standards and multiplying the result by three and rounded to 1 significant figure. The windows are expressed in absolute minutes where the retention variation is minimal throughout the chromatographic run and as a percentage of the retention time in methods whose variation is proportional to retention time. The retention window is applied to retention times from a specified standard during initial or daily calibration. These retention window values are entered into the method used for processing of the chromatographic data for computerized identification/rejection of detected peaks. The analyst can override the identification/rejection of a peak by providing documentation of his decision.

## C. CONTROL SPIKES FOR METHOD CERTIFICATION

1. A composite stock control spiking solution is prepared by weighing approximately 50 mg of DIMP and 50 mg of DMMP into one 50-mL volumetric flask, then diluting to volume with distilled water. The actual concentrations in the composite stock spiking solution prepared in the above manner were:

| Analyte | Concentration of Composite Stock Control Spiking Solution (ug/mL) |
|---|---|
| DIMP | 1,142 |
| DMMP | 1,046 |

2. A composite secondary stock control spiking solution is prepared by adding 1 mL of the composite stock control spiking solution into a 50-mL volumetric flask, then diluting to volume with distilled water. The actual concentration in the secondary composite stock spiking solution prepared in the above manner was:

| Analyte | Concentration of Secondary Composite Stock Control Spiking Solution (ug/mL) |
|---|---|
| DIMP | 22.8 |
| DMMP | 20.9 |

3. Certification control spike samples are prepared using the composite secondary stock control spiking solution, microsyringes, and volumetric pipettes as shown in Table 2. Each certification control spike sample is prepared by adding the specified volume of the secondary stock control spiking solution to "standard soil" contained in a 60-mL amber glass vial.

### D. DAILY CONTROL SPIKE SAMPLES

With each daily lot of environmental samples, analyze the daily control spike samples shown in Table 3.

## V. PROCEDURE

### A. EXTRACTION OF CERTIFICATION CONTROL SPIKES ONLY

The certification control spikes are extracted beginning with Section 5.C.4.

### B. HANDLING OF ENVIRONMENTAL SAMPLES

1. Soil core sections received from the field in polybutyrate tube sections are subsampled in the laboratory using a stainless steel coring tube inserted lengthwise into the end of the core section. This procedure allows a composite subsample of the entire core length to be taken for analysis.

2. The core subsample is taken from the center of the core section to avoid analyzing soil which has contracted the polybutyrate tube walls. Subsampling using the coring device is repeated until sufficient quantity of soil is removed to perform all the required analyses.

Table 2. Preparation of Certification Control Spike Samples

| Volume of Composite Secondary Stock Control Spiking Solution Used (mL) | Weight of Standard Soil Used (g) | Certification Control Spike Sample Prepared | Concentration of Prepared Sample (ug/g) DIMP | DMMP |
|---|---|---|---|---|
| 0 | 10 | Blank | 0 | 0 |
| 0.050 | 10 | A | 0.114 | 0.105 |
| 0.100 | 10 | B | 0.228 | 0.209 |
| 0.200 | 10 | C | 0.457 | 0.418 |
| 0.400 | 10 | D | 0.913 | 0.837 |
| 1.00 | 10 | E | 2.28 | 2.09 |
| 2.00 | 10 | F | 4.57 | 4.18 |

Source: ESE

Table 3. Preparation of Daily Control Spikes

| Daily Control Spike Number | Volume of Composite Secondary Stock Control Spiking Solution Used (mL) | Weight of Standard Soil Used (g) | Concentration of Prepared Sample (ug/g) DIMP | DMMP |
|---|---|---|---|---|
| Blank | 0 | 10 | 0 | 0 |
| Low-Level | 0.100 | 10 | 0.228 | 0.209 |
| High-Level | 1.00 | 10 | 2.28 | 2.09 |
| High-Level | 1.00 | 10 | 2.28 | 2.09 |

Source: ESE

3. The center core subsamples are transferred to the dull side of clean aluminum foil. This combined subsample is thoroughly mixed using a clean stainless steel spatula and is transferred to a clean glass container with Teflon-lined lid for storage prior to removal of aliquots for analysis.

4. For surface soil samples, the entire contents of the subsample bottle are transferred to the dull side of clean aluminum foil. The subsample is thoroughly mixed with a stainless steel spatula and returned to the original subsample bottle or to a clean glass container for storage prior to removal of aliquots for analysis.

## C. EXTRACTION OF ENVIRONMENTAL SAMPLES

1. Place 10 g of "standard soil" into each of four separate 60-mL amber vials.

2. Spike these four vials as specified in Table 3. Cap and allow to equilibrate for 1 hour.

3. Environmental samples are to be extracted within 7 days of sample collection. Transfer a 10-g portion to a 60-mL amber vial.

4. Add 20 mL of distilled water with a volumetric pipette. Cap tightly.

5. Shake the culture tube in a horizontal position for 4 hours on a wrist-action shaker.

6. Allow the particulate to settle or centrifuge, if necessary (indicate in the laboratory record book whether centrifugation was required).

7. With a disposable pipette, transfer approximately 1 to 2 mL of the extract to an autosampler vial. The extract is now ready for instrumental analysis.

8. Transfer the remainder of the extract to an 8-mL amber glass vial and cap tightly. Save as a backup sample.

9. Store samples at 4 °C ± 2 °C until analysis. Samples must be analyzed within 40 days of sample extraction.

D. ANALYSIS

1. Perform daily instrument calibration as described in Section 4.B.

2. Place the sample extracts in the autosampler tray and inject a 5-uL volume of each sample extract.

## VI. CALCULATIONS

A. Determine the concentration of each component according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(Vt)}{(Ws)}$$

where:
- A = Concentration of each component found in the sample extract by comparison with the qappropriate standard curve (ug/mL),
- Vt = Volume of total extract (mL), and
- Ws = Weight of initial sample extracted (g).

B. Final results will be reported on a dry-weight basis.

## VII. REFERENCES

None.

## VIII. DATA

See Attachments

## IX. ATTACHMENTS (Note Attachments Not Included)

SAMPLES: _____

_____

SAMPLE FRACTION: SS

EXTRACTION SOLVENT: HPLC H20

TT9 CHECKLIST

1. Check out lot folder ___
2. Weigh 10 g of sample into 40mL amber vial
   Use THAMA SOIL for QC ___
3. Spike the samples, LET STAND FOR 1 HOUR ___
4. Add 20 mL HPLC H20 ___
5. Shake for 4 hours on wrist action shaker ___
6. Centrifuge if needed ___
7. Transfer 5-6mL to shorty vial ___
8. Place samples in fridge C, check samples off of clipboard ___
9. Complete lot folder and relinquish to analyst ___

| QC REQUIRED | YES | NO |
|---|---|---|
| L-1 | ___ | ___ |
| H-1 | ___ | ___ |
| H-2 | ___ | ___ |
| BLK | ___ | ___ |
| MS | ___ | ___ |
| MSD | ___ | ___ |

HPLC H20 LOT#_____

| | |
|---|---|
| Chain of Custody | |
| | |

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# TITLE: DETERMINATION OF EXPLOSIVES IN SOIL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (METHOD LW12)

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TITLE: DETERMINATION OF EXPLOSIVES IN SOIL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (METHOD LW12)

## I. SUMMARY

### A. ANALYTES

This method is applicable to the Class 1 analysis of the following organic compounds in environmental soil samples.

Analytes

1,3-Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
HMX (octahydro-1,3,5,7-tetranitro-s-tetrazocine)
Nitrobenzene
Nitroglycerin
2-Nitrotoluene
PETN (pentaerythritol tetranitrate)
RDX (hexahydro-1,3,5-trinitro-s-triazine)
Tetryl (N-methyl-N,2,4,6-tetranitrobenzenamine)
1,3,5-Trinitrobenzene
2,4,6-Trinitrotoluene

### B. MATRIX

This method is applicable to all relatively dry environmental soil matrices.

### C. GENERAL METHOD

This method employs extraction of one gram of an environmental soil using 2 mL of acetonitrile. Extraction is accomplished by vortexing followed by sonication of the sample for 18 hours. The resulting extract is filtered and diluted 1 to 8 with water. The target analytes are separated on a high perfromance liquid chromatography (HPLC) column using isocratic elution, and detected using ultraviolet (UV) absorbance at 230 nanometers (nm).

## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The certification testing ranges in micrograms per gram (ug/g) are:

| Analyte | Tested Range (µg/g) |
|---|---|
| 1,3-Dinitrobenzene | 0.496-24.8 |
| 2,4-Dinitrotoluene | 0.424-21.2 |
| 2,6-Dinitrotoluene | 0.524-26.2 |
| HMX | 0.666-33.3 |
| Nitrobenzene | 0.548-11.0 |
| 2-Nitrotoluene | 0.307-61.4 |
| Nitroglycerin | 4.00-200 |
| PETN | 4.00-80.0 |
| RDX | 0.438-21.9 |
| Tetryl | 0.404-20.2 |
| 1,3,5-Trinitrobenzene | 0.488-24.4 |
| 2,4,6-Trinitrotoluene | 0.456-22.8 |

### B. SENSITIVITY

The instrumental responses for each compound, reported in peak area units at the certified reporting limit (CRL) are:

| Analyte | CRL (µg/L) | Area Counts |
|---|---|---|
| 1,3-Dinitrobenzene | 0.496 | 40000 |
| 2,4-Dinitrotoluene | 0.424 | 27500 |
| 2,6-Dinitrotoluene | 0.524 | 20000 |
| HMX | 0.666 | 40000 |
| Nitrobenzene | 2.41 | 50000 |
| Nitroglycerin | 4.00 | 4000 |
| 2-Nitrotoluene | 0.307 | 8000 |
| PETN | 4.00 | 4000 |
| RDX | 0.587 | 35000 |
| Tetryl | 0.731 | 40000 |
| 1,3,5-Trinitrobenzene | 0.488 | 45000 |
| 2,4,6-Trinitrotoluene | 0.456 | 30000 |

## C. REPORTING LIMITS

The CRL and upper certified limit (UCL) for each analyte in environmental soil samples are:

| Analyte | CRL (µg/g) | UCL (µg/g) |
|---|---|---|
| 1,3-Dinitrobenzene | 0.496 | 24.8 |
| 2,4-Dinitrotoluene | 0.424 | 21.2 |
| 2,6-Dinitrotoluene | 0.524 | 26.2 |
| HMX | 0.666 | 33.3 |
| Nitrobenzene | 2.41 | 27.4 |
| Nitroglycerin | 4.00 | 200 |
| 2-Nitrotoluene | 0.307 | 61.4 |
| PETN | 4.00 | 80 |
| RDX | 0.587 | 21.9 |
| Tetryl | 0.731 | 20.2 |
| 1,3,5-Trinitrobenzene | 0.488 | 24.4 |
| 2,4,6-Trinitrotoluene | 0.456 | 22.8 |

## D. INTERFERENCES

Any materials which are extracted from soil with acetonitrile, coelute with the explosives through the HPLC column, and which absorb ultraviolet radiation at 230 nm may cause interferences. Carryover from analysis of a highly contaminated sample can result in apparent contamination of the succeeding samples analyzed. Such contamination is often manifested by the presence of unusually broad chromatographic peaks nested among narrower peaks. This interference is minimized by reanalyzing heavily contaminated samples following dilution, running blanks after heavily contaminated samples until carry over is removed, and/or rinsing the system with a mobile phase containing a high proportion of organic modifier until the contamination is removed.

## E. ANALYSIS RATE

After instrument calibration, one analyst can analyze approximately 8 samples per 8-hour day.

## F. SAFETY INFORMATION

The target compounds in this method are toxic explosives and some are known carcinogens, e.g. 2,4-Dinitrotoluene. The preparation of all standards should be

performed in a laboratory hood. Adequate dermal protection must be used when handling samples and standards.

Most of these compounds are either primary or secondary explosives and should be handled with care to avoid contact with electrostatic shocks or impacts. Tetryl, RDX, and PETN have intermediate sensitivity between initiating explosives and explosives used as bursting charges. However, PETN is used as an initiating agent and is extremely sensitive to initiation (more sensitive than either Tetryl or RDX). Tetryl is toxic when taken internally or by skin contact. RDX, HMX,and TNT are used as bursting charge explosives. Although TNT is less sensitive to friction and impact than many other high explosives, it can be detonated with moderate force when confined between metal surfaces such as on the threads of bolts. TNT will form sensitive materials in the presence of alkalies.

## III. APPARATUS AND CHEMICALS

### A. GLASSWARE/HARDWARE

1. 6 mL vials with screw top cap and teflon septa (Wheaton "shorty" vials).
2. 1.6 mL amber vials with Teflon septa and screw top closures (Shimadzu autosampler vials).
3. Vortex mixer, Scientific Products Vortex-Genie, Model S8223.
4. Ultrasonic bath, Bransson 3200.
5. Filter assembly, Gelman Acrodisc CR 0.45 micrometer (µm).
6. 5 mL syringe, Hamilton 10005TLL.
7. 250 µL syringe, Hamilton 7255NR.
8. 1000 µL syringe, Hamilton 1001LTN.
9. Class A Volumetric flasks - 100, 25 and 50 mL.
10. Class A Volumetric pipets - 0.5, 1.0, 2.0, 5.0, and 10.0 mL.
11. Micro pipets - 50, 100, and 200 µL.
12. Balance, capable of weighing to 0.0001 gram.
13. Rainin P-5000 Pipetman, calibrated at 1.4 mL.

### B. INSTRUMENTATION AND ANALYTICAL CONDITIONS

1. HPLC: Shimadzu model LC-6A high-performance liquid chromatograph with Shimadzu model SIL-6A autosampler (or equivalent).
2. Detector: Shimadzu SPD-6A variable wavelength ultraviolet (UV) absorbance detector (or equivalent), set at 230 nanometers.

3. Column: Ultrasphere octadecyldimethylsilyl (ODS), reverse-phase column, 25 centimeters (cm) length x 4.6 millimeters (mm) I.D., 5 µm particle size (Beckman Instr. Co., San Ramon, CA.).
4. Mobile phase: 40.5 percent methanol, 9.5 percent acetonitrile, and 50 percent water, (V/V).
5. Flow rate: 1.0 milliliters per minute (mL/min).
6. Sample Injection Volume: 250 microliters (uL).

## C. ANALYTES

| Analyte | USAEC Abbrev. | CAS Number |
|---|---|---|
| 1,3-Dinitrobenzene | 13DNB | 99-65-01 |
| 2,4-Dinitrotoluene | 24DNT | 121-14-2 |
| 2,6-Dinitrotoluene | 26DNT | 606-20-2 |
| HMX | HMX | 2691-41-0 |
| Nitrobenzene | NB | 98-95-3 |
| Nitroglycerin | NG | 53-63-0 |
| 2-Nitrotoluene | 2NT | 88-72-2 |
| PETN | PETN | 75-11-5 |
| RDX | RDX | 121-84-4 |
| Tetryl | TETRYL | 479-45-8 |
| 1,3,5-Trinitrobenzene | 135TNB | 25377-32-6 |
| 2,4,6-Trinitrotoluene | 246TNT | 118-96-7 |

## D. REAGENTS AND SARMS

1. The standards used for target compound certification and calibration are U.S. Army Environmental Center (USAEC) supplied standard analytical reference materials (SARMS). Equivalent standards may be used as long as they have been characterized according to Section 6.5.3 of the USATHAMA Quality Assurance Plan (2nd Edition, March, 1987).

   USAEC SARMS were used in this certification, except for the appended analyte 2-Nitrotoluene, and their lot numbers are listed below:

| Analyte | SARM LOT NUMBER |
|---|---|
| 1,3-Dinitrobenzene | 2250 |
| 2,4-Dinitrotoluene | 1147 |
| 2,6-Dinitrotoluene | 1148 |
| HMX | 1217 |
| Nitrobenzene | 2177 |
| Nitroglycerin | 1150 |
| PETN | 1151 |
| RDX | 1130 |
| Tetryl | 1149 |
| 1,3,5-Trinitrobenzene | 1153 |
| 2,4,6-Trinitrotoluene | 1129 |
| 2-Nitrotoluene | No SARM - Aldrich lot #08506MV (99+% purity, Aldrich Chemical Co., Milwaukee, WI) |

2. Methanol (HPLC grade - American Burdick & Jackson, McGaw Park, Illinois).
3. Water (HPLC grade - American Burdick & Jackson, McGaw Park, Illinois).
4. Acetonitrile (HPLC grade - American Burdick & Jackson, McGaw Park, Illinois).

## IV. CALIBRATION

### A. INITIAL CALIBRATION

1. Preparation of Standards

Precertification Calibration

Separate primary stock standards (SPSS) for each target analyte are prepared according to the dilution scheme presented in Table IV-1 (see Appendix 3). The SPSS solutions should be prepared fresh every 12 months. Tetryl needs to be made fresh if degradation is evident, but at least every 12 months.

Each separate stock solution is made to volume with acetonitrile. Two SARMS (nitroglycerin and PETN) are provided by USAEC as solutions containing a total of 200 mg. The entire contents of the vials were used to prepare the respective primary stock standards.

Aliquots of the separate primary stock standards (SPSS) are used to prepare the combined stock standard (CSS) by dilution to a final volume of 50 mL using acetonitrile as described in Table IV-2 (see Appendix 3). The CSS solutions should be prepared fresh every 6 months. All primary stocks and combined stocks should be stored in a freezer.

For precertification calibration, duplicate composite calibration standards (CCS-1 through CCS-7) are prepared from the combined stock standard (CSS) as given in Tables IV-3 and IV-4 (see Appendix 3). HPLC-grade water is used for dilution to final volumes for the composite calibration standards. Standards are prepared fresh when run.

Certification Calibration

Standards CCS-1, CCS-3, CCS-5, CCS-6, CCS-7, and a bland described in Tables IV-3 and IV-4 (see Appendix 3) are prepared. These solutions are prepared fresh for every run.

2. Initial Calibration

Standards dilutions D, D2, D4, C2, B, and a blank described in Tables IV-9 and IV-10 (see Appendix 3) are prepared. These solutions are prepared fresh for every run.

3. Instrument Calibration

To calibrate the instrument, 250 µL of each standard in Tables IV-9 and IV-10 (see Appendix 3) is injected into the instrument in the same manner as a sample extract. Each duplicate composite calibration standard is analyzed during precertification calibration, and the single dilutions of the composite standards are analyzed during initial calibration. Initial calibration is repeated after analysis of seven sample lots, or upon failure of the CCS (see Section IV.B.2).

4. Independent Reference Standard

An independent stock will be prepared to serve as a reference standard for explosives in soil. The independent reference standard must be analyzed along with the initial and precertification calibration standards, and the results must be within $\pm$ 25% of the expected value, for the calibration standards to be considered valid. If the analysis of the independent reference standard fails, the source of the problem must be identified and corrected. The results of the second analysis of the independent reference standard must be within the acceptable limits

before the analysis of samples may proceed. A reference, if available, is required at least with every initial calibration.

5. Analysis of Calibration Data

After analyzing the standards (i.e., one blank and five standards), the data are tabulated and graphed. For precertification calibration, the duplicate calibration data are analyzed using the lack of fit (LOF) and zero intercept (ZI) tests (USATHAMA QA Plan, 2nd Edition, March, 1987).

## B. DAILY CALIBRATION

1. Instrument Calibration

For daily calibration the highest concentration standard, a continuing calibration standard (CCS), will be analyzed as stated in Section VII.D of the USATHAMA QA Plan, January 1990.

2. Analysis of Calibration Data

The response for the target compounds during initial calibration does not have to be less than 25 percent different from the response obtained during the previous initial calibration, because each run is an initial calibration. Since reference solutions are not readily available responses could be monitored to evaluate trends changes in stocks. It is advised that stocks for standards and spike solutions be staggered to ensure that degradation of the solutions is dissimilar.

The response of the target compounds in the end run standard must be less than 25 percent different from response factors obtained from the same standard analyzed during the initial calibration. If the response is greater than 25 percent different, the standard will be reanalyzed. If reanalysis still fails the 25-percent criterion, a new initial calibration must be performed and all analyses since the last acceptable calibration must be repeated. After seven daily calibrations have been completed, (analysis of seven sample lots), the initial calibration must be repeated.

3. Retention Time Windows

Retention windows will be determined on a daily basis using the calibration standards used throughout the analytical run. Daily window determinations will yield a window based on daily instrument performance. Additionally because the calibration curve is being used

variation of the retention time with analyte concentration can be taken into account as well as daily instrument drift. The following procedure will be used to calculate retention time windows:

1. The mean retention time will be calculated as the mean retention time of the daily calibration standards.

2. The standard deviation of the daily retention window will be determined using the retention times of the daily calibration standards and the continuing calibration standards.

3. The daily retention window for an analyte will be the mean retention time plus or minus three times the standard deviation.

All of the retention times for reported analytes in samples and continuing calibration standards will be tested against the calculated windows. If an analyte retention time is outside of the calculated window documentation must be provided in order to justify the qualitative identification of the peak.

## V. CERTIFICATION TESTING

See Attachment 1.

## VI. SAMPLING HANDLING AND STORAGE

### A. SAMPLING PROCEDURE

Samples will be collected using adequate dermal and inhalation protection and must follow Sections 5.6 and 5.7 of the USATHAMA Quality Assurance Plan (March 1987).

### B. CONTAINERS

Amber colored glass jars with Teflon-lined lids are required.

### C. HOLDING TIME LIMITS

Samples must be extracted within 7 days of sampling date, and the extract must be analyzed within 40 days of extraction date. Samples should be stored at 4 °C ± 2 °C until extraction.

### D. SOLUTION VERIFICATION

Verification of the calibration standards is based on the analyses of daily QC spikes and analysis of independent reference standards (if available). Since stable reference solutions are not readily available, staggered preparation of stock solutions for control spikes and standards needs to be implemented to ensure acceptable solution verification. An unextracted control spike solution could be analyzed weekly as a check to avoid extraction and storage affects. The response of this check must be within 25 percent of the true value or $\pm$ 2 standard deviations for recent performance (last 7 runs). If criteria cannot be met for the target compounds new stock solutions must be prepared.

## VII. PROCEDURE

### A. SAMPLE PREPARATION

A portion of the sample is spread out on a sheet of aluminum foil and allowed to air dry overnight under a hood. The dried soil needs to be submitted for moisture determination.

### B. SAMPLE EXTRACTION

One gram of dried soil is weighed into a 6 mL vial and with a pipet 2.0 mL of acetonitrile is added. The sample is vortexed for 30 seconds and then placed in a sonicator for 18 hours. Following settling, a portion of the extract is removed with a dispo pipet and placed into a 5 mL syringe equipped with a 0.45 µm acrodisc filter. The extract is filtered and collected into a 1 mL sample vial. The extract is stored in the dark at in the freezer until analysis. Prior to analysis, 200 µL of the extract is mixed with 1400 µL of water directly in the autosampler vial.

### C. CHEMICAL REACTIONS

This method does not involve any chemical reactions.

### D. INSTRUMENTAL ANALYSIS

Instrumental analysis involves injection of 250 µL of the diluted extract onto the analytical column under the conditions as specified in Sec. III.B. The integrated output of the UV detector is used in the calculations of Section VIII. All responses with each analyte's retention window which correspond to concentrations greater than or equal to the respective CRL must be confirmed by analysis on a second HPLC column with dissimilar bonded phase material

(e.g., CN instead of ODS). The conditions for this second column confirmation are described in Attachment 2.

## E. CONFIRMATION ANALYSIS

Analytes that are tentatively identified on the primary column must be confirmed by analysis on a different column with a different liquid phase. In order to confirm an analyte a response must be present in the retention windows for the analyte on both the primary column and the confirmation column. The retention windows will be calculated the same way for both columns. Decision points to be made for the identification and reporting of a target analyte are:

1. Is there a response in the retention window of a target analyte on the primary column and the response is above the CRL?

   No. No further action is necessary and the analyte is reported as <CRL.

   Yes. Analyze the sample extract on the confirmation column.

2. Is there a response on the confirmation column in the retention window of the target analyte and the response is above the criterion of detection?

   No. The analyte is not confirmed and the analyte is reported as <CRL adjusted for any dilutions required.

   Yes. Determine ability to identify peak.

3. Is the peak well defined?

   Yes. The analyte is confirmed and the response of the target analyte is reported from the primary column analysis.

   No. There is considerable interference on the confirmation column analysis which in the analyst's judgement precludes their ability to identify a peak in the retention window of interest. The analyte is considered as not confirmable. The analyte will be reported with the concentration calculated from the primary column and flagged with a "Q".

Definition: COD = one half of the detection limit

## VIII. CALCULATIONS

A linear regression equation is calculated from calibration data by using the response versus the concentration for each compounds. The concentration of a target compound in the soil extract is calculated by substituting the response into the calibration curve equation. The following formula is then used to calculate the concentration (ug/g) of each compound in the soil.

$$\text{Conccentration } (\mu g/g) = \frac{EC \times EV \times (DV + EAV/EAV)}{SW}$$

Where: EC is the extract concentration from the standard curve in µg/mL.
EV is the extraction volume (2 mL).
DV is the dilution volume (1400 µL).
EAV is the extract aliquot volume (200 µL), and
SW is the soil sample weight in grams (1 g).

## IX. DAILY QUALITY CONTROL

### A. CONTROL SAMPLES

Daily quality control samples consist of a standard matrix method blank (USAEC Standard soil), duplicate spikes at the upper concentration of the certified range, and a single spike at approximately twice the CRL. These quality control samples should be carried throughout the entire method at the same time samples are run. Separate stock solutions for the daily control spike samples are prepared in the same manner as for the calibration stock solutions [see Section IV.A.1 and Tables IV-1 (Attachment 3)]. The spiking combined stock solution made up as described in Table 4-7 and is used as spiking solution A, Table IV-8 (see Atachment 3).

Daily Spiking Scheme

Daily spiking should be performed into USAEC standard soil using spiking solution A, Table IV-7 (see Appendix 3). The spiking scheme presented in Table IV-8 should be used. Control analytes for this method are RDX, NB, TNB, 246-TNT, 24DNT, NG, PETN, and 2NT. Since the vast majority of lots do not have NG and PETN requested, these analytes are only spiked when they are requested as analytes in the lot.

## B. CONTROL CHARTS

Control charts are prepared for all of the control analytes being analyzed for using the percent recovery data from both the duplicate high level spikes and the low level spikes calculated according to the following equation:

$$\%\ Recovery = \frac{Found\ Conc.}{Spiked\ Concentration} \times 100\ percent$$

The found response is corrected for method blank response prior to calculation of the found concentration. Method blank correction is by instrument response.

Preparation of control charts requires the following data:

1. Average percent recovery (X) of the two high concentration spiked QC samples in each lot,
2. Difference (R) between the two high concentration spiked QC samples in each lot,
3. Three-point moving average (X) percent recovery control chart for the low level spike in each lot, and
4. Three-point moving average difference (R) control chart for the high concentration spike.

For values that fall outside the control limits and data points that are deemed as outliers, the data will be evaluated and corrective action will be taken.

## X. REFERENCES

U.S. Army Toxic and Hazardous Materials Agency 1987, USATHAMA QA Program (December 1985, 2nd Ed., March 1987).

## IX. ATTACHMENTS

A. **Appendix 1** - Section V. Certification Testing

B. **Appendix 2** - Second Column Confirmation

C. **Appendix 3** - Tables IV-1 Through IV-10

# ATTACHMENT 1 -
# SECTION V. CERTIFICATION TESTING

## V. CERTIFICATION TESTING AND DATA

Spiked samples for certification testing are prepared in USAEC standard soil (USAEC Standard Soil). A combined stock spiking solution is made up as shown in Section IV, then is used as spiking solution A. A 1 to 5 dilution of the combined stock is made into acetonitrile to obtain spiking solution B as shown in Table IV.5(see Attachment 3). Spiking is performed for certification testing on four separate days. The spiking scheme is given in Table IV.6 (see Attachment 3). Analysis of the spiked samples follows the procedure outlined in Section VII.

The target versus found data are analyzed using the lack-of-fit (LOF) and zero intercept (ZI) tests (USATHAMA QA Plan, March, 1987).

ATTACHMENT 3 -
Tables IV -1 Through IV -10

Table IV-1. Preparation of Separate Primary Stock Standards (SPSS).

| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | PETN | RDX | TETRYL | 135TNB | 246TNT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| mg SARM added | 12.4 | 10.6 | 13.1 | 41.6 | 13.7 | 200 | 200 | 1109.5 | 10.1 | 12.2 | 11.4 |
| Final Volume (mL) | 10.0 | 10.0 | 10.0 | 25.0 | 10.0 | 50.0 | 50.0 | 100.0 | 10.0 | 10.0 | 10.0 |
| Conc.of SPSS, µg/mL | 1240 | 1060 | 1310 | 1664 | 1370 | 4000 | 4000 | 1095 | 1010 | 1220 | 1140 |

Note: Each SARM diluted to the volume indicated with acetonitrile. All values listed are nominal concentrations and volumes. Actual values will vary slightly each time they are prepared.

Source: ESE.

Table IV-2. Preparation of Composite Stock Standard (CSS-A and CSS-B)

| | CSS-A | | | | | | | | | | CCS-B |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | PETN | RDX | 135TNB | 246TNT | TETRYL |
| mL SPSS added | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 2.5 | 2.5 | 1.0 | 1.0 | 1.0 | 1.0 |
| Final Volume (mL) | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 |
| Conc. of CSS (ug/mL) | 24.8 | 21.2 | 26.2 | 33.28 | 27.4 | 200 | 200 | 21.9 | 24.4 | 22.9 | 20.2 |

Note: Acetonitrile used for dilution to the 50 mL final volume for the Composite Stock Standard (CSS). Tetryl intermediate is prepared separately due to observation that degradation occurs more rapidly when combined with other analytes at higher concentrations. All values are nominal concentrations and volumes. Actual values will vary slightly each time they are prepared.

Source: ESE.

Table IV-3. Preparation of Precertification and Certification Composite Calibration Standards 1 Through 4 (CCS-1 through CCS-4).

| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | PETN | RDX | TETRYL | 135TNB | 246TNT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Conc. of CSS-1 (ug/mL) | 2.480 | 2.120 | 2.620 | 3.330 | 2.740 | 20.0 | 20.0 | 2.19 | 2.02 | 2.44 | 2.28 |
| Conc. of CSS-2 (ug/mL) | 0.992 | 0.848 | 1.048 | 1.330 | 1.096 | 8.00 | 8.00 | 0.876 | 0.808 | 0.976 | 0.912 |
| Conc. of CSS-3 (ug/mL) | 0.496 | 0.424 | 0.524 | 0.666 | 0.548 | 4.00 | 4.00 | 0.438 | 0.404 | 0.488 | 0.456 |
| Conc. of CSS-4 (ug/mL) | 0.148 | 0.212 | 0.262 | 0.333 | 0.274 | 2.00 | 2.00 | 0.219 | 0.202 | 0.244 | 0.228 |

Note: CCS-1 prepared by diluting 5.0 mL of CSS to 50 mL with HPLC-grade water.
CCS-2 prepared by diluting 2.0 mL of CSS to 50 mL with HPLC-grade water.
CCS-3 prepared by diluting 1.0 mL of CSS to 50 mL with HPLC-grade water.
CCS-4 prepared by diluting 0.5 mL of CSS to 50 mL with HPLC-grade water.

Source: ESE.

Table IV-4. Preparation of Precertification and Certification Composite Calibration Standards 1 Through 4 (CCS-5 through CCS-7).

| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | PETN | RDX | TETRYL | 135TNB | 246TNT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Conc. of CSS-5 (ug/mL) | 0.0992 | 0.0848 | 0.1048 | 0.133 | 0.1.96 | 0.800 | 0.800 | 0.0876 | 0.0876 | 0.0976 | 0.0912 |
| Conc. of CSS-6 (ug/mL) | 0.992 | 0.848 | 1.048 | 1.330 | 1.096 | 8.00 | 8.00 | 0.876 | 0.808 | 0.976 | 0.912 |
| Conc. of CSS-7 (ug/mL) | 0.496 | 0.424 | 0.524 | 0.666 | 0.548 | 4.00 | 4.00 | 0.438 | 0.404 | 0.488 | 0.456 |

Note: CCS-5 prepared by diluting 0.2 mL of CSS to 50 mL with HPLC-grade water.
CCS-6 prepared by diluting 0.1 mL of CSS to 50 mL with HPLC-grade water.
CCS-7 prepared by diluting 0.05 mL of CSS to 50 mL with HPLC-grade water.

Source: ESE.

Table IV-5. Spiking Solutions for Soil Validation Concentrations (ug/mL).

| Spiking Solution | HMX | RDX | TNB | DNB | TETRYL | NB | NG | TNT | 26DNT | 24DNT | PETN |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A | 33.3 | 21.9 | 24.4 | 24.8 | 20.2 | 27.4 | 200 | 22.8 | 26.2 | 21.2 | 200 |
| B | 6.66 | 4.38 | 4.88 | 4.96 | 4.04 | 5.48 | 40 | 4.56 | 5.24 | 4.24 | 40 |

Table IV-6. Spiking Scheme for Soil Validation (ug/g).

| Level | Spiking Volume | Spiking Solution | Weight Soil | HMX | RDX | TNB | DNB | TETRYL | NB | NG | TNT | 26DNT | 24DNT | PETN |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1/2 X | 0.05 mL | B | 1 g | 0.333 | 0.21 | 0.244 | 0.24 | 0.202 | 0.274 | 2.00 | 0.228 | 0.262 | 0.212 | 2.00 |
| 1 X | 0.10 mL | B | 1 g | 0.666 | 0.43 | 0.488 | 0.49 | 0.404 | 0.548 | 4.00 | 0.456 | 0.524 | 0.424 | 4.00 |
| 2 X | 0.20 mL | B | 1 g | 1.33 | 0.87 | 0.976 | 0.99 | 0.808 | 1.10 | 8.00 | 0.912 | 1.05 | 0.848 | 8.00 |
| 5 X | 0.50 mL | B | 1 g | 3033 | 2.19 | 2.44 | 2.48 | 2.02 | 2.74 | 20.0 | 2.28 | 2.62 | 2.12 | 20.0 |
| 10 X | 0.20 mL | A | 1 g | 6.66 | 4.38 | 4.88 | 4.96 | 4.04 | 5.48 | 40.0 | 4.56 | 5.24 | 4.24 | 40.0 |
| 20 X | 0.40 mL | A | 1 g | 13.3 | 8.76 | 9.76 | 9.92 | 8.08 | 11.0 | 80.0 | 9.12 | 10.5 | 8.48 | 80.8 |
| 50 X | 1.0 mL | A | 1 g | 33.3 | 21.9 | 24.4 | 24.8 | 20.2 | 27.4 | 200 | 22.8 | 26.2 | 21.2 | 200 |

Table IV-7. Preparation of Combined Spike Composite Stock Standard (SCSS).

| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | PETN | RDX | TETRYL | 135TNB | 246TNT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| mL SPSS added to | 1.0 | 1.0 | 1.0 | 1.0 | 2.0 | 2.5 | 2.5 | 1.0 | 1.0 | 1.0 | 1.0 |
| Final Volume (mL) | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 |
| Conc. of SCSS (ug/mL) | 24.8 | 21.2 | 26.2 | 33.28 | 27.4 | 200 | 200 | 21.9 | 20.2 | 24.4 | 22.9 |

Note: Acetonitrile used for dilution to the 50 mL final volume for the Spike Composite Stock Standard (SCSS).

Source: ESE.

Table IV-8. Daily Spiking Scheme

| mL of Spiking Solution A uses | gm of USAEC Std. Soil | HMX | RDX | TNB | DNB | TETRYL | NB | NG | TNT | 26DNT | 24DNT | PETN |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Blank 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Low Level | 1 | 1.64 | 1.10 | 1.22 | 1.24 | 1.01 | 2.74 | 10.0 | 1.14 | 1.31 | 1.06 | 10.0 |
| High Level | 1 | 13.3 | 8.76 | 9.76 | 9.92 | 8.08 | 22.0 | 80.0 | 9.12 | 10.5 | 8.48 | 80.0 |

Table IV-9. Preparation of 25 mL Composite Calibration Stock Standards for Daily/Initial Calibration

| Analyte | Conc. of SPSS, ug/mL | Volume of SPSS added, mL | Concentration of CCS-A, ug/mL |
|---|---|---|---|
| 1,3 DNB | 1032 | 0.55 | 22.7 |
| 24DNT | 2144 | 0.25 | 21.4 |
| 26DNT | 1140 | 0.55 | 25.1 |
| HMX | 1056 | 0.6 | 25.3 |
| NB | 3200 | 0.25 | 32.0 |
| NG | 4000 | 1.25 | 32.0 |
| 2NT | 1892 | 0.25 | 18.9 |
| PETN | 4000 | 1.25 | 200 |
| RDX | 1350 | 0.45 | 24.3 |
| 1,3,5 TNB | 584 | 0.95 | 22.2 |
| 2,4,6 TNT | 1740 | 0.3 | 20.9 |
| | | | Concentration of CCS-B, ug/mL |
| TETRYL | 2050 | 0.25 | 20.5 |

The above standards are given unique alphanumeric designates according to procedure of Appendix B. Dilutions are made as follows:

- B Level Prepared by diluting 1.0 mL each of CCS-A and CCS-B to 10 mL with 1.0 mL acetonitrile and remainder HPLC-grade water
- C2 Level Prepared by diluting 0.2 mL each of CCS-A and CCS-B to 10 mL with 30/70% acetonitrile/HPLC-grade water
- D4 Level Prepared by diluting 0.2 mL each of CCS-A and CCS-B to 50 mL with 30/70% acetonitrile/HPLC-grade water
- D2 Level Prepared by diluting 0.1 mL each of CCS-A and CCS-B to 50 mL with 30/70% acetonitrile/HPLC-grade water
- D Level Prepared by diluting 0.1 mL each of CCS-A and CCS-B to 100 mL with 30/70% acetonitrile/HPLC-grade water

Table IV-10. Concentration of Daily Initial Calibration Levels in ug/mL

| Analyte | 13DNB | 24DNT | 26DNT | HMX | NB | NG | 2NT | PETN | RDX | TETRYL | 135TNB | 246TNT |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| D | 0.0227 | 0.0214 | 0.0251 | 0.0253 | 0.0320 | 0.200 | 0.0189 | 0.200 | 0.0243 | 0.0205 | 0.0222 | 0.0209 |
| D2 | 0.0454 | 0.0429 | 0.0502 | 0.0507 | 0.0640 | 0.400 | 0.0378 | 0.400 | 0.0486 | 0.0410 | 0.0444 | 0.0418 |
| D4 | 0.0908 | 0.0858 | 0.100 | 0.010 | 0.128 | 0.800 | 0.0757 | 0.800 | 0.0972 | 0.0820 | 0.0888 | 0.0835 |
| C2 | 0.454 | 0.429 | 0.502 | 0.507 | 0.640 | 4.0 | 0.378 | 4.0 | 0.486 | 0.410 | 0.444 | 0.418 |
| B | 2.27 | 2.14 | 2.51 | 2.53 | 3.20 | 20.0 | 1.89 | 20.0 | 2.43 | 2.05 | 2.22 | 2.09 |

Note: All values listed are for nominal concentrations given in previous table. Actual values will vary slightly as new SPSS are prepared.

ATTACHMENT 2 -
SECOND COLUMN CONFIRMATION

## CONFIRMATION FOR LW12 METHOD BASED UPON CLASS 2 CERTIFIED METHOD FOR UW32

All analytes in LW12 are included in the list of UW32 analytes with the exception of Nitroglycerine and PETN. Therefore, the confirmation method for UW32 will be used to determine the presence or absence of all LW12 compounds except Nitroglycerin and PETN. The extracts are diluted just as the LW12 method describes and ran using the conditions listed for the confirmation. The responses for these samples are only compared to the reference standard in the confirmation and may not be related to the LW12 primary analysis due to differences in injection volume, wavelength, etc. The presence of a particular analyte will be confirmed in the response on the confirmaTIon column is at least 80% of the response in the reference standard. Allowing responses 20% below the reference standard to be confirmed is due to the differences in the CRL of the two methods. For example, a sample with a hit just above the CRL for RDX in the LW12 analysis may have a response slightly below the reference standard in the confirmation analysis, but the presence will be confirmed.

## CONFIRMATION FOR METHOD UW32

This method is a Class 2 certified method which screens for the presence or absence of contaminants, which in this case are the UW32 analytes listed below. All analytes as well as the surrogate 3,4-DNT are separated using the confirmation column, with the exception of the Nitrotoluenes. All three of them form one single peak and can be confirmed as a group only. Samples extracted using the USAEC method UW32 are measured in relation to the Certified Reporting Limit (CRL) set to a desired level and reported as "greater than" (confirmed), or "less than" (unconfirmed) the CRL. A tested concentration range is not applicable since only the CRL concentration is tested. The sample extracts, which are in a 33% acetonitrile/67% water matrix, are analyzed according to the conditions below. Class 2 certification as outlined by USAEC was performed on August 15, 1991 and subjected to the Rank Sum Test. The certification passed the criterion for acceptability. The chosen level for measurement Was the lowest calibration standard for the UW32 method, which is approximately 20% below the CRL. Instrumentation used is the same as described in Section III.B.1 and 2.

The chromatographic conditions are as follows:

| | |
|---|---|
| Column | Zorbax Cyano, 250 X 4.6 mm, 5 $\mu$m packing |
| Mobile Phase | 50% HPLC grade Methanol/50% HPLC Water |
| Flow Rate | 1 mL/min |
| Injection Volume | 500 $\mu$L |
| Detection Wavelength | 250 nm |
| Temperature | Ambient |

The elution order and the retention times during certification are as follows. Slight shifts in the retention times may be expected from run to run.

| | |
|---|---|
| Nitrobenzene | 8.27 min |
| 1,3-DNB | 9.35 min |
| 1,3,5-TNB | 9.82 min |
| Nitrotoluenes | 10.48 min |
| 2,6-DNT | 11.42 min |
| 2,4-DNT | 12.02 min |
| 2,4,6-TNT | 13.10 min |
| 4-Amino-2,6-DNT | 13.55 min |
| 2-Amino-4,6-DNT | 14.65 min |
| 3,4-DNT | 15.12 min |
| RDX | 17.17 min |
| Tetryl | 25.50 min |
| HMX | 32.38 min |

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# DETERMINATION OF THIODIGLYCOL AND CHLOROACETIC ACID IN SOIL BY GAS CHROMATOGRAPHY

January, 1989

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Prepared for:

U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
Aberdeen Proving Ground, MD. 21010-5401

**USATHAMA METHOD NUMBER: _____**
**ANALYSIS OF THIODIGLYCOL AND CHLOROACETIC ACID IN ENVIRONMENTAL SOIL SAMPLES**

## I. SUMMARY

### A. ANALYTES

This method is applicable to the quantitative determination of thiodiglycol and chloroacetic acid in environmental soil samples.

### B. MATRIX

This method is applicable to all environmental soil matrices.

### C. GENERAL METHOD

A measured weight of sample is extracted with alkaline methanol on a wrist-action shaker. A portion of the methanol is filtered, and removed by evaporation under a nitrogen stream. The extract is acidified, buffered, and brought to volume with water. Chromatographic conditions described in this method permit the separation and measurement of the two analytes in the methanol extract. Analyte identification is performed using retention times, and quantitative analysis is performed using a standard curve of area counts.

## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The tested concentration ranges in "standard soil" samples are:

| Analyte | Tested Concentration Range (ug/g)* |
|---|---|
| Thiodiglycol | 2.55 to 102.0 |
| Chloroacetic acid | 7.54 to 302.0 |

*ug/g = micrograms per gram.

B. SENSITIVITY

The normalized response (integrator counts corrected for attenuation) at the "standard soil" reporting limits are:

| Analyte | Area Counts |
|---|---|
| Thiodiglycol | 630,000 |
| Chloroacetic acid | 680,000 |

C. REPORTING LIMIT

The certified reporting limits in standard soil as calculated according to the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) certified reporting limit program are:

| Analyte | Reporting Limit (ug/g) | Upper Certified Range (ug/g) |
|---|---|---|
| Thiodiglycol | 3.94 | 102.0 |
| Chloroacetic acid | 18.0 | 302.0 |

D. INTERFERENCES

1. Interferences in methods employing ultraviolet (UV) detection at short wavelengths can pose problems in analysis of this method. The interference can usually be minimized by preventing contact of reagents, glassware, apparatus, and samples with any plastic materials.
2. Solvents, reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be demonstrated to be free from interferences that have retention times equal to those of the compounds of interest.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze eight samples in an 8-hour day. One analyst can perform

approximately eight extractions in an 8-hour day.

### F. SAFETY INFORMATION

This method involves the use of methanol and acid. Adequate dermal and eye protection should be used, and proper ventilation available. Chloroacetic acid is highly toxic and z strong irritant and should be handled with care.

## III. APPARATUS AND CHEMICALS

### A. GLASSWARE/HARDWARE

1. Eight 50 milliliter (mL) centrifuge tubes, screw top;
2. Eight 25 mL pipettes;
3. Eight 10 mL Gastight syringes;
4. Gelman Acrodisc CR filter assemblies, 0.45 micrometer (um);
5. Eight 10 mL pipettes;
6. Eight scintillation vials;
7. Nitrogen manifold (8 ports);
8. 0.5 mL pipettes; and
9. 15 mL graduated centrifuge tubes.

### B. INSTRUMENTATION AND OPERATING PARAMETERS

1. Altex Model 322 gradient high-pressure liquid chromatograph (HPLC) equipped with a Perkins Elmer LC-75 variable wavelength UV visible detector and interfaced to a Shimadzu C-R3A computing integrator.
2. IEC Model 5000CU centrifuge.
3. Burrell Model 75 wrist-action shaker.
4. Detector: Perkin Elmer LC-75 variable wavelength detector [ = 215 nanometers (nm)].
5. Column: Ultrasphere octadecylsilane (ODS) [4.6-millimeter (mm) inside diameter (ID) by 25 centimeters (cm)].
6. Particle Size: 5 um.

7. Guard column: 2.6 mm ID by 10 cm packed with Whatman Co: Pell ODS.
8. Silica precolumn: 2.6 mm ID by 10 cm packed with 60-230 mesh silica gel (ICN Pharmaceuticals, Inc.).
9. Flow rate and mobile phase: 1 milliliter per minute (mL/min) of 0.06 molar (M) phosphate buffer [7.94 grams (g) of sodium dihydrogen phosphate hydrate ($NaH_2PO_4 \cdot H_2O$) and 0.16 mL of 85-percent phosphoric acid ($H_3PO_4$) dissolved in 1 liter (L) of water].
10. Temperature: Room temperature.
11. Injection volume: 250 microliters (uL) fixed loop.
12. Retention times: Thiodiglycol -- 11.9 minutes (min) $\pm$ 0.4 min: chloroacetic acid -- 4.5 min $\pm$ 0.4 min. Retention time windows represent 3 standard deviations of a control spike measured during the 4 days of certification.

## C. ANALYTES

| Analyte | CHEMICAL ABSTRACT Service (CAS) Registry Number |
|---|---|
| Thiodiglycol | 111-48-8 |
| Chloroacetic acid | 79-11-8 |

## D. REAGENTS

1. HPLC-grade methanol (Burdick and Jackson);
2. 4 normal (N) sodium hydroxide -- 160 g of sodium hydroxide dissolved in 1 L of HPLC water;
3. HPLC water (Burdick and Jackson);
4. Nitrogen gas;
5. Basic methanol solution -- mix 40 mL of 4 N sodium hydroxide and 1,000 mL of methanol;
6. Acid solution -- 1:4 (volume:volume) mixture of concentrated $H_2SO_4$ and HPLC water;
7. Concentrated buffer solution -- 79.4 g of $NaH_2PO_4 \cdot H_2O$ and 1.6 mL of 85-percent $H_3PO_4$ in one liter of HPLC water;

8. Chloroacetic acid (Aldrich Gold Label), 99+ percent purity.
9. 2,2'-Thiodiethanol (Thiodiglycol) (Aldrich), 99+ percent purity.

## IV. CALIBRATION

### A. INITIAL CALIBRATION

1. Preparation of Standards

Combined primary stock calibration standards are prepared by weighing approximately 0.5 grams (g) of thiodiglycol and 3.5 g of chloroacetic acid into a 100-mL volumetric flask; then dilute to volume with HPLC water. The concentrations of each compound in the primary stock calibration standard prepared this way is 5,000 ug/mL and 35,000 ug/mL, respectively.

A composite secondary stock calibration standard is prepared by adding 10 mL of the primary stock calibration standard to volume with HPLC water in a 100-mL volumetric flask. The concentration in the composite secondary stock calibration standard for each analyte is 500 and 3,500 ug/mL, respectively.

Composite working calibration standards are prepared using HPLC water, the composite secondary stock calibration standard and volumetric pipettes as shown in Table IV-1.

2. Instrument Calibration

To calibrate the instrument, at least 250 uL of each standard in Table IV-1 is injected into the instrument in the same manner as a sample extract. Duplicate composite calibration standards are analyzed during precertification calibration, and the single dilutions of the composite standards are analyzed during initial calibration. Currently an independent reference standard is not available for thiodiglycol and chloroacetic acid. Meanwhile an independent stock will be prepared to serve as

Table IV-1. Preparation of Initial Instrument Calibration Standards

| Calibration Standard | Volume Composite 2° Stock Standard Used | Final Volume (mL) | Concentration of Each Analyte in Instrument Calibration Standard (ug/L) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Chloroacetic Acid |
| A | 1.0 | 10 | 50 | 350 |
| B | 1.0 | 25 | 20 | 140 |
| C | 0.5 | 25 | 10 | 70 |
| D | 0.2 | 25 | 4.0 | 28 |
| E | 0.1 | 25 | 2.0 | 14 |
| F | 0.05 | 25 | 1.0 | 7.0 |
| G | 0.025 | 25 | 0.50 | 3.5 |
| H | 0 | 25 | 0 | 0 |

Source: Hunter/ESE, 1989.

a reference standard. The reference must be analyzed along with the initial and precertification calibration standards, and the results must be within ± 25% of the true value for the calibration to be considered valid. If the analysis of the reference standard fails, the source of the problem must be identified and corrected. The initial calibration and analysis of the independent reference standard must be repeated. The result of the second analysis of the independent reference standard must be within the acceptable limits, as specified by the source of the standard, before the analysis of samples may proceed. Since initial calibrations are performed daily, a reference is required at least weekly.

3. Analysis of Calibration Data

After analyzing the standards (i.e., one blank and nine standards) in duplicate, the data are tabulated and graphed. Data are analyzed using the lack of fit (LOF) and zero intercept (ZI) tests (USATHAMA QA Plan, 2nd Edition, March 1987). All pre-certification calibration data passed the LOF-ZI tests, therefore calibrations are linear.

4. Calibration Checks

At the end of the daily instrumental analysis, the highest working calibration standard is injected into the instrument. The response or the recovery of this end-of-day analysis should be ± 25% of the response or recovery obtained from the analysis of the same working calibration standard curve analyzied that day. If the first calibration check does not pass this criterion, then the standard may be reinjected and compared to the initial response. The post run standard data is documented and the percent drift is calculated on the chromatographic logsheet. An explanation must be provided if the calibration check is not acceptable for the data to be reported, otherwise the samples are rerun.

**B. DAILY CALIBRATION**

For daily calibration an initial calibration curve and QC checks will be performed as stated in Section IV.A.

## V. CERTIFICATION TESTING

**A. PREPARATION OF CONTROL SPIKES FOR METHOD CERTIFICATION**

1. A combined primary certification spiking solution was prepared by weighing 0.509 g of thiodiglycol and 1.510 g of chloroacetic acid into a 100-mL volumetric flask and diluting to volume with HPLC water. The concentration of the primary certification spiking solution was 5,090 ug/mL and 15,100 ug/mL, respectively.
2. A secondary certification spiking solution was prepared by pipetting 10.0 mL of the primary certification spiking solution to a 100-mL volumetric flask and diluting to volume with HPLC water. The concentration of the secondary certification spiking solution was 509 and 1,510 ug/mL, respectively.
3. Certification spikes were prepared as shown in Table V-1 and were used to determine the accuracy, range, and reporting limits of the analytes to which this method applies. In each case, 10 g of standard soil was spiked with the appropriate certification spiking solution. The standard soil used for certification in this method was an uncontaminated background soil from the Rocky Mountain Arsenal (RMA) area in Denver, CO., rather than the USATHAMA standard soil. (see Attachment 3 for USATHAMA soil spike data.)

Table V-1. Preparation of Certification Control Spikes

| Certification Control Spike Sample Prepared | Control Stock Used | Volume Spiked* (mL) | Concentration of Each Analyte in the Prepared Sample (ug/g) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Chloroacetic Acid |
| Blk | ---- | 0 | 0 | 0 |
| 1S | 2° | 0.05 | 2.55 | 7.54 |
| 2S | 2° | 0.10 | 5.09 | 15.1 |
| 3S | 2° | 0.20 | 10.2 | 30.2 |
| 4S | 2° | 0.50 | 25.5 | 75.4 |
| 5S | 1° | 0.10 | 50.9 | 151 |
| 6S | 1° | 0.20 | 102 | 302 |
| 7S | 1° | 0.50 | 255 | 754 |

* to 10 g of standard soil

Source: Hunter/ESE 1989

B. **ANALYSIS OF CERTIFICATION SPIKES**

Certification Control Spikes are analyzed by the procedures outlined in Section VII. Instrumentation is calibrated as in Sec. IV.

## VI. SAMPLE HANDLING AND STORAGE

A. **SAMPLING PROCEDURE**

There are no special considerations required due to the nature of these compounds. Soil samples may be collected as grab samples or cores. The samples need to be chilled to 4 deg. C immediately following sampling.

B. **CONTAINERS**

Sampling containers used are 1.2 Litre glass amber jars with a teflon-lined cap for grab samples, or polybutyrate tubes for core samples.

C. **STORAGE CONDITIONS**

Samples are to be kept on ice in coolers until shipped to the analytical lab. Upon arrival, the samples are stored at 4 deg. C in a walk-in refrigerator.

D. **HOLDING TIME LIMITS**

The holding time limit is seven days from the time of sampling to extraction of the sample and 40 days from extraction to analysis.

E. **SOLUTION VERIFICATION**

Calibration standards are verified with daily control spikes and analysis of reference samples. When fresh control spike stock solutions are prepared. They must be verified to determine that:

1. the previous spiking solution had or had not deteriorated

2. the new solution was correctly prepared

Therefore, the combined primary spike stock solutions (IX.A.1) will be checked against working standards before initial use and again within seven days before subsequent use.

## VII. PROCEDURE

### A. EXTRACTIONS

1. Extraction of Certification Control Spikes Only

   The certification control spikes are extracted beginning with Sec. VII.2.d.

2. Extraction of Environmental Samples

   a. Place 10 g of soil into a 50-mL centrifuge tube.

   b. Add 25.0 mL of basic methanol (Sec. III.D.5.) solution to each centrifuge tube.

   c. Shake the samples on a wrist-action shaker for 20 min.

   d. Centrifuge the samples at approximately 2,000 revolutions per minute (rpm) for 5 min.

   e. Equip a 10-mL Gastight syringe with a 0.45-um Acrodisc CR filter assembly. Pipette 10.0 mL of the extract into the syringe, pass it through the filter, and collect the filtrate in a 15 mL graduated centrifuge tube.

   f. Reduce the extract volume to exactly 1 mL using a nitrogen stream without heating. Note: Final volume critical. Extra methanol can cause chromatographic problems and going below i mL causes loss of chloroacetic acid by volitization.

   g. Add approximately 4 mL of HPLC water and mix in the centrifuge tube. Acidify the extract to pH 2 with the 1:4 acid solution (approximately 8 drops).

   h. Add 0.5 mL of the concentrated buffer solution to the extract. Bring to 10-mL final volume with HPLC water and mix thoroughly. In some cases (i.e. USATHAMA

Standard soil) a second filtration is needed.

i. Transfer some of the extract to a 5-mL, amber-glass, septum-sealed vial for storage at 4 degrees Celsius ( C). The solution is now ready for analysis by HPLC.

B. CHEMICAL REACTIONS

There is no chemical reation stage.

C. INSTRUMENT ANALYSIS

Daily calibration performed as described in Sec. IV.A. and B. with instrument condition described in Sec. III.B. For daily control spikes of USATHAMA Standard soil a 12 min. delay is required after each run because of a late eluting broad peak. Caution should be used with environmental samples in case a broad eluting peak is present.

## VIII. CALCULATIONS

Determine the concentration of each analyte according to the following formula:

$$\text{Concentration (ug/mL)} = \frac{(A)(Vt)}{(Vs)(1 - M)}$$

where: A = Concentration (ug/mL) of each analyte found in the extract by comparison with the appropriate standard curve (ug/mL),

Vt = Final volume of extract solution (25 mL), and

Vs = Weight of initial sample extracted (10 g).

M = Moisture expressed as a fraction

Wet weight and moisture results are reported to the USATHAMA data management system for dry weight calculations.

## IX. DAILY QUALITY CONTROL

### A. CONTROL SAMPLES

1. Preparation of the Combined Primary Spike Stock Solution (CPSSS): The CPSSS is prepared by weighing .75 g of thiodiglycol and 3.5 g of chloroacetic acid into a 100 mL volumetric flask and diluting to volume with HPLC water. The concentrations of the CPSSS are 7,500 and 35,000 ug/mL respectively. (prepare fresh monthly)
2. Preparation of the Secondary Spike Stock Solution (SSSS): An intermediate spike solution is made by diluting 10 mL of the CPSSS (1) with HPLC water into a 100 mL volumetric flask. The concentrations of this solution are 750 and 3,500 ug/mL. (prepare fresh daily)
3. Using the SSSS, the Daily Control Spikes are made by spiking the appropriate volume of stock solutions to 10 g of standard soil as shown in Table IX - 1.

### B. CONTROL CHARTS

In the initial construction of the control charts, data from the laboratory certification analysis will be used. Data from spiked QC samples, within a lot, will be compared to control chart limits to demonstrate that analysis of the lot are under control and will be used to update the charts. X-R control charts will be used in the Quality Assurance (QA) Program. Control charts are prepared for each target analyte using percent recovery (found concentration - method blank/ spiked concentration x 100) data from the duplicate spiked QC samples variations in spiking solution concentrations.

To prepare control charts, the analyst should have access to the following data:

1. Average (x) percent recovery for the two high concentration spiked QC samples in each lot,

Table IX-1. Preparation of Daily Control Spikes

| Daily Control Spike | Control Stock Used | Volume Spiked* (mL) | Concentration of Each Analyte in the Prepared Sample (ug/g) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Chloroacetic Acid |
| Blank | None | 0 | 0 | 0 |
| Low | SSSS | 0.1 | 5 | 35 |
| High | CPSSS | 0.05 | 25 | 175 |
| High | CPSSS | 0.05 | 25 | 175 |

* to 10g of standard soil

Source: Hunter/ESE, 1989.

2. Difference (R) between the percent recoveries for the two high concentration spiked QC samples in each lot,
3. Three-point moving average (x) spkie recovery of the low-concentration spike QC sample, and
4. Three-point moving difference (R) between the percent recoveries for the low-concentration spike QC sample.

The initial control chart shall be prepared using the four pairs of certification data closest to the spiking concentration used during analysis. The average (x), average range (R), and control limits for x and R shall be updates after each lot for the first 20 lots. Limits established after Lot 20 will be used for the next 20 lots. Control charts will be updated after each 20 lots thereafter using the most recent 40 lots of QC data. In updating the control charts, the new data must be combined with the individual values of previous percent recoveries and not the mean of all previous data. Initial control chart limits are presented in Table IX-2.

Table IX-2. Initial Control Limits for Thiodiglycol in Soil

METHOD:
COMPOUNDS: Thiodiglycol and Chloroacetic acid in Soil
UNITS: UGG

3 PT. MOVING AVERAGE $\bar{X}$ - R

| CODE | CONC. | UCL $\bar{X}$ | UWL $\bar{X}$ | = $\bar{X}$ | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| TDGCL | 4.46 | 127.4 | 119.9 | 104.7 | 89.6 | 82.1 | 57.0 | 45.4 | 22.1 |
| CLC2A | 35.0 | 111.6 | 102.7 | 84.7 | 66.8 | 57.8 | 67.7 | 53.9 | 26.3 |

SINGLE DAY $\bar{X}$ - R

| CODE | CONC. | UCL $\bar{X}$ | UWL $\bar{X}$ | = $\bar{X}$ | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| TDGCL | 22.3 | 109.3 | 105.1 | 96.6 | 88.0 | 83.8 | 22.2 | 17.1 | 6.8 |
| CLC2A | 175 | 185.0 | 158.0 | 103.9 | 49.9 | 22.9 | 140.8 | 108.2 | 43.1 |

## X. REFERENCES

U.S. Army Toxic and Hazardous Materials Agency, (USATHAMA). 1987. QA Plan (December 1985, 2nd Edition, March 1987).

## XI. DATA

### A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION

These compounds cannot be seen on GC/MS and GC/FID. Therefore, standards can only be verified by independently prepared references.

### B. PRE-CERTIFICATION CALIBRATION

Attachment 1

### C. DAILY CALIBRATION AND CHROMATOGRAM

Attachment 2

### D. CERTIFICATION DATA (RMA Standard Soil)

Attachment 3

### E. ONE DAY CERTIFICATION WITH USATHAMA STANDARD SOIL

Attachment 4

# USATHAMA METHOD UT02
# ISOPROPYLMETHYL PHOSPHONIC ACID, METHYL PHOSPHONIC ACID, AND FLUOROACETIC ACID IN WATER

## I. SUMMARY

### A. ANALYTES

This method is applicable for the quantitative analysis of isopropylmethyl phosphonic acid (IMPA), methyl phosphonic acid (MPA), and fluoroacetic acid (FC2A) in water by ion chromatography. Note: IMPA and ethyl methyl phosphonic acid (EMPA) coelute. This method was certified using IMPA only, however, reported data could include total IMPA and EMPA.

### B. MATRIX

This method is applicable to most ground and surface waters. Industrial waste waters with total conductivities over 800 microSiemens per centimeter (uS/cm) need dilution to prevent column over-loading, thereby raising sample detection limits.

### C. GENERAL METHOD

Samples are analyzed by gradient ion chromatography and the results are calculated against a calibration curve of the three analytes prepared in deionized water. Samples containing particulate matter must be filtered before analyzing.

## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The tested concentration range for IMPA, FC2A, and MPA is 100 ug/L to 9000 ug/L.

### B. SENSITIVITY

The approximate peak areas in microvolts-minutes (uV-min) [with the conductivity detector on 30 microSiemens per volt (uS/V)

scale] for a 100 ug/L standard are 17,000 for IMPA, 28,000 for FC2A, and 19,000 for MPA.

C. **CERTIFIED REPORTING LIMITS**

The certified reporting limits in water, calculated using the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) reporting limit program, are 100 ug/L for IMPA, 128 ug/L for MPA, and 100 ug/L for FC2A. The upper certified range for each is 9000 ug/L.

D. **INTERFERENCES**

A compound that coelutes with an analyte is an interference. Formic acid coelutes with fluoroacetic acid; ethylmethyl phosphonic acid coelutes with IMPA. Carbonate elutes just after MPA and high concentrations can interfere with accurate determination of MPA. Samples with high concentrations of anions can cause column over-loading, requiring the sample to be diluted. High concentrations of transition metals can cause low recovery for both IMPA and MPA.

E. **ANALYSIS RATE**

After instrument calibration, one analyst can analyze approximately 12 samples in an 8-hour day.

F. **SAFETY INFORMATION**

Care should be taken when preparing or handling sodium hydroxide (NaOH), and sulfuric acid ($H_2SO_4$) reagents.

## III. APPARATUS AND CHEMICALS

A. **HARDWARE/GLASSWARE**

1. Class A volumetric flasks (1000 mL, 100 mL, 50 mL);
2. Syringe-mounted, 25 millimeter (mm) disposable filter, with 0.45 micro-pore Versapor™ acrylic copolymer filter.

B. **INSTRUMENTATION**

1. Ion chromatograph (Dionex Model 4000, with gradient analytical pump capabilities);
2. Anion separator column (Dionex AS4-A);
3. Anion guard column (Dionex AG4-A);
4. Anion membrane suppressor (Dionex AMMS);
5. Integrator (Maxima® integration system or equivalent), and
6. Anion trap column (Dionex ATC).

Parameters:

a. Eluents: (1) 0.5 millimolar (mM) and
(2) 98 mM sodium hydroxide.

b. Suppressor reagent: 40 mM sulfuric acid.

c. Flow rate and pressure: 1.5 mL/min at 750 pounds per square inch (psi) (typical).

d. Detector range: 30 uS full-scale.

e. Injection loop: 50 uL.

f. Gradient program: Enter the following program into the gradient-pump microprocessor:

| Time | Eluent | Percent | Notes | Flow (mL/min) |
|---|---|---|---|---|
| 0.0 | 1 | 100 | | 1.5 |
| 0.1 | 1 | 100 | (inject) | 1.5 |
| 0.2 | 1 | 100 | (inject off) | 1.5 |
| 2.0 | 1 | 100 | (gradient start) | 1.5 |
| 11.0 | 2 | 80 | (gradient stop) | 1.5 |
| 11.1 | 1 | 100 | (flush) | 1.5 |

C. **ANALYTES**

The Chemical Abstract Service (CAS) Registry Numbers are:

| Analyte | CAS Registry No. |
|---|---|
| IMPA | * |
| MPA | * |
| FC2A | 62-74-8 |

* not available

D. **REAGENTS AND SARMS**

1. ASTM Type I grade deionized water (DIW).
2. Sodium hydroxide [5 normal (N), prepared from carbonate-free, Dilut-It® analytical concentrate (Baker)].

3. Sulfuric acid ($H_2SO_4$), reagent grade [Baker, American Chemical Society (ACS)].

4. Standards [Standard Analytical Reference Materials (SARMs)]:

IMPA (SARM Compound 1264);

MPA (SARM Compound 1390);

FC2A (Fluka Chemical Company, Lot 261038).

## IV. CALIBRATION

### A. INITIAL CALIBRATION

1. Preparation of Calibration Standards.

a. Prepare 4,000 ug/mL individual calibration stock solutions (ICSS) by weighing 200 mg of IMPA and MPA standard material, and 256.3 mg of sodium fluoroacetate into separate 50 mL volumetric flasks and diluting each to volume with DIW. Prepare fresh at least semiannually and store at 4°C.

b. Prepare a 100 ug/mL combined calibration stock solution (CCSS) by adding 2.5 mL of each ICSS to a 100 mL volumetric flask and diluting to volume with DIW. Prepare fresh at least quarterly, and store at 4°C.

c. The working calibration standards are prepared fresh for each lot as follows:

| Standard | Concentration (ug/L) | Volume (mL) of CCSS to 100 mL with DIW |
|---|---|---|
| Blank | 0 | 0 |
| A | 100 | 0.1 |
| B | 200 | 0.2 |
| C | 400 | 0.4 |
| D | 1,000 | 1.0 |
| E | 2,000 | 2.0 |
| F | 4,000 | 4.0 |
| G | 6,000 | 6.0 |
| H | 10,000 | 10.0 |

Note: ug/L = micrograms per liter.

2. Instrument Calibration.

a. Establish a stable baseline with eluent No. 1 (0.5 mM sodium hydroxide) at 100 percent (20 to 30 min).

b. With integrator ready to receive trigger signal from ion chromatograph (due to narrow peak windows, manual start of integrator is not recommended), inject high standard (standard H - 10,000 ug/L).

c. Upon completion of Standard H run, test integration parameters for correct baseline placement and proper peak starts and peak ends.

d. Proceed with calibration standard run, from high to low, and a blank. Allow 16 min. between injections.

e. A reference standard, prepared independent of the calibration stock, is analyzed and must be within ± 10% of the true value. If the reference is not within ± 10% of the true value, it will be reanalyzed. If the reference is still outside of criteria, the reason must be determined and appropriate corrective action taken.

f. Analyze method blank and daily control spike samples.

3. Analysis of Calibration Data.

After analyzing the standards (i.e., one blank and eight standards), the data are tabulated and graphed. For precertification calibration, the duplicate calibration data are analyzed using the lack-of-fit (LOF) and zero-intercept (ZI) tests (USATHAMA QA Plan, 2nd Edition, March, 1987). The three analytes passed LOF/ZI tests, therefore, calibration curves are linear. Attachment 1 contains the precertification data.

4. Calibration Checks.

After every 12 samples and at the end of each day's analyses, the H Standard (10,000 ug/L) is reanalyzed. The reference standard is also reanalyzed at the end of each day's analyses. If the measured concentration for these standards is not ± 10% of the true value, the instrument is recalibrated and all samples since the last acceptable

calibration check are reanalyzed. After seven runs, ± 2 standard deviations of the percent recovery will be evaluated as a criteria.

B. **DAILY CALIBRATION**

Daily calibration and initial calibration curve and QC checks will be performed as stated in Sec.IV.A.

## V. CERTIFICATION TESTING

A. **PREPARATION OF SPIKING SOLUTION**

1. Individual Spike Stock Solutions (ISSS): Prepare separate 4,000 ug/mL stock standards (independent of stock calibration standards) as in Sec. IV.A.1. Prepare fresh at least semiannually and store at 4°C.
2. Combined Spike Stock Solution (CSSS): Prepare a 100 ug/mL combined intermediate spike solution by adding 2.5 mL of each control spike stock to a 100 mL volumetric flask and diluting to volume with DIW. Prepare fresh at least quarterly and store at 4°C.

B. **PREPARATION OF CERTIFICATION CONTROL SPIKE SAMPLES**

1. Control spike samples to be used in the method documentation are prepared as follows:

| Spike Level | Spike Conc. (ug/L) | Volume (mL) of CCSS Diluted to 50 mL |
|---|---|---|
| 0X | 0 | 0 |
| 0.5X | 100 | 0.05 |
| 1X | 200 | 0.1 |
| 2X | 400 | 0.2 |
| 5X | 1000 | 0.4 |
| 10X | 2000 | 1.0 |
| 20X | 4000 | 2.0 |
| 45X | 9000 | 4.5 |

2. The control spike samples are prepared as specified in Sec. V.B.1 each day for four consecutive days and analyzed as described in Sec.VII.C. Attachment 2 contains the method certification data.

## VI. SAMPLE HANDLING AND STORAGE

### A. SAMPLING PROCEDURE AND PRESERVATION

Samples for IMPA, FC2A and MPA analysis should be collected in amber glass containers with minimum headspace. Samples must be maintained in a temperature-controlled room at 4 degrees Celsius (°C).

### B. SAMPLING CONTAINERS

Samples are collected in amber-glass jars with teflon-lined lids (60 mL volume is adequate).

### C. STORAGE CONDITIONS

Samples are shipped and stored in the laboratory at 4°C.

### D. HOLDING TIME LIMITS

The holding time between sampling and analysis is forty days.

### E. SOLUTION VERIFICATION

Verification of the calibration standards is based on the analysis of daily QC spikes and analysis of independently prepared reference standards. The CSSS spiking stock solution should be verified within at least seven days prior to use. Since this method does not require sample extractions, verification can be accomplished by running a separate dilution of the CSSS on the same day the samples are analyzed.

## VII. PROCEDURE

Daily quality control spikes (see Sec.IX for preparation) and environmental samples are analyzed as follows:

### A. SEPARATIONS

Samples are analyzed by direct injection without prior digestion. The only separations are those during the analysis.

### B. CHEMICAL REACTION

There are no chemical reactions.

## VIII. CALCULATIONS

A standard curve is constructed using the linear regression equation for concentration versus response (peak area) using the least-squares method. The correlation coefficient must be 0.995 or greater. Using the linear regression equation, the sample concentration for each analyte is calculated from the measured peak area. If the sample concentration is within the certified range, the sample concentration will be reported as calculated in Sec.VIII. If the sample concentration is either not detected or below the certified reporting limit, the concentration will be reported as less than the certified reporting limit. If the sample concentration for any analyte is greater than the highest certified range, the sample should be diluted within the certified range and reanalyzed. The concentration in the diluted sample and the dilution factor are reported. The highest certified concentration range for all analytes is 9000 ug/L as given in Sec.II.C.

## IX. DAILY QUALITY CONTROL

### A. PREPARATION OF DAILY CONTROL SPIKE SAMPLES

1. Individual Spike Stock Solutions (ISSS): Prepare separate 4,000 ug/mL stock standards (independent of stock calibration standards) by weighing 200 mg of IMPA and MPA standard material, and 256.3 mg of sodium fluoroacetate into separate 50 mL volumetric flasks and diluting each to volume with DIW. Prepare fresh at least semiannually and store at 4°C.
2. Combined Spike Stock Solution (CSSS): Prepare a 100 ug/mL combined intermediate spike solution by adding 2.5 mL of each control spike stock to a 100 mL volumetric flask and diluting to volume with DIW. Prepare fresh at least quarterly and store at 4°C.
3. Since this method does not require sample extractions, spike stock solution verification can be accomplished by running a separate dilution of the CSSS on the same day the samples are analyzed.

4. With each daily lot of environmental samples, prepare the daily control spike samples as follows:

| Daily Control Spike | Spike Concentration (ug/L) | Volume (mL) of CSSS Diluted to 50 mL with DIW |
|---|---|---|
| Blank | 0 | 0 |
| Low Spike | 400 | 0.2 |
| High Spike | 2000 | 1.0 |
| High Spike | 2000 | 1.0 |
| Extended Range Spike | 8000 | 4.0 |

B. **CONTROL CHARTS**

Control charts are prepared using the percent recovery data from both the duplicate high level spikes and the low level spike calculated according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Found Conc.}}{\text{Spiked Concentration}} \times 100 \text{ percent}$$

The found response is corrected for method blank response, when necessary, prior to calculation of the found concentration. Preparation of control charts requires the following data:

1. Average percent recovery of the two high concentration spiked QC samples in each lot,
2. Difference between the two high concentration spiked QC samples in each lot.
3. Three-point moving average percent recovery for the low level spike in each lot, and
4. Three-point moving average difference for the low concentration spike.

For values that fall outside the control limits and data points that are deemed as outliers, the data will be evaluated and corrective action will be taken. Initial warning and control limits are presented in Table I.

**Table I: Initial Warning and Control Limits**

METHOD: Ion Chromatography
COMPOUND: IMPA, MPA, FC2A - water
UNITS: UGL

3 PT. MOVING AVERAGE $\bar{X}$ - R

CONC. = 400

| | UCL $\bar{X}$ | UWL $\bar{X}$ | = X | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|
| IMPA | 93.0 | 91.0 | 86.9 | 82.8 | 80.7 | 15.4 | 12.3 | 6.0 |
| MPA | 121.0 | 116.7 | 108.2 | 99.6 | 95.4 | 32.2 | 25.6 | 12.5 |
| FC2A | 105.4 | 101.4 | 93.5 | 85.6 | 81.7 | 29.9 | 23.8 | 11.6 |

SINGLE DAY $\bar{X}$ - R

CONC. = 2000

| | UCL $\bar{X}$ | UWL $\bar{X}$ | = X | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|
| IMPA | 100.9 | 100.2 | 98.7 | 97.3 | 96.6 | 3.8 | 2.9 | 1.1 |
| MPA | 107.4 | 105.9 | 102.8 | 99.6 | 98.1 | 8.2 | 6.3 | 2.5 |
| FC2A | 102.5 | 101.8 | 100.4 | 99.0 | 98.3 | 3.6 | 2.8 | 1.1 |

## X. REFERENCES

A. U.S. Army Toxic and Hazardous Materials Agency Quality Assurance Plan (December 1985, 2nd Edition, March 1987).

B. Dionex Model 4000i Instrument operation Manual. The Practice of Ion Chromatography, Frank Smith, Jr., and Richard C. Chang.

## XI. DATA

A. **OFF-THE-SHELF-CHARACTERIZATION** - Not required since stock solutions are USATHAMA SARMS.

B. **PRECERTIFICATION CALIBRATION DATA** - See attachment 1.

C. **DAILY CALIBRATION FOR CERTIFICATION AND CERTIFICATION DATA.**

See attachment 2.

D. **REFERENCE METHOD** - none available.

E. **EXAMPLE CHROMATOGRAM** - see attachment 3.

F. **LOT FOLDER ORGANIZATION INCLUDING METHOD SUMMARY** - see attachment 4.

Contract No. DAAA15-87-D0015
Task Order 00002AN

# DEVELOPMENT OF STANDARD ANALYTICAL METHODS EXPLOSIVES IN WATER BY HIGH PRESSURE LIQUID CHROMTOGRAPHY CERTIFICATION CALIBRATION REPORT

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**Prepared for:**

**U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY**
**Aberdeen Proving Ground, MD. 21010-5401**

Environmental Science & Engineering, Inc.
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Gainesville, Florida

TITLE: DETERMINATION OF DIISOPROPYLMETHYLPHOSPHONATE AND DIMETHYLMETHYLPHOSPHONATE IN ENVIRONMENTAL WATER SAMPLES (METHOD T8)

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IV. STANDARDS

V. PROCEDURE

VI. CALCULATIONS

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VIII. ATTACHMENTS

TITLE: DETERMINATION OF DIISOPROPYLMETHYLPHOSPHONATE AND DIMETHYLMETHYLPHOSPHONATE IN ENVIRONMENTAL WATER SAMPLES (METHOD T8)

## I. APPLICATION

This method is applicable to the quantitative determination of the following compounds in environmental water samples:

Diisopropylmethylphosphonate (DIMP)
Dimethylmethylphosphonate (DMMP)

### A. TESTED CONCENTRATION RANGE

The tested concentration range in "standard water" samples are:

| Analyte | Tested Concentration Range (µg/L) |
|---|---|
| DIMP | 10.48 to 209.6 |
| DMMP | 15.24 to 304.8 |

### B. SENSITIVITY

The normalized responses (integrator counts corrected for attenuation) at the standard water detection limits are:

| Analyte | Area Count |
|---|---|
| DIMP | 3,650 |
| DMMP | 3,950 |

### C. DETECTION LIMITS

The "standard water" detection limits, calculated according to the U.S. Army Environmental Center (USAEC) detection limit program are:

| Analyte | Detection Limit (µg/L) | Upper Certified Range (µg/L) |
|---|---|---|
| DIMP | 10.5 | 210.0 |
| DMMP | 15.2 | 305.0 |

### D. GENERAL METHOD

This method invloves the direct injection of an aliquot of an environmental water sample onto a gas chromatographic (GC) column and analyzed by GC with flame photometric detection.

### E. INTERFERENCES

Reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be free from interferences which have retention times equal to the retention times of the compounds of interest.

### F. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 samples in an 8-hour day.

### G. PRESERVATION AND HOLDING TIMES

Samples should be collected in amber glass bottles with teflon liners and stored at 4° ± 2 °C. Samples must be analyzed within 40 days after collection.

## II. CHEMISTRY

### A. CHEMICAL ABSTRACT SERVICE (CAS) NUMBERS

The CAS registry numbers for the compounds are:

| Analyte | CAS Registry Number |
|---|---|
| DIMP | 1445-75-6 |
| DMMP | 756-79-6 |

### B. CHEMICAL REACTIONS

A measured volume of sample is directly injected onto the gas chromatographic column. Chromatographic conditions are described which

permit the separation and measurement of DIMP and DMMP in "standard" or environmental water samples. Qualitative identification is performed using retention times, and quantitative analysis is performed using standard curves.

## III. APPARATUS

### A. INSTRUMENTATION

A Hewlett - Packard 5890 gas chromatograph (GC) with a flame photometric detector (FPD) equipped with a Hewlett - Packard 7673 automatic sampler and interfaced to a PE Nelson Turbochrom Chromatographic Workstation.

### B. PARAMETERS

1. Instrument: HP 5890A equipped with an autosampler (Model 7673)

2. Detector: FPD with selective spectral detection of phosphorus [530-nanometer (nm) filter]

3. Column: 5-percent SP-1000 on 100/120 supelcoport glass column [2-m x 2-mm inside diameter (ID)]

4. Gas flow:
   Helium--50 mL/min
   Hydrogen--100 mL/min
   Air 1--120 mL/min

5. Temperature:
   Injector--240 °C
   Detector--240 °C
   Oven--60 °C (no hold), then programmed at 10 °C/min to 100 °C, then 50 °C/min to 240 °C, hold for 2.2 minutes.

6. Injection volume: 5.0 $\mu$L

7. Retention times:

| Analyte | Retention Time (min) |
|---|---|
| DIMP | 6.0 ± 0.1 |
| DMMP | 6.5 ± 0.1 |

8. Retention windows: DIMP ± 0.1 min
DMMP ± 0.1 min

### C. HARDWARE/GLASSWARE

1. Volumetric flasks [50-, 100-, and 1,000-mL];
2. Volumetric pipettes (1.0- and 2.0-mL);
3. Microsyringes (100- and 1,000-μL);
4. Pasteur pipettes (disposable);
5. Graduated cylinder (100-mL);
6. Amber glass vials (8-mL with Teflon®-lined screw caps);
7. Glass vials (2-mL with Teflon®-lined, crimp-seal caps for use with an automatic sampler);
8. Amber bottles (60 mL with Teflon®-lined screw caps);
9. Aluminum foil;
10. Stainless steel spatulas; and
11. Analytical balance [Mettler AE160 or equivalent, with 0.0001-gram (g) sensitivity].

### D. CHEMICALS

1. Sodium chloride (analytical-grade);
2. Sodium sulfate (analytical-grade);
3. "Standard water" [distilled water containing 100 milligrams per liter (mg/L) each of sulfate and chloride];
4. DIMP [Standard Analytical Reference Material (SARM), identification No. PA2334, obtained from USAEC]; and
5. DMMP (SARM, identification No. PA1827, obtained from USAEC).

## IV. STANDARDS

### A. INITIAL INSTRUMENT CALIBRATION STANDARDS

1. Individual stock calibration standards are prepared by weighing approximately 50 mg each of DIMP and DMMP into separate 50-mL volumetric flasks, then diluting to volume with deionized water. This standard is stable for 1 year if stored in the dark at 4° ± 2 °C. The

actual concentrations in the stock calibration standards prepared in the above manner were:

| Analyte | Concentration of Individual Calibration Standard (μg/mL) |
|---|---|
| DIMP | 958 |
| DMMP | 1,120 |

2. A composite secondary stock calibration standard is prepared by adding 1 mL of each individual stock calibration standard to deionized water in a 100-mL volumetric flask and then diluting to volume with deionized water. The actual concentrations in the composite secondary stock calibration standard prepared in the above manner were:

| Analyte | Concentration of Composite Secondary Stock Calibration Standard (μg/mL) |
|---|---|
| DIMP | 9.58 |
| DMMP | 11.20 |

This stock is stable for 1 year if stored in the dark at 4° ± 2 °C.

3. Composite working calibration standards are prepared using the composite secondary stock calibration standard, microsyringes, and volumetric pipettes as shown in Table 1. Each composite working calibration standard is prepared by adding the specified volume of the composite secondary stock calibration standard to deionized water contained in a 100-mL volumetric flask and then diluting to volume with deionized water.

4. For initial instrument calibration, inject 5.0 μL of each of the working calibration standards prepared in Table 1. Plot the linear regression of the normalized response versus the concentration of standard injected for each standard to obtain a working curve. This curve must have a correlation coefficient of 0.995 or greater.

## B. DAILY INSTRUMENT CALIBRATION STANDARDS

1. A minimum of three working calibration standards and one blank are analyzed daily for instrument calibration.

   a. At a minimum, 5.0 $\mu$L of each of the following working calibration standards (from Table 1) are analyzed daily:

      (1) Blank,
      (2) Working calibration standard .5A,
      (3) Working calibration standard D, and
      (4) Working calibration standard F.

   b. Plot the linear regression of the normalized response versus the concentration of standard injected for each standard to obtain a working curve. This curve must have a correlation coefficient of 0.995 or greater.

   c. At the end of the daily instrumental analysis, inject 5.0 $\mu$L of working calibration standard D. The response of this end-of-day analysis should be $\pm$ 25 percent of the response obtained from the analysis of working calibration standard D analyzed earlier in the day. If not, the instrument should be recalibrated and the sample extracts reanalyzed.

2. Retention Time Windows

   Retention time windows for a method are established based on historical data of daily performance of the method or by using retention windows from a reference method. If a reference method with established windows is not available, the windows are calculated by calculating the relative standard deviation of the retention time variation throughout the run for all standards, control samples and continuing calibration standards, and multiplying the result by three and rounding to 1 significant figure. The windows are expressed in absolute minutes where the retention variation is minimal thoughout the chromatographic run and as a percentage of the retention time in methods whose variation is proportional to retention time. The retention window is applied to retention times from a specified

standard during initial or daily calibration. These retention window values are entered into the method used for processing the chromatographic data for computerized identification /rejection of detected peaks. The analyst can override the identification/rejection of a peak by providing documentation of his/her decision.

## C. CONTROL SPIKES FOR METHOD CERTIFICATION

1. A composite stock control spiking solution is prepared by weighing approximately 50 mg of DIMP and 70 mg of DMMP into one 50-mL volumetric flask, then diluting to volume with deionized water. The actual concentrations in the composite stock spiking solution prepared in the above manner were:

| Analyte | Concentration of Composite Stock Control Spiking Solution (µg/mL) |
|---|---|
| DIMP | 1,048 |
| DMMP | 1,524 |

2. A composite secondary stock control spiking solution is prepared by adding 1 mL of the composite stock control spiking solution into a 100-mL volumetric flask, then diluting to volume with "standard" water. The actual concentration in the secondary composite stock spiking solution prepared in the above manner was:

| Analyte | Concentration of Secondary Composite Stock Control Spiking Solution (µg/mL) |
|---|---|
| DIMP | 10.48 |
| DMMP | 15.24 |

3. Certification control spike samples are prepared using the composite secondary stock control spiking solution, microsyringes, and volumetric pipettes as shown in Table 2. Each certification control spike sample is prepared by adding the specified volume of the

secondary stock control spiking solution to "standard water" contained in a 100-mL volumetric flask and then diluting to volume with "standard water." (To prepare "standard water" for control spikes, weigh 1.48 g of anhydrous sodium sulfate into a 1-liter volumetric flask and dilute to volume with distilled water. Weigh 1.65 g of reagent-grade dry sodium chloride into a separate 1-liter volumetric flask and dilute to volume with distilled water. Transfer 100 mL of each of the two solutions to a 1-liter volumetric flask and dilute to volume with distilled water to produce 1 liter of "standard water.")

### D. DAILY CONTROL SPIKE SAMPLES

With each daily lot of environmental samples, analyze the daily control spike samples shown in Table 3.

## V. PROCEDURE

### A. CERTIFICATION

Certification control spike samples and environmental water samples require no further preparation. Approximately 1 mL of each is transferred into an autosampler vial using disposable pipettes, followed by instrumental analysis (direct injection onto the GC column). The remainder of the samples should be refrigerated at 4° ± 2 °C in amber glass vials until analysis.

### B. ANALYSIS

1. Perform daily instrument calibration as described in Section IV.A.4.
2. Place the sample extracts in the autosampler tray and inject 5 $\mu$L of each sample.

## VI. CALCULATIONS

The concentration (in $\mu$g/L) for each analyte is taken directly from the standard curve.

## VII. REFERENCES

None.

## VIII. DATA

See Attachments

## XI. ATTACHMENTS

A. **ATTACHMENT A** - Control Spikes for Method Certification

B. **ATTACHMENT B** - Preparation of Control Spike Samples for Certification

Table 1. Preparation of Composite Working Calibration Standards

| Volume of Composite Secondary Stock Calibration Standard Used (mL) | Final Volume (mL) | Composite Working Calibration Standard Prepared | Concentration of Prepared Standard (µg/L) DIMP | DMMP |
|---|---|---|---|---|
| 0. | 100 | Blank | 0. | 0. |
| 0.050 | 100 | .5A | 4.79 | 5.60 |
| 0.100 | 100 | A | 9.58 | 11.20 |
| 0.200 | 100 | B | 19.16 | 22.40 |
| 0.400 | 100 | C | 38.32 | 44.80 |
| 0.800 | 100 | D | 76.64 | 89.60 |
| 2.00 | 100 | E | 191.60 | 224.00 |
| 4.00 | 100 | F | 383.20 | 448.00 |

Source: ESE

Table 2. Preparation of Certification Control Spike Samples

| Volume of Composite Secondary Stock Control Spiking Solution Used (mL) | Final Volume (mL) | Certification Control Spike Sample Prepared | Concentration of Prepared Standard (µg/L) DIMP | DMMP |
|---|---|---|---|---|
| 0. | 100 | Blank | 0. | 0. |
| 0.100 | 100 | A | 10.48 | 15.24 |
| 0.200 | 100 | B | 20.96 | 30.48 |
| 0.400 | 100 | C | 41.92 | 60.96 |
| 0.800 | 100 | D | 83.84 | 121.92 |
| 2.00 | 100 | E | 209.60 | 304.80 |

Source: ESE

Table 3. Preparation of Daily Control Spikes

| Daily Control Spike Number | Volume of Composite Secondary Stock Control Spiking Solution Used (mL) | Final Volume (mL) | Concentration of Prepared Standard (µg/L) DIMP | DMMP |
|---|---|---|---|---|
| Blank | 0. | 100 | 0. | 0. |
| Low-Level | 0.2 | 100 | 20.96 | 30.48 |
| High-Level | 0.8 | 100 | 83.84 | 121.92 |
| High-Level | 0.8 | 100 | 83.84 | 121.92 |

Source: ESE

Environmental Science & Engineering, Inc.
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TITLE: DETERMINATION OF EXPLOSIVES IN WATER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (METHOD UW32)

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XI. ATTACHMENTS

TITLE: DETERMINATION OF EXPLOSIVES IN WATER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (METHOD UW32)

## I. SUMMARY

### A. ANALYTES

This method is applicable to the Class 1 analysis of the following nitroaromatic organic compounds in environmental water samples:

Analytes

1,3-Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
HMX (octahydro-1,3,5,7-tetranitro-s-tetrazocine)
Nitrobenzene
RDX (hexahydro-1,3,5-trinitro-s-triazine)
Tetryl (N-methyl-N,2,4,6-tetranitrobenzenamine)
1,3,5-Trinitrobenzene
2,4,6-Trinitrotoluene
4-Amino, 2,6-Dinitrotoluene
2-Amino, 4,6-Dinitrotoluene
2-Nitrotoluene
3-Nitrotoluene
4-Nitrotoluene

### B. MATRIX

This method is applicable to all environmental water matrices.

### C. GENERAL METHOD

The method employs solid phase extraction of 500 mL of an environmental water sample using a tube packed with Porapak R. The water sample is spiked with a surrogate (3,4-Dinitrotoluene) prior to the solid phase extraction. The target compounds are desorbed with 3 mL of acetonitrile, concentrated to 2 mL with a gentle stream of nitrogen and diluted to a final volume of 6 mL with water. The compounds are separated by high performance liquid chromatography (HPLC) using isocratic elution and detected using ultraviolet (UV) absorbance at 250 nanometers (nm).

## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The certification testing ranges in micrograms per liter ($\mu$g/L) are:

| Analyte | Tested Concentration Range ($\mu$g/L) |
|---|---|
| 1,3-Dinitrobenzene | 0.138 - 55.0 |
| 2,4-Dinitrotoluene | 0.053 - 21.2 |
| 2,6-Dinitrotoluene | 0.061 - 24.4 |
| HMX | 1.21 - 120.8 |
| Nitrobenzene | 0.645 - 129.0 |
| RDX | 1.17 - 116.8 |
| Tetryl | 1.08 - 43.0 |
| 1,3,5-Trinitrobenzene | 0.148 - 59.2 |
| 2,4,6-Trinitrotoluene | 0.280 - 112.0 |
| 4-Amino, 2,6-Dinitrotoluene | 0.052 - 20.8 |
| 2-Amino, 4,6-Dinitrotoluene | 0.055 - 22.0 |
| 2-Nitrotoluene | 0.307 - 122.6 |
| 3-Nitrotoluene | 0.292 - 116.8 |
| 4-Nitrotoluene | 0.301 - 120.4 |

## B. SENSITIVITY

The instrumental responses for each compound, reported in peak area units at the certified reporting limit (CRL) are:

| Analyte | CRL (µg/L) | Area Counts |
|---|---|---|
| 1,3-Dinitrobenzene | 0.611 | 110,000 |
| 2,4-Dinitrotoluene | 0.0637 | 13,000 |
| 2,6-Dinitrotoluene | 0.0738 | 8,500 |
| HMX | 1.21 | 83,000 |
| Nitrobenzene | 0.645 | 62,000 |
| RDX | 1.17 | 84,000 |
| Tetryl | 1.56 | 300,000 |
| 1,3,5-Trinitrobenzene | 0.449 | 73,000 |
| 2,4,6-Trinitrotoluene | 0.635 | 100,000 |
| 4-Amino,2,6-Dinitrotoluene | 1.571 | 200,000 |
| 2-Amino,4,6-Dinitrotoluene | 0.157 | 22,000 |
| 2-Nitrotoluene | 0.406 | 45,000 |
| 3-Nitrotoluene | 1.40 | 83,000 |
| 4-Nitrotoluene | 1.11 | 51,000 |

## C. REPORTING LIMITS

The certified reporting limits (CRLs) and upper certified limit (UCL) for each analyte in environmental water samples are:

| Analyte | CRL (µg/L) | | UCL (µg/L) |
|---|---|---|---|
| 1,3-Dinitrobenzene | 0.611 | (0.306) | 55.0 |
| 2,4-Dinitrotoluene | 0.0637 | (0.032) | 21.2 |
| 2,6-Dinitrotoluene | 0.0738 | (0.037) | 24.4 |
| HMX | 1.21 | (0.647) | 120.8 |
| Nitrobenzene | 0.645 | (0.394) | 129.0 |
| RDX | 1.17 | (0.615) | 116.8 |
| Tetryl | 1.56 | (0.782) | 107.5 |
| 1,3,5-Trinitrobenzene | 0.449 | (0.224) | 59.2 |
| 2,4,6-Trinitrotoluene | 0.635 | (0.318) | 112.0 |
| 4-Amino,2,6-Dinitrotoluene | 1.57 | (0.787) | 20.8 |
| 2-Amino,4,6-Dinitrotoluene | 0.157 | (0.079) | 22.0 |
| 2-Nitrotoluene | 0.406 | (0.203) | 122.6 |
| 3-Nitrotoluene | 1.40 | (0.699) | 116.8 |
| 4-Nitrotoluene | 1.11 | (0.556) | 120.4 |

* Value in () is criterion of detection (COD)

## D. INTERFERENCES

Any materials which are adsorbed from water on the cartridge, coelute with the explosives through the HPLC column, and which absorb ultraviolet radiation at 250 nm may cause interferences. The Porapak R material must be thoroughly cleaned to minimize interference. A late eluting component arising from the Porapak R requires a 6 minute delay in sample injection following analysis of an extract. Since this analysis generally uses an autosampler, this problem is not significant. If, however, the analysis is done with manual injection, the injections need to be delayed. Carryover from analysis of a highly contaminated sample can result in apparent contamination of the succeeding samples analyzed. Such contamination is often manifest by the presence of unusually broad chromatographic peaks nested among narrower peaks. This interference is minimized by analyzing apparent heavily contaminated samples at the end of a run, or running blanks after heavily contaminated samples until carryover is removed, and/or rinsing the system with a mobile phase containing a high proportion of organic modifier until the contamination is removed.

## E. ANALYSIS RATE

After instrument calibration, one analyst can analyze approximately 12 samples in an 8-hour day.

## F. SAFETY INFORMATION

The target compounds in this method are toxic explosives and some are known carcinogens, e.g. 2,4-Dinitrotoluene. The preparation of all standards should be performed in a laboratory hood. Adequate dermal protection must be used when handling samples and standards. Most of these compounds are either primary or secondary explosives and should be handled with care to avoid contact with electrostatic shocks or impacts. Tetryl and RDX have intermediate sensitivity between initiating explosives and explosives used as bursting charges. Tetryl is toxic when taken internally or by skin contact. RDX, HMX, and 246TNT are used as bursting charge explosives. Although 246TNT is less sensitive to friction and impact than many other high explosives, it can be detonated with moderate force when confined between metal surfaces such as on the threads of bolts. 246TNT will form sensitive materials in the presence of alkalies.

## III. APPARATUS AND CHEMICALS

### A. GLASSWARE/HARDWARE

1. Sorbent Cartridge - 6 mL Disposable Solid Extraction Columns (J.T. Baker, Phillipsburg, N.J.) were used. 0.5 g of cleaned Porapak R was added to each tube and a frit placed on top of the sorbent.
2. Baker 10 Solid Phase Extraction System, (J.T. Baker, Phillipsburg, NJ) including manifold, 75 mL reservoirs and adapters.
3. Class A Volumetric flasks - 10, 100 and 500 mL.
4. Class A Volumetric pipets - 0.5, 1.0, 2.0 mL.
5. Aspirator.
6. Disposable micro pipets - 25, 50, 100 and 200 $\mu$L.
7. Graduated centrifuge tubes.

### B. INSTRUMENTATION AND INSTRUMENTAL CONDITIONS

1. HPLC - Primary: Shimadzu model SCL-10A HPLC with Shimadzu model SIL-10A autosampler (or, equivalent).
   - Confirmation: Shimadzu model LC-6A with Shimadzu SIL-10A autosampler or equivalent.
2. Detector: Shimadzu SPD-10A variable wavelength ultraviolet (UV) absorbance detector set at 250 nm.
3. Column: Phenomenex ODS (octadecylsilane), reverse-phase column, 25 centimeters (cm) length x 4.6 millimeters (mm) I.D., 5 micrometers ($\mu$m) particle size (Phenomenex, Inc. Torrance, Ca.).
4. Shimadzu SIL6B autosampler.
5. Mobilephase:Isocratic, 55%methanol/45% water (V/V).
6. Flow rate: 0.8 milliliters per minute (mL/min).
7. Sample Volume: 500 $\mu$L.

C. ANALYTES

| Analyte | USAEC Abbrev. | CAS Number | Melting* Point C | Spec.* Gravity |
|---|---|---|---|---|
| 1,3-Dinitrobenzene | 13DNB | 99-65-01 | 89-90 | 1.546 |
| 2,4-Dinitrotoluene | 24DNT | 121-14-2 | 70.5 | 1.3208 |
| 2,6-Dinitrotoluene | 26DNT | 606-20-2 | 70.5 | 1.3208 |
| HMX | HMX | 2691-41-0 | | |
| Nitrobenzene | NB | 98-95-3 | 5.7 | 1.1987 |
| RDX | RDX | 121-84-4 | 205-206 | 1.82 |
| Tetryl | TETRYL | 479-45-8 | 130-132 | 1.57 |
| 1,3,5-Trinitrobenzene | 135TNB | 25377-32-6 | 122 | 1.688 |
| 2,4,6-Trinitrotoluene | 246TNT | 118-96-7 | 80.1 | 1.654 |
| 4-A,2,6-Dinitrotoluene | 4A26DT | 1946-51-0 | 2-A,4,6- | |
| Dinitrotoluene | 2A46DT | 118-96-7 | | |
| 2-Nitrotoluene | 2NT | 88-72-2 | | 1.1629 |
| 3-Nitrotoluene | 3NT | 99-08-1 | | 1.1571 |
| 4-Nitrotoluene | 4NT | 99-99-0 | 51.7 | 1.299 |
| 3,4-Dinitrotoluene (Surr.) | 34DNT | 610-39-9 | 61 | 1.32 |

*Merck Index, 10th Ed.

D. REAGENTS AND SARMS

1. The standards used for target compound certification and calibration are USAEC supplied standard analytical reference materials (SARMS) except for the additional compounds added to the method (2A46DT, 4A26DT, 2NT, 3NT, 4NT and the surrogate 34DNT). Standard Material for the additional compounds were obtained from 2 sources: Aldrich Chemical Co. (2NT, 3NT, 4NT, and 34DNT); and, the Naval Surface Weapons Center -NSWC (2A46DT and 4A26DT). HPLC characterizations of the non USAEC SARMS showed that only the compounds of interest were present. The USAEC SARMS used for this certification, and their lot numbers are listed below:

| Analyte | SARM LOT NUMBER |
|---|---|
| 1,3-Dinitrobenzene | 2250 |
| 2,4-Dinitrotoluene | 1147 |
| 2,6-Dinitrotoluene | 1148 |
| HMX | 1217 |
| Nitrobenzene | 2177 |
| RDX | 1130 |
| Tetryl | 1149 |
| 1,3,5-Trinitrobenzene | 1154 |
| 2,4,6-Trinitrotoluene | 1129 |

2. Methanol (HPLC grade - American Burdick & Jackson, McGaw Park, IL).
3. Water (ASTM Type II/HPLC grade - American Burdick & Jackson, McGaw Park, IL).
4. Acetonitrile (HPLC grade - American Burdick & Jackson, McGaw Park, IL).
5. Acetone (HPLC grade - American Burdick & Jackson, McGaw Park, IL).
6. Porapak R, 80-100 Mesh - (Supleco, Inc., Bellefont, PA), cleaned by six acetone extractions, six acetonitrile extractions and six methanol extractions followed by air drying. (75 cc of the resin was in contact with 200 mL of solvent for 10 minutes in a sonicator at each extraction step).

## IV. CALIBRATION

Table IV-1 summarizes the preparation for calibration stock solutions and calibration standards used during precertification and certification. Table IV-2 summarizes the proposed preparation scheme for calibration stock solutions and calibration standards to be used during analysis for initial and daily calibration standards. To summarize the information presented, individual and separate primary stock solutions (SPSS's) are prepared for each compound by dissolving a known amount of the SARM in 10 or 25 mL of acetonitrile. The nominal concentrations for these SPSS solutions range from 500 to 2500 $\mu$g/mL. Two subsets of combined primary stock solutions are prepared to prevent degradation due to specific combinations of compounds. These two Stock A solutions are made up in 25 mL final volumes with acetonitrile. The range of concentrations in these two solutions is from ~ 2 to 15 $\mu$g/mL for the proposed preparation scheme. A 10 mL final volume of each calibration standard is then prepared by dilution of the stocks to have a 33% acetonitrile concentration to match the acetonitrile concentrations in the final extract of the samples.

### A. INITIAL CALIBRATION

1. Preparation of Standards

Precertification and Certification Calibration Standards. Separate primary stock standards (SPSS) and calibration standards for precertification and certification for each target compound were prepared according to the dilution scheme presented in Table IV-1.

Initial and Daily Calibration Standards. Table IV-2 presents the scheme for preparing separate primary stock solutions, combined stock solutions and calibration standards used for initial and daily calibrations during actual sample analysis. For initial calibration, Standards E2, E4, D, D2, D5, C, C2, C5, B, and a blank described in Table IV-2 are prepared. These solutions are prepared fresh as needed but not stored for use longer than one month at 4° ± 2 °C (10 mL of standard usually only lasts 8 runs). Reference materials are not readily available for verification of the calibration curve, therefore independently prepared stock solutions are prepared for reference samples. The SPSS solutions should be prepared fresh every 12 months and stored in the dark at 4° ± 2 °C. Tetryl needs to be made fresh if degradation is evident but at least every 12 months. Each separate stock solution is made to volume with acetonitrile. The Stock A solutions should be prepared fresh

every 6 months (if 2 separate Stock A solutions are not made, degradation of some compounds may occur). HPLC-grade water is used for dilution to final volumes for the composite calibration standards (acetonitrile concentrations need to be no greater than 33%). Daily calibration standards are D, D5, C2, B, and a blank as outlined in Table IV-2.

2. Instrument Calibration

Inject 500 $\mu$L of each calibration standard presented in Table IV-2, and analyze a reference standard following calibration and at the end of the run. Instrument conditions and the column are described in Sec.III.B.

Independent Reference Standard.
An independent stock will be prepared to serve as a reference standard for explosives in water. The independent reference standard was analyzed during precertification and certification calibration. The results were within $\pm$ 25% and are reported in Attachment

The independent reference must be analyzed with initial calibration run and results must be within $\pm$ 25% of the expected value, for the calibration to be considered valid. If the analysis of the independent reference standard fails, the source of the problem must be identified and corrected. The results of the second analysis of the independent reference standard must be within the acceptable limits before the analysis of samples may proceed. Since a new initial calibration is performed daily, a reference is required at least weekly.

3. Analysis of Calibration Data.

After analyzing the standards (i.e., one blank and seven standards), the data are tabulated and graphed. For precertification calibration, the duplicate calibration data are analyzed using the lack-of-fit (LOF) and zero-intercept (ZI) tests (USATHAMA QA Plan, January, 1990). Calibration curves for all compounds are considered quadratic even though 4 compounds showed linear relationships during precertification.

## B. DAILY CALIBRATION.

1. Preparation of Standards
Standards D, D5, C2, B, and a Blank are prepared as shown is Table IV-2.

2. Instrument Calibration and Calibration Checks

At the beginning of each analytical run, inject 500 uL of the four daily calibration standards. Found concentrations are determined from the quadratic regression equations of initial calibration, therefore the percent recovery (100*Found/Target) must be within 25% of the true value (100%). After every seven lots, a new initial calibration curve is run. At the end of the analytical run, the CCS-1 standard will be analyzed. As above the response must be within 25%. If not, the standard is reanalyzed. If it is still not within 25% a new initial calibration curve is run for analysis of the samples.

3. Analysis of Calibration Data

The response of the target compounds in the end run standard (B) must be less than 25 percent different from response factors obtained from Standard B analyzed at the beginning of the day. If the response is greater than 25 percent different, the standard will be reanalyzed. If reanalysis still fails the 25-percent criterion, a new initial calibration must be performed and all analyses since the last acceptable calibration must be repeated. After seven calibrations have been completed, the end of run response must agree to within two times the standard deviation of the mean response rather than a percentage. Failure of the tighter criteria will not be an automatic requirement for reanalyses if documentation exists to ensure that data quality of the samples is not affected by instrument drift (i.e increase in sensitivity and all samples less than the CRL). In addition, drifts outside criteria and within 25 percent should be evaluated in the light of expected method performance.

4. Retention Time Windows

Retention windows will be determined on a daily basis using the calibration standards used throughout the analytical run. Daily window

determinations will yield a window based on daily instrument performance. Additionally because the calibration curve is being used variation of the retention time with analyte concentration can be taken into account as well as daily instrument drift. The following procedure will be used to calculate retention time windows:

1. The mean retention time will be calculated as the mean retention time of the daily calibration standards.

2. The standard deviation of the daily retention window will be determined using the retention times of the daily calibration standards and the continuing calibration standards.

3. The daily retention window for an analyte will be the mean retention time plus or minus three times the standard deviation.

All of the retention times for reported analytes in samples and continuing calibration standards will be tested against the calculated windows. If an analyte retention time is outside of the calculated window documentation must be provided in order to justify the qualitative identification of the peak.

Justification for overriding the retention windows will include examining the relative retention window (RRT)(RRT = retention time of analyte/retention time of the surrogate). The RRT will compensate for changes in retention time due to sample loading and small changes in retention time due to other factors. RRT windows will be calculated in the same manner as the retention windows.

## V. CERTIFICATION TESTING

Spiked samples for certification testing are prepared in standard water (ASTM Type II grade water containing 100 mg/L of sulfate and chloride, see Sections 5.6.1 and 5.7.1 Table 5-1, of the USATHAMA QA Plan, January, 1990 Edition). Three sets of two composite stock solutions (Stocks A, B and C) (see Table V-1) are prepared for use as a spiking solution. Spiking is performed for certification testing on four separate days. Analysis of the spiked samples follows the procedure outlined in Section VII. The target versus found data are analyzed using the lack-of-fit (LOF) and zero-intercept (ZI) tests (USATHAMA QA Plan, January, 1990). The result of these tests and the certification data are presented in Attachment 3 for each target analyte.

## VI. SAMPLE HANDLING AND STORAGE

### A. SAMPLING PROCEDURE

Samples will be collected using adequate dermal and inhalation protection and must follow Sections 6.10 and 6.11 of the USATHAMA Quality Assurance Plan (January 1990).

### B. CONTAINERS

One liter amber colored glass jars with Teflon-lined lids are required.

### C. STORAGE CONDITIONS

Samples and extracts should be kept chilled to 4 °C ± 2 °C and in the dark.

### D. HOLDING TIME LIMITS

Samples must be extracted within 7 days of sampling date, and the extract must be analyzed within 40 days of extraction date.

### E. SOLUTION VERIFICATION

Verification of the calibration standards is based on the analyses of daily QC spikes and analysis of independent reference standards (during initial calibration). The combined intermediate stock solutions for control spikes are analyzed weekly to verify if target concentrations are still valid. The recovery of this check must be within 25 percent of the true value or ± 2 standard deviations of recent performance (last 7 runs). If criteria cannot be met for the target compounds,newstock spiking solutions need to be prepared.

## VII. PROCEDURE

### A. EXTRACTION

A 6 mL Baker Disposable Extraction Column with a frit on one end is repacked with 0.5 g of cleaned Porapak R another frit is added to the top of the column, to help pack it and prevent channeling. Preconditioning of the column is then required and once preconditioning starts, care should be taken to prevent the column from going dry until elution is complete. The column is

preconditioned by rinsing with 15 mL of acetonitrile and 30 mL of water. 500 mL of the sample is measured and passed through the column at a rate of 10 mL/minute (Note: **DO NOT ALLOW THE COLUMN TO GO DRY**). The column is then slowly eluted with 1 mL of acetonitrile at a flow rate of no greater than 3 mL/min. in a graduated centrifuge tube. Be sure all of the acetonitrile is blown out of the column. The eluent is then concentrated with a gentle stream of nitrogen to a 2 mL final volume. The extract is then diluted to a 6 mL final volume with ASTM Type II/HPLC water.

## B. CHEMICAL REACTIONS

This method does not involve any chemical reactions.

## C. INSTRUMENTAL ANALYSIS

Instrumental analysis involves injection of 500 uL of the extract onto the analytical column described in Section III.B. The instrumental conditions are specified in Section III.B. and the integrated output of the UV detector is used in the calculations of Section VIII. All responses within each analyte's retention window which corresponds to concentrations greater than or equal to the respective CRL must be confirmed by analysis on a second HPLC column with dissimilar bonded phase material (e.g. CN instead of ODS). The conditions for this second column confirmation are described in Appendix A.

## D. CONFIRMATION ANALYSIS

Analytes that are tentatively identified on the primary column must be confirmed by analysis on a different column with a different liquid phase. In order to confirm an analyte a response must be present in the retention windows for the analyte on both the primary column and the confirmation column. The retention windows will be calculated the same way for both columns. Decision points to be made for the identification and reporting of a target analyte are:

1. Is there a response in the retention window of a target analyte on the primary column and the response is above the CRL?

   No. No further action is necessary and the analyte is reported as <CRL.

Yes. Analyze the sample extract on the confirmation column.

2. Is there a response on the confirmation column in the retention window of the target analyte and the response is above the criterion of detection?

   No. The analyte is not confirmed and the analyte is reported as <CRL adjusted for any dilutions required.

   Yes. Determine ability to identify peak.

3. Is the peak well defined?

   Yes. The analyte is confirmed and the response of the target analyte is reported from the primary column analysis.

   No. There is considerable interference on the confirmation column analysis which in the analyst's judgement precludes their ability to identify a peak in the retention window of interest. The analyte is considered as not confirmable. The analyte will be reported with the concentration calculated from the primary column and flagged with a "Q".

## VIII. CALCULATIONS

Quadratic regression equations are calculated from the initial calibration curve data by regressing the response versus the concentration for each compound. The concentration of a target compound in the sample extract is calculated by substituting the response into the calibration curve equation. The same injection volume is used for standards and sample extracts. The following formula is used to calculate sample concentrations (SC).

$$SC\ (\mu g/L) = \frac{EC \times EV}{SV}$$

Where EC = Extract concentration determined from calibration curve in $\mu g/L$.

EV = Extract volume (6 mL).

SV = Sample volume (500 mL).

## IX. DAILY QUALITY CONTROL

### A. CONTROL SAMPLES

Daily quality control samples consist of a standard matrix method blank (ASTM Type I water), a single low level spike at approximately twice the certified reporting limit, duplicate high spikes at 10 times the low spike, and a single level extended range spike at approximately 80% of the upper certified range. These quality control samples should be carried throughout the entire method at the same time samples are run. Table IX-1 documents the preparation of stock spiking solutions and preparation of spiking solutions. Separate Primary Stock Standards (SPSS) at various concentrations near 1000 ug/mL are weighed using approximately 10 mg of each compound and diluted to 10 mL with acetonitrile. Each SPSS solution is diluted to prepare 2 different combined intermediate stock solutions, one for the low and high spikes and one for extended range spikes (CISS-CS and CISS-ERS) as presented in Table IX-1. Table IX-1 also shows the current control analytes for method UW14 which are: RDX, NB, 135TNB, 246TNT, and 24DNT. The proposed additional control analytes for this method are 2A-46DT and 2NT.

### B. CONTROL CHARTS.

Control charts are prepared for the control analytes spiked for this method. Percent recovery data from both the duplicate high level spikes, the low level and extended range spikes are calculated according to the following equation:

$$\text{Percent Recovery} = \frac{\text{Found Concentration}}{\text{Spiked Concentration}} \times 100$$

Normally unspiked values (method blank) have no response and are not required to be subtracted from the found concentration of the spikes prior to calculation of percent recovery. However, some compounds (HMX and RDX) have responses in the method blank. Since the calibration curves are quadratic, the proper correction is by concentration and not by instrument response.

Preparation of control charts requires the following data:

1. Average percent recovery (X) of the two high concentration spiked QC samples in each lot,
2. Difference (R) between the two high concentration spiked QC samples in each lot,
3. Three-point moving average (X) percent recovery control chart for the low level and extended range spike in each lot, and
4. Three-point moving average difference (R) control chart for the low level and extended range spike.

For values that fall outside the control limits and data points that are deemed as outliers, the data will be evaluated and corrective action will be taken. Table IX-2 presents the initial control limits for low spikes, high spikes, and extended range spikes.

## X. REFERENCES

A. U.S. Army Enviromental Center, 1987, USAEC QA Program (December 1985, 2nd Ed., March 1987).

## XI. ATTACHMENTS

**A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION**

**B. INITIAL/PRECERTIFICATION CALIBRATION - see ATTACHMENT 1.**

Response of each target analyte is tabulated at each calibration target concentration. The results from the lack of fit (LOF) and zero intercept (ZI) tests are presented.

**C. DAILY CALIBRATION DURING CERTIFICATION - see ATTACHMENT 2.**

Calibration responses and required percentage on the end run standard. Chromatograms for 2X and 200X four day certification spikes.

**D. STANDARD CERTIFICATION DATA - see ATTACHMENT 3.**

Table IV-1 Preparation of Calibration Stock Solutions and Initial Daily Calibration Solutions

| Separate Primary Stock Solutions (SPSS) | | | | Combined Intermediate Stock Solution (CISS) | | |
|---|---|---|---|---|---|---|
| Compound | SARM Wt. (mg) | Final Vol. (mL) | Conc. (µg/mL) | SPSS (mL) | Final Vol. (mL) | Conc. (µg/mL) |
| HMX | 85 | 25 | 3400 | 1.0 | 25 | 136.0 |
| RDX | 85 | 25 | 3400 | 1.0 | 25 | 136.0 |
| 135TNB | 36 | 25 | 1440 | 1.0 | 25 | 57.6 |
| 13DNB | 46 | 25 | 1840 | 1.0 | 25 | 73.6 |
| TETRYL | 56 | 25 | 2240 | 0.5 | 10 | 112.0 |
| NB | 52 | 25 | 2080 | 1.0 | 25 | 83.2 |
| 34DNT | 65 | 25 | 2600 | 1.0 | 25 | 104.0 |
| 246TNT | 52 | 25 | 2080 | 1.0 | 25 | 83.2 |
| 2A26DT | 27 | 25 | 1080 | 0.5 | 25 | 21.6 |
| 2A46DT | 27 | 25 | 1080 | 0.5 | 25 | 21.6 |
| 26DNT | 12.5 | 25 | 500 | 0.5 | 25 | 10.0 |
| 24DNT | 11.5 | 25 | 460 | 0.5 | 25 | 9.2 |
| 2NT | 33 | 25 | 1320 | 1.0 | 25 | 52.8 |
| 4NT | 80 | 25 | 3200 | 1.0 | 25 | 128.0 |
| 3NT | 80 | 25 | 3200 | 1.0 | 25 | 128.0 |

Stock ER–Combined Stock Solution for ER

| Compound | SPSS (mL) | Final Vol. (mL) | Conc. (mg/mL) |
|---|---|---|---|
| NB | 0.7 | 10 | 145.6 |
| 246TNT | 0.6 | 10 | 124.8 |
| 26DNT | 0.5 | 10 | 25.0 |
| 24DNT | 0.5 | 10 | 23.0 |
| 2NT | 0.9 | 10 | 118.8 |

Note: All standards are made to volume with 100% ACN

Tetryl intermediate stock is made to 10 mL due to possible degradation when combined with the other analytes

The extended range (ER) stock solution is required for only those analytes requiring a broader range than the E5 and B levels can produce

The surrogate 34DNT is included in the combines stock solution

Table IV-2 Initial Calibration Soultion Concentrations

| Designation | E5 | D | D2 | D5 | C | C2 | B | ER |
|---|---|---|---|---|---|---|---|---|
| Vol. (mL) | 0.5 | 1.0 | 0.2 | 0.5 | 0.1 | 0.2 | 1.0 | 1.0 |
| Stock | C | C | B | B | A | A | A | ER |
| Compound | | | | | | | | |
| HMX | 68 | 136.0 | 272.0 | 680.0 | 1360 | 2720 | 13600 | |
| RDX | 68 | 136.0 | 272.0 | 680.0 | 1360 | 2720 | 13600 | |
| 135TNB | 28.8 | 57.6 | 115.2 | 288.0 | 576 | 1152 | 5760 | |
| 13DNB | 36.8 | 73.6 | 147.2 | 368.0 | 736 | 1472 | 7360 | |
| TETRYL | 56.0 | 112.0 | 224.0 | 560.0 | 1120 | 2240 | 11200 | |
| NB | 41.6 | 83.2 | 166.4 | 416.0 | 832 | 1664 | 8320 | 14560 |
| 34DNT | 52.0 | 104.0 | 208.0 | 520.0 | 1040 | 2080 | 10400 | |
| 246TNT | 41.6 | 83.2 | 166.4 | 416.0 | 832 | 1664 | 8320 | 12480 |
| 2A26DT | 10.8 | 21.6 | 43.2 | 108.0 | 216 | 432 | 2160 | |
| 2A46DT | 10.8 | 21.6 | 43.2 | 108.0 | 216 | 432 | 2160 | |
| 26DNT | 5.0 | 10.0 | 20.0 | 50.0 | 100 | 200 | 1000 | 2500 |
| 24DNT | 4.6 | 9.2 | 18.4 | 46.0 | 92 | 184 | 920 | 2300 |
| 2NT | 26.4 | 52.8 | 105.6 | 264.0 | 528 | 1056 | 5280 | 11800 |
| 4NT | 64.0 | 128.0 | 256.0 | 640.0 | 1280 | 2560 | 12800 | |
| 3NT | 64.0 | 128.0 | 256.0 | 640.0 | 1280 | 2560 | 12800 | |

Note: All standard solutions are prepared in 30% ACN/70% water to a final volume of 10 mL.

Table IX-1 Preparation of Daily Control Spike Stocks, Working Spike Solutions, and Daily Control Spikes

| Compound | SPSS (µg/mL) | CISS-CS | | LSPK (µg/L) | HSPK (µg/L) |
|---|---|---|---|---|---|
| | | Amount SPSS to 50 mL (mL) | CISS-CS Conc (µg/mL) | 0.1 mL CISS-CS to 500 mL | 1.0 mL CISS-CS to 500 mL |
| RDX | 2925 | 0.2 | 11.70 | 2.34 | 23.40 |
| 135TNB | 1125 | 0.2 | 4.5 | 0.90 | 9.0 |
| NB | 1625 | 0.2 | 6.50 | 1.30 | 13.0 |
| 246TNT | 1587 | 0.2 | 6.35 | 1.27 | 12.70 |
| 2A46DT | 790 | 0.1 | 1.58 | 0.316 | 3.16 |
| 24DNT | 317.5 | 0.1 | 0.635 | 0.127 | 1.27 |
| 2NT | 1015 | 0.2 | 4.06 | 0.812 | 8.12 |

| Compound | CISS-ER | | ER SPIKE (µg/L) |
|---|---|---|---|
| | Amount SPSS to 25 mL (mL) | CISS-ER Conc. (µg/mL) | ER Target if 0.5 mL CISS-ER to 500 mL |
| RDX | 0.8 | 93.6 | 93.6 |
| 135TNB | 1.0 | 45.0 | 45.0 |
| NB | 1.5 | 97.5 | 97.5 |
| 246TNT | 1.4 | 88.9 | 88.9 |
| 2A46DT | 0.5 | 15.8 | 15.8 |
| 24DNT | 1.3 | 16.5 | 16.5 |
| 2NT | 2.4 | 97.4 | 97.4 |

SPSS = Separate control spike stock
CISS-CS = Combined intermediate stock solution for control spike
LSPK = Low spike
HSPK = High spike
ER = Extended range

Table IX-2 Preparation of Surrogate Spike

| Compound | Separate Stock (μg/mL) | Surrogate Spike Solution | | Surrogate Spike |
|---|---|---|---|---|
| | | Amount of stock to 100 mL (mL) | SS conc. (μg/mL) | Target when 0.5mL S to 500 mL (μg/L) |
| 34DNT | 1250 | 0.5 | 6.25 | 6.25 |

SS= Surrogate spike

Table IX-2. Initial Control Limits for Daily Control Spikes (page 1 of 2).

METHOD:
COMPOUNDS: EXPLOSIVES
UNITS: UGL

3 PT. MOVING AVERAGE X - R

| CODE | CONC. | UCL $\bar{X}$ | UWL $\bar{X}$ | = X | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| 13DNB | 1.375 | 111.4 | 108.1 | 101.7 | 95.3 | 92.0 | 24.3 | 19.4 | 9.4 |
| 24DNT | 0.212 | 96.9 | 91.2 | 79.9 | 68.6 | 62.9 | 42.7 | 34.0 | 16.6 |
| 26DNT | 0.224 | 109.3 | 101.1 | 84.7 | 68.3 | 60.1 | 61.9 | 49.3 | 24.0 |
| HMX | 1.208 | 102.8 | 100.2 | 95.2 | 90.1 | 87.5 | 19.2 | 15.3 | 7.4 |
| NB | 1.29 | 96.9 | 92.7 | 84.3 | 75.8 | 71.6 | 31.9 | 25.4 | 12.4 |
| RDX | 1.168 | 95.2 | 92.9 | 88.4 | 83.9 | 81.7 | 17.0 | 13.5 | 6.6 |
| TETRYL | 4.3 | 88.8 | 86.6 | 82.1 | 77.6 | 75.4 | 16.9 | 13.4 | 6.6 |
| 135TNB | 1.48 | 78.3 | 76.5 | 72.8 | 69.2 | 67.4 | 13.8 | 11.0 | 5.3 |
| 246TNT | 1139.4 | 129.4 | 109.4 | 89.4 | 79.4 | 75.4 | 60.1 | 29.3 | |
| 4A26DNT | 2148.4 | 131.6 | 113.8 | 96.1 | 87.2 | 66.9 | 53.3 | 26.0 | |
| 2A46DNT | 0135.0 | 116.9 | 100.9 | 84.8 | 76.8 | 60.6 | 48.3 | 23.5 | |
| 2NT | 1.2228.9 | 102.8 | 90.5 | 78.2 | 72.0 | 46.5 | 37.0 | 18.0 | |
| 3NT | 2.9210.8 | 102.6 | 86.3 | 70.1 | 61.9 | 61.5 | 49.0 | 23.9 | |
| 4NT | 3.0104.5 | 98.7 | 87.2 | 75.7 | 69.9 | 43.5 | 34.6 | 16.9 | |

SINGLE DAY X - R

| CODE | UCL CONC. | UWL $\bar{X}$ | = X | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL X | UWL R | $\bar{R}$ | R |
|---|---|---|---|---|---|---|---|---|---|
| 13DNB | 13108.5 | 103.6 | 101.8 | 100.0 | 99.0 | 4.7 | 3.6 | 1.5 | |
| 24DNT | 2.1108.5 | 103.1 | 92.2 | 81.4 | 76.0 | 28.3 | 21.7 | 8.6 | |
| 26DNT | 2.4114.6 | 107.8 | 94.2 | 80.6 | 73.8 | 35.4 | 27.2 | 10.9 | |
| HMX | 12108.6 | 107.7 | 99.8 | 91.9 | 88.0 | 20.6 | 15.8 | 6.3 | |
| NB | 12193.6 | 99.0 | 89.8 | 80.6 | 76.0 | 24.0 | 18.5 | 7.3 | |
| RDX | 11169.9 | 107.0 | 101.4 | 95.8 | 92.9 | 14.7 | 11.3 | 4.5 | |
| TETRYL | 43.098.8 | 96.9 | 93.3 | 89.7 | 87.8 | 9.5 | 7.3 | 2.9 | |
| 135TNB | 1481.1 | 100.0 | 97.7 | 95.4 | 94.3 | 5.9 | 4.5 | 1.8 | |
| 246TNT | 11104.0 | 99.5 | 90.7 | 81.8 | 77.3 | 23.2 | 17.8 | 7.1 | |
| 4A26DNT | 20.8 | 120.5 | 118.0 | 113.0 | 108.0 | 105.5 | 13.1 | 10.0 | 4.0 |
| 2A46DNT | 5.50 | 111.7 | 107.5 | 99.2 | 90.8 | 86.6 | 21.9 | 16.8 | 6.7 |
| 2NT | 12105.6 | 102.3 | 95.7 | 89.0 | 85.7 | 17.3 | 13.3 | 5.3 | |
| 3NT | 29102.7 | 99.4 | 92.8 | 86.2 | 83.0 | 17.2 | 13.2 | 5.3 | |
| 4NT | 30100.7 | 97.2 | 90.1 | 83.0 | 79.5 | 18.5 | 14.2 | 5.7 | |

Table IX-2. Initial Control Limits for Daily Control Spikes (page 2 of 2).

METHOD:
COMPOUNDS: EXPLOSIVES
UNITS: UGL

3 PT. MOVING AVERAGE X - R

| CODE | UCL CONC. | UWL $\bar{X}$ | = $\bar{X}$ | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL X | UWL R | $\bar{R}$ | R |
|---|---|---|---|---|---|---|---|---|---|
| 13DNB | 5598.6 | 97.1 | 94.3 | 91.4 | 90.0 | 10.8 | 8.6 | 4.2 | |
| 24DNT | 2103.3 | 99.9 | 93.2 | 86.4 | 83.0 | 25.5 | 20.3 | 9.9 | |
| 26DNT | 2408.9 | 105.6 | 98.9 | 92.2 | 88.9 | 25.2 | 20.1 | 9.8 | |
| HMX | 120.8 | 105.0 | 103.7 | 101.1 | 98.4 | 97.1 | 9.9 | 7.9 | 3.8 |
| NB | 1298.0 | 95.5 | 90.6 | 85.6 | 83.1 | 18.7 | 14.9 | 7.3 | |
| RDX | 116.8 | 103.1 | 100.5 | 95.5 | 90.5 | 88.0 | 18.9 | 15.1 | 7.3 |
| TETRYL | | | | | | | | | |
| 135TNB | 5902.1 | 100.5 | 99.4 | 98.2 | 97.7 | 4.2 | 3.4 | 1.6 | |
| 246TNT | 1128.6 | 96.5 | 92.4 | 88.3 | 86.3 | 15.4 | 12.3 | 6.0 | |
| 4A26DNT | | | | | | | | | |
| 2A46DNT | 2207.6 | 104.3 | 97.7 | 91.0 | 87.7 | 19.9 | 9.7 | 4.8 | |
| 2NT | 122.6 | 101.4 | 99.0 | 94.0 | 89.1 | 86.6 | 18.7 | 14.9 | 7.3 |
| 3NT | 116.8 | 102.9 | 99.7 | 93.5 | 87.2 | 84.1 | 23.7 | 18.9 | 9.2 |
| 4NT | 120.4 | 100.3 | 97.3 | 91.3 | 85.2 | 82.2 | 22.8 | 18.1 | 8.9 |

Contract No. DAAA15-87-D-0015
Delivery Order 001 (0002AW)

# DETERMINATION OF THIODIGLYCOL AND THIODIGLYCOLIC ACID IN WATER BY GAS CHROMATOGRAPHY

January, 1989

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Prepared for:

U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
Aberdeen Proving Ground, MD. 21010-5401

# USATHAMA METHOD NUMBER: UW22
# ANALYSIS OF THIODIGLYCOL AND THIODIGLYCOLIC ACID IN ENVIRONMENTAL WATER SAMPLES

## I. SUMMARY

### A. ANALYTES

This method is applicable to the quantitative determination of thiodiglycol and thiodiglycolic acid in environmental water samples.

### B. MATRIX

This method is applicable to all environmental water matrices.

### C. GENERAL METHOD

A measured volume of the sample is concentrated by boiling. The concentrated sample is passed through an Amberlight XAD-7 resin column and further concentrated by another boiling step. The extract is buffered and brought to volume with water. Liquid chromatographic conditions described in the method permit the separation and measurement of the two analytes in the extract. Analyte identification is performed using retention times, and quantitative analysis is performed using a standard curve of area counts.

## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The tested concentration ranges in "standard water" samples are:

| Analyte | Tested Concentration Range (ug/L)* |
|---|---|
| Thiodiglycol | 48.8 to 4880 |
| Thiodiglycolic acid | 44.6 to 1780 |

*ug/L = micrograms per Liter.

B. SENSITIVITY

The normalized response (integrator counts corrected for attenuation) at the "standard water" reporting limits are:

| Analyte | Area Counts |
|---|---|
| Thiodiglycol | 520,000 |
| Thiodiglycolic acid | 340,000 |

C. REPORTING LIMIT

The certified reporting limits in standard water as calculated according to the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) certified reporting limit program are:

| Analyte | Reporting Limit (ug/L) | Upper Certified Range (ug/L) |
|---|---|---|
| Thiodiglycol | 65.9 | 4,880 |
| Thiodiglycolic acid | 52.7 | 1,800 |

D. INTERFERENCES

1. Interferences in methods employing ultraviolet (UV) detection at short wavelengths can pose problems in analysis of this method. The interference can usually be minimized by preventing contact of reagents, glassware, apparatus, and samples with any plastic materials.
2. Solvents, reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be demonstrated to be free from interferences that have

retention times equal to those of the compounds of interest.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze eight samples in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

F. SAFETY INFORMATION

This method involves the use of acids. Adequate dermal and eye protection should be used, and proper ventilation available.

## III. APPARATUS AND CHEMICALS

A. GLASSWARE/HARDWARE

1. Gelman Acrodisc CR filter assemblies, 0.45 um;
2. 1,000 mL beaker;
3. 10 cm diameter watch glasses;
4. 50 mL beaker;
5. 10 mL graduated centrifuge tubes;
6. Glass transfer funnels;
7. Cleanup columns (20 cm x 1 cm);
8. 5 mL Gastight syringe.

B. INSTRUMENTATION AND OPERATING PARAMETERS

1. Altex Model 322 gradient high-pressure liquid chromatograph (HPLC) equipped with a Perkin Elmer LC-75 variable wavelength UV visible detector, a Waters Wisp 712 autosampler and interfaced to a Shimadzu C-R3A computing integrator.
2. Detector: Perkin Elmer LC-75 variable wavelength detector [ = 215 nanometers (nm)].
3. Column: Ultrasphere octadecylsilane (ODS) [4.6-millimeter (mm) inside diameter (ID) by 25 centimeters (cm)].

4. Particle Size: 5 um.
5. Guard column: 2.6 mm ID by 5 cm packed with Whatman Co: Pell ODS.
6. Silica precolumn: 2.6 mm ID by 5 cm packed with 60-230 mesh silica gel (ICN Pharmaceuticals, Inc.).
7. Flow rate and mobile phase: 1 millliliter per minute (mL/min) of 0.06 molar (M) phosphate buffer [7.94 grams (g) of sodium dihydrogen phosphate hydrate ($NaH_2PO_4 \cdot H_2O$) and 0.16 mL of 85-percent phosphoric acid ($H_3PO_4$) dissolved in 1 liter (L) of water].
8. Temperature: Room temperature.
9. Injection volume: 250 microliters (uL) fixed loop.
10. Retention times: Thiodiglycol -- 9.9 minutes (min) $\pm$ 0.4 min; thiodiglycolic acid -- 5.5 min $\pm$ 0.4 min. Retention time windows represent 3 standard deviations of a control spike measured during the 4 days of certification.

## C. ANALYTES

| Analyte | USATHAMA ACRONYM | CHEMICAL ABSTRACT Service (CAS) Registry Number |
|---|---|---|
| Thiodiglycol | TDGCL | 111-48-8 |
| Thiodiglycolic acid | TDGCLA | 123-93-3 |

## D. REAGENTS

1. HPLC-grade methanol (Burdick and Jackson);
2. 6 normal (N) sodium hydroxide;
3. HPLC water (Burdick and Jackson);
4. 6N sulfuric acid;
5. Concentrated buffer solution -- 79.4 g of $NaH_2PO_4 \cdot H_2O$ and 1.6 mL of 85-percent $H_3PO_4$ and HPLC water;
6. Amberlite XAD-7 resin -- The resin is prepared by shaking 50 g of resin with 100 mL of methanol for 15 min. on a wrist action shaker. The methanol is decanted, and the operation is repeated sequentially with three 100 mL

portions of methanol followed by four 100 mL portions of HPLC -grade water.

7. 2,2'-Thiodiethanol (Thiodiglycol) (Aldrich), 99+ percent purity.
8. Thiodiglycolic acid (Aldrich), 98 % purity.

## IV. CALIBRATION

### A. INITIAL CALIBRATION

1. Preparation of Standards

A Combined primary stock calibration standards are prepared by weighing approximately 0.5 grams (g) of thiodiglycol and 0.45 g of thiodiglycolic acid into a 50 mL volumetric flask; then dilute to volume with HPLC water. The concentrations of each compound in the primary stock calibration standard prepared this way is 10,000 ug/mL and 9,000 ug/mL, respectively.

A composite secondary stock calibration standard is prepared by adding 5.0 mL of the primary stock calibration standard to volume with HPLC water in a 50 mL volumetric flask. The concentrations in the composite secondary stock calibration standard for each analyte are 1,000 and 900 ug/mL, respectively.

Prepare calibration standards as shown in Table IV-1.

2. Instrument Calibration

To calibrate the instrument, at least 250 uL of each standard in Table IV-1 is injected into the instrument in the same manner as a sample extract. Duplicate composite calibration standards are analyzed during precertification calibration, and the single dilutions of the composite standards are analyzed during initial calibration. Currently an independent reference standard is not available for thiodiglycol and thiodiglycolic acid. Meanwhile an independent stock will be prepared to serve as

Table IV-1. Preparation of Initial Instrument Calibration Standards

| Calibration Standard | Volume Composite 2° Stock Standard Used | Final Volume (mL) | Concentration of Each Analyte in Instrument Calibration Standard (ug/L) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Thiodiglycolic Acid |
| A | 5.0 | 10 | 500 | 450 |
| B | 2.0 | 10 | 200 | 180 |
| C | 1.0 | 10 | 100 | 90 |
| D | 0.5 | 10 | 50 | 45 |
| E | 0.2 | 10 | 20 | 18 |
| F | 0.1 | 10 | 10 | 9.0 |
| G | 0.05 | 10 | 50 | 4.5 |
| H | 0 | 10 | 0 | 0 |

Source: Hunter/ESE, 1989.

a reference standard. The reference must be analyzed along with the initial and precertification calibration standards, and the results must be within ± 25% of the true value for the calibration to be considered valid. If the analysis of the reference standard fails, the source of the problem must be identified and corrected. The initial calibration and analysis of the independent reference standard must be repeated. The result of the second analysis of the independent reference standard must be within the acceptable limits, as specified by the source of the standard, before the analysis of samples may proceed. Since initial calibrations are performed daily, a reference is required at least weekly.

3. Analysis of Calibration Data

After analyzing the standards (i.e., one blank and seven standards) in duplicate, the data are tabulated and graphed. Data are analyzed using the lack of fit (LOF) and zero intercept (ZI) tests (USATHAMA QA Plan, 2nd Edition, March 1987). All pre-certification calibration data passed the LOF-ZI tests, therefore calibrations are linear.

4. Calibration Checks

At the end of the daily instrumental analysis, the highest working calibration standard is injected into the instrument. The response or the recovery of this end-of-day analysis should be ± 25% of the response or recovery obtained from the analysis of the same working calibration standard curve analyzed that day. If the first calibration check does not pass this criterion, then the standard may be reinjected and compared to the initial response. The post run standard data is documented and the percent drift is calculated on the chromatographic logsheet. An explanation must be provided if the calibration check is not acceptable for the data to be reported, otherwise the samples are rerun. An independent

reference sample is analyzed and must be within ± 25% of the true value. After seven runs, ± 2 standard deviations of the percent recovery will be evaluated as a criteria.

B. DAILY CALIBRATION

For daily calibration an initial calibration curve and QC checks will be performed as stated in Section IV.A.

V. CERTIFICATION TESTING

A. PREPARATION OF CONTROL SPIKES FOR METHOD CERTIFICATION

1. A combined primary certification spiking solution was prepared by weighing 0.407 g of thiodiglycol and 0.223 g of thiodiglycolic acid into a 100-mL volumetric flask and diluting to volume with HPLC water. The concentration of the primary certification spiking solution was 4,070 ug/mL and 2,230 ug/mL, respectively.
2. A secondary certification spiking solution was prepared by pipetting 3.0 mL of the primary certification spiking solution to a 25 mL volumetric flask and diluting to volume with HPLC water. The concentration of the secondary certification spiking solution was 488 and 446 ug/mL, respectively.
3. Certification spikes were prepared as shown in Table V-1 and were used to determine the accuracy, range, and reporting limits of the analytes to which this method applies. In each case, 500 mL of standard water was spiked with the appropriate certification spiking solution.

B. ANALYSIS OF CERTIFICATION SPIKES

Certification Control Spikes are analyzed by the procedures outlined in Section VII. Instrumentation is calibrated as in Sec. IV.

Table V-1. Preparation of Certification Control Spikes

| Certification Control Spike Sample Prepared | Control Stock Used | Volume Spiked* (mL) | Concentration of Each Analyte in the Prepared Sample (ug/L) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Thiodiglycolic Acid |
| Blk | ---- | 0 | 0 | 0 |
| 1S | 2° | 0.05 | 45.8 | 44.6 |
| 2S | 2° | 0.10 | 97.7 | 89.2 |
| 3S | 2° | 0.20 | 195 | 178 |
| 4S | 2° | 0.50 | 448 | 446 |
| 5S | 1° | 0.10 | 977 | 892 |
| 6S | 1° | 0.20 | 1950 | 1780 |
| 7S | 1° | 0.50 | 4880 | 4460 |

* to 500 mL of HPLC water

Source: Hunter/ESE 1989

## VI. SAMPLE HANDLING AND STORAGE

### A. SAMPLING PROCEDURE

There are no special considerations required due to the nature of organosulfur compounds. The samples need to be chilled to 4 deg. C immediately following sampling.

### B. CONTAINERS

Sampling containers used are 1.2 Litre glass amber jars with a teflon-lined cap.

### C. STORAGE CONDITIONS

Samples are to be kept on ice in coolers until shipped to the analytical lab. Upon arrival, the samples are stored at 4 deg. C in a walk-in refrigerator.

### D. HOLDING TIME LIMITS

The holding time limit is seven days from the time of sampling to extraction of the sample and 40 days from extraction to analysis.

### E. SOLUTION VERIFICATION

Calibration standards are verified with daily control spikes and reference samples. When fresh control spiking solutions are prepared. They must be verified to determine that:

1. the previous spiking solution had or had not deteriorated
2. the new solution was correctly prepared

Therefore, the secondary control spike solutions (Sec. IX) will be checked against working standards before initial use and again within seven days before subsequent use.

## VII. PROCEDURE

Environmental samples and daily quality control spikes (see Sec. IX) are prepared for analysis and analyzed as follows:

A. EXTRACTIONS

1. Measure 500 mL of the water sample into 1 L beaker.
2. Add a boiling chip (Teflon) and concentrate the sample by boiling the water on a hot plate to a volume of 50 mL. The boildown time should be as rapid as possible and should not exceed 2.5 hours.
3. Check the pH of the sample with pH indicator paper. Adjust to pH 7 ± 1 with sulfuric acid or sodium hydroxide solution if necessary.
4. Transfer the sample to a column (20 cm x 1 cm) packed with Amberlite XAD-7 resin. Allow the sample to pass through the column at a rate of 1 mL/min. and collect the effluent in the same beaker. Rinse the column with 50 mL of HPLC water into the same beaker to give a total volume of approximately 100 mL.
5. Reduce the volume of the solution to less than 25 mL by boiling, and then quantitatively transfer to a 50 mL beaker.
6. Reduce the solution volume to less than 5 mL by boiling on a hot plate.
7. Transfer the solution into a 10 mL graduated centrifuge tube, rinsing quantitatively with HPLC-grade water. Add 0.5 mL concentrated buffer solution to the extract. Dilute to the 5 mL mark with HPLC water.
8. Filter the sample through a 0.45 um filter and transfer to a 5 mL, amber, septum-sealed vial for storage at 4° C. The solution is now ready for analysis by HPLC.

B. CHEMICAL REACTIONS

There is no chemical reaction stage.

C. INSTRUMENT ANALYSIS

Daily calibration performed as described in Sec. IV.A. and B. with instrument condition described in Sec. III.B.

## VIII. CALCULATIONS

Determine the concentration of each analyte according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(Vt)}{(Vs)}$$

where: A = Concentration (ug/mL) of each analyte found in the extract by comparison with the appropriate standard curve (ug/mL),

Vt = Final volume of extract solution (5 mL), and

Vs = Volume of initial sample extracted (0.5 L).

The method is corrected for accuracy obtained during method certification to obtain final results in the USATHAMA data management system.

## IX. DAILY QUALITY CONTROL

### A. CONTROL SAMPLES

1. A Primary Control Spike Solution (PCSS): The PCSS is prepared by weighing 0.6 g of thiodiglycol and 0.5 g of thiodiglycolic acid into a 100 mL volumetric flask and diluting to volume with HPLC water. The concentrations of the PCSS are 6,000 and 5,000 ug/mL respectively. (prepare fresh monthly)
2. Secondary Control Spike Solution (SCSS): The secondary control spike solution is made by diluting 5 mL of the PCSS (1) with HPLC water into a 50 mL volumetric flask. The concentrations of this solution are 600 and 500 ug/mL. (prepare fresh daily)
3. Using the SCSS, the Daily Control Spikes are made by spiking the appropriate volume of stock solutions to 500 mL of standard water as shown in Table IX - 1.

Table IX-1. Preparation of Daily Control Spikes

| Daily Control Spike | Control Stock Used | Volume Spiked* (mL) | Concentration of Each Analyte in the Prepared Sample (ug/L) | |
|---|---|---|---|---|
| | | | Thiodiglycol | Thiodiglycolic Acid |
| Blank | None | 0 | 0 | 0 |
| Low | SCSS | 0.1 | 120 | 100 |
| High | SCSS | 1.5 | 1800 | 1500 |
| High | SCSS | 1.5 | 1800 | 1500 |

* to 500 mL of standard water

Source: Hunter/ESE, 1989.

B. CONTROL CHARTS

In the initial construction of the control charts, data from the laboratory certification analysis will be used. Data from spiked QC samples, within a lot, will be compared to control chart limits to demonstrate that analysis of the lot are under control and will be used to update the charts. X-R control charts will be used in the Quality Assurance (QA) Program. Control charts are prepared for each target analyte using percent recovery (found concentration - method blank/ spiked concentration x 100) data from the duplicate spiked QC samples variations in spiking solution concentrations.

To prepare control charts, the analyst should have access to the following data:

1. Average (x) percent recovery for the two high concentration spiked QC samples in each lot,
2. Difference (R) between the percent recoveries for the two high concentration spiked QC samples in each lot,
3. Three-point moving average (x) spkie recovery of the low-concentration spike QC sample, and
4. Three-point moving difference (R) between the percent recoveries for the low-concentration spike QC sample.

The initial control chart shall be prepared using the four pairs of certification data closest to the spiking concentration used during analysis. The average (x), average range (R), and control limits for x and R shall be updates after each lot for the first 20 lots. Limits established after Lot 20 will be used for the next 20 lots. Control charts will be updated after each 20 lots thereafter using the most recent 40 lots of QC data. In updating the control charts, the new data must be combined with the individual values of previous percent recoveries and not the mean of all previous data. Initial control chart limits are presented in Table IX-2.

Table IX-2. Initial Warning and Control Limits

METHOD:
COMPOUNDS: Thiodiglycol and Thiodiglycolic acid in Water
UNITS: UGL

3 PT. MOVING AVERAGE $\bar{X}$ - R

| CODE | CONC. | UCL $\bar{X}$ | UWL $\bar{X}$ | = $\bar{X}$ | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| TDGCL | 100 | 89.6 | 80.9 | 63.3 | 46.2 | 37.6 | 65.4 | 52.1 | 25.4 |
| TDGCLA | 100 | 104.1 | 98.3 | 86.7 | 75.2 | 69.4 | 43.6 | 34.7 | 17.0 |

SINGLE DAY $\bar{X}$ - R

| CODE | CONC. | UCL $\bar{X}$ | UWL $\bar{X}$ | = $\bar{X}$ | LWL $\bar{X}$ | LCL $\bar{X}$ | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| TDGCL | 2000 | 73.8 | 72.2 | 69.0 | 65.8 | 64.2 | 8.3 | 6.4 | 2.5 |
| TDGCLA | 1800 | 95.1 | 94.4 | 93.0 | 91.6 | 90.9 | 3.6 | 2.8 | 1.1 |

## X. REFERENCES

U.S. Army Toxic and Hazardous Materials Agency, (USATHAMA). 1987. QA Plan (December 1985, 2nd Edition, March 1987).

## XI. DATA

### A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION

These compounds cannot be seen on GC/MS and GC/FID. Therefore, standards can only be verified by independently prepared references.

### B. PRE-CERTIFICATION CALIBRATION

Attachment 1

### C. DAILY CALIBRATION AND CHROMATOGRAM

Attachment 2

### D. CERTIFICATION DATA

Attachment 3

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ESE, 1994

Table V. Preparation of Certification Control Spike Samples

| Control Spike Samples Prepared | Volume of Composite Primary Control Spiking Solution Used (mL) | Volume of Standard Water Used(mL) | Concentration of Control Spike Samples (ug/L) DMDS | OXAT | DITH | CPMS | BTZ | CPMSO2 | CPMSO |
|---|---|---|---|---|---|---|---|---|---|
| Blank | 0 | 800 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 0.08 | 800 | 1.11 | 0.99* | 1.11 | 1.26 | 2.11 | 1.06* | 1.13* |
| 3 | 0.16 | 800 | 2.28 | 1.98 | 2.22 | 2.53 | 4.22 | 2.12* | 2.26* |
| 4 | 0.32 | 800 | 4.56 | 3.95 | 4.44 | 5.06 | 10.6 | 4.23 | 4.22 |
| 5 | 0.80 | 800 | 11.4 | 9.88 | 11.1 | 12.6 | 21.1 | 10.6 | 10.6 |
| 6 | 1.6 | 800 | 22.8 | 19.8 | 22.2 | 25.3 | 42.2 | 21.2 | 21.2 |
| 7 | 3.2 | 800 | NA+ | 39.5 | NA | NA | NA | 42.3 | 42.2 |
| 8 | 8.0 | 800 | NA | NA | NA | NA | NA | 106 | 106 |

*Not detected during certification and, consequently, data are not presented in Att. 1.
NA = not analyzed. Spikes were made at higher concentrations to supplement the non-detected data (see * above) for some analytes; only selected higher concentration spikes were analyzed since only selected data needed to be supplemented.

Source: ESE, 1988.



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Table IX. Initial Control Limits for Organosulfur Compounds in Water

ESE CERTIFICATION LIMITS

METHOD: OS in Water
UNITS: ug/L

3 PT. MOVING AVERAGE X - $\bar{R}$

| CODE | CONC. | UCL_ X | UWL_ X | = X | LWL_ X | LCL_ X | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| DMDS | 3.42 | 87.5 | 85.2 | 80.5 | 75.8 | 73.5 | 17.6 | 14.0 | 6.8 |
| OXAT | 3.95 | 88.4 | 85.7 | 80.2 | 74.7 | 71.9 | 20.7 | 16.5 | 8.1 |
| DITH | 2.22 | 97.1 | 92.2 | 82.6 | 72.9 | 68.1 | 36.4 | 29.0 | 14.1 |
| CPMS | 2.53 | 101.3 | 95.5 | 83.9 | 72.3 | 66.5 | 43.8 | 34.8 | 17.0 |
| BTZ | 4.22 | 105.8 | 103.9 | 100.2 | 96.5 | 94.6 | 14.0 | 11.2 | 5.4 |
| CPMSO | 4.23 | 82.1 | 79.6 | 74.6 | 69.6 | 67.1 | 18.9 | 15.1 | 7.3 |
| CPMSO2 | 9.04 | 85.6 | 83.8 | 80.1 | 76.4 | 74.6 | 13.9 | 11.1 | 5.4 |

SINGLE DAY X - $\bar{R}$

| CODE | CONC. | UCL_ X | UWL_ X | = X | LWL_ X | LCL_ X | UCL R | UWL R | $\bar{R}$ |
|---|---|---|---|---|---|---|---|---|---|
| DMDS | 17.1 | 82.8 | 80.1 | 74.6 | 69.2 | 66.4 | 14.2 | 10.9 | 4.3 |
| OXAT | 19.8 | 82.1 | 80.8 | 78.3 | 75.8 | 74.5 | 6.5 | 5.0 | 2.0 |
| DITH | 11.1 | 83.8 | 81.3 | 76.3 | 71.2 | 68.7 | 13.1 | 10.0 | 4.0 |
| CPMS | 12.6 | 82.9 | 80.6 | 75.9 | 71.3 | 69.0 | 12.1 | 9.3 | 3.7 |
| BTZ | 42.2 | 99.1 | 97.5 | 94.4 | 91.3 | 89.7 | 8.2 | 6.3 | 2.5 |
| CPMSO | 21.2 | 85.9 | 83.6 | 79.0 | 74.5 | 72.2 | 11.9 | 9.2 | 3.7 |
| CPMSO2 | 45.2 | 98.6 | 95.4 | 89.1 | 82.7 | 79.5 | 16.7 | 12.8 | 5.1 |



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Table IX-1. Preparation of Daily Control Spike Samples

| Daily Control Spike Level | Volume of Primary Composite Control Spiking Solution* (mL) | Nominal Concentration of Daily Control Spike Samples (ug/g) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | DMDS | OXAT | DITH | CPMS | BTZ | CPMSO | CPMSO2 |
| Blank | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Low | 0.20 | 2.0 | 2.0 | 1.6 | 2.0 | 2.0 | 4.0 | 4.0 |
| High | 1.00 | 10.0 | 10.0 | 8.0 | 10.0 | 10.0 | 20.0 | 20.0 |
| High | 1.00 | 10.0 | 10.0 | 8.0 | 10.0 | 10.0 | 20.0 | 20.0 |

Source: ESE, 1988.


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Table IX-1. Preparation of Daily Control Spike Samples

| Daily Control Spike | Spike solution (mL)* | DMDS (ug/L) | OXAT (ug/L) | DITH (ug/L) | CPMS (ug/L) | BTZ (ug/L) | CPMSO (ug/L) | CPMSO2 (ug/L) |
|---|---|---|---|---|---|---|---|---|
| Blank | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Low Spike | 0.16 | 3.0 | 4.06 | 3.0 | 3.0 | 4.0 | 8.0 | 10.0 |
| High Spike 1 | 0.80 | 15.0 | 20.0 | 15.0 | 15.0 | 20.0 | 40.0 | 50.0 |
| High Spike 2 | 0.80 | 15.0 | 20.0 | 15.0 | 15.0 | 20.0 | 40.0 | 50.0 |

* Spiked into 800 mL of Standard water.

Source: ESE, 1988.



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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

# TITLE: DETERMINATION OF ORGANOSULFUR COMPOUNDS IN SOIL BY GAS CHROMATOGRAPHY (METHOD LL03)

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**TITLE: DETERMINATION OF ORGANOSULFUR COMPOUNDS IN SOIL BY GAS CHROMATOGRAPHY (METHOD LL03)**

## I. SUMMARY

### A. ANALYTES

This method is applicable to the Class 1 analysis of the following organosulfur compounds in environmental soil samples:

Dimethyldisulfide (DMDS)
1,4-Oxathiane (OXAT)
1,4-Dithiane (DITH)
p-Chlorophenylmethylsulfide (CPMS)
Benzothiazole (BTZ)
p-Chlorophenylmethylsulfoxide (CPMS0)
p-Chlorophenylmethylsulfone (CPMSO2)

### B. MATRIX

This method is applicable to all environmental soil and sediment matrices.

### C. GENERAL METHOD

Ten grams (g) of soil are dried with 10 g of anhydrous sodium sulfate and extracted with methylene chloride for 4 hours on a wrist-action shaker. The extract is analyzed by gas chromatography (GC) using flame-photometric detection (FPD) in the sulfur mode.


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## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The tested concentration ranges for the target analytes are as follows:

| Analyte | Concentration Range (ug/g) |
|---|---|
| DMDS | 0.692 to 13.8 |
| OXAT | 0.856 to 17.1 |
| DITH | 0.571 to 11.4 |
| CPMS | 1.08 to 21.6 |
| BTZ | 0.528 to 13.2 |
| CPMSO | 2.25 to 45.0 |
| CPMSO2 | 2.37 to 47.4 |

Note: ug/g = micrograms per gram.

### B. SENSITIVITY

The instrumental responses for each compound, reported in peak area units at the certified reporting limit (CRL), are as follows:

| Analyte | Certified Reporting Limit (ug/g) | Area Counts |
|---|---|---|
| DMDS | 0.692 | 8,800 |
| OXAT | 0.856 | 2,300 |
| DITH | 1.47 | 2,400 |
| CPMS | 1.08 | 2,271 |
| BTZ | 1.08 | 6,690 |
| CPMSO | 2.25 | 6,400 |
| CPMSO2 | 2.37 | 4,489 |


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## C. REPORTING LIMITS

The CRL and upper certified limits (UCLS) for each analyte in environmental soil samples are as follows:

| Analyte | CRL (ug/g) | UCL (ug/g) |
|---|---|---|
| DMDS | 0.692 | 13.8 |
| OXAT | 0.856 | 17.1 |
| DITH | 1.47 | 11.3 |
| CPMS | 1.08 | 21.6 |
| BTZ | 1.08 | 13.2 |
| CPMSO | 2.25 | 45.0 |
| CPMSO2 | 2.37 | 47.4 |

## D. INTERFERENCES

Solvents, reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be free from interferences that have retention times equal to the retention times of the compounds of interest. This method is subject to interference from coeluting species that respond to FPD. Carryover from analysis of a highly contaminated sample can result in apparent contamination of the succeeding samples analyzed. Such contamination is often manifested by the presence of unusually broad chromatographic peaks nested among narrower peaks. This interference is minimized by reanalyzing heavily contaminated samples following dilution, running blanks after heavily contaminated samples until carryover is removed, and/or baking off the column at the column temperature maximum until the contamination is removed.

## E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 sample extracts in an 8-hour day. One analyst can perform approximately 10 extractions in an 8-hour day.


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### F. SAFETY INFORMATION

The target compounds in this method are toxic. The preparation of all standards should be performed in a laboratory hood. Adequate dermal protection must be used when handling samples and standards.

## III. APPARATUS AND CHEMICALS

### A. GLASSWARE/HARDWARE

1. Amber bottles (60-milliliter (mL) with Teflon -lined screw caps).
2. Class A Volumetric flasks (5-, 10-, 25-, 50- and 100-mL).
3. Class A Volumetric pipettes (0.5 to 25 mL).
4. Microsyringes (250- and 1,000-mL).
5. Pasteur pipettes (disposable).
6. Micropipettes [50-, 100-, 200-, and 1,000-microliter (uL)].
7. Amber-glass vials (9-mL, with Teflon-lined crimp caps).
8. Glass vials (2-mL, with Teflon -lined crimp-seal caps for use with an automatic sampler).
9. Stainless-steel spatulas.
10. Analytical balance (Mettler AE160 or equivalent, with 0.0001-g sensitivity).
11. Stainless-steel spatulas.
12. Wrist-action shaker.

### B. INSTRUMENTATION

1. Gas chromatograph with an FPD (Varian 3400 or equivalent), equipped with an automatic sampler (Varian 8100 or equivalent) and a computer data system (PE Nalson 2700 Turbochroim or equivalent).

2. Chromatographic conditions:

| | | |
|---|---|---|
| a. | Column: | 5-percent SP-1000 on Chromosorb [6-foot (ft) by 2-millimeter (mm) inside diameter (ID) by 6 mm outside diameter (OD)]. |
| b. | Injector temperature: | 200 degrees Celsius (°C). |
| c. | Temperature Program: | 80°C, hold 3 minutes (min), heat at 32 degrees Celsius per minute (°C/min) to 240°C, hold 7 min. |


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| | | |
|---|---|---|
| d. | Detector temperature: | 285°C. |
| e. | Gas flow: | Helium at 30 milliliters per minute (mL/min), hydrogen at 140 mL/min, Air 1 at 80 mL/min, Air 2 at 170 mL/min. |
| f. | Injection volume: | 7 uL. |
| g. | Retention times: | A retention time window of ± 0.02 min. of the retention time of the high standard for each analyte will be used for compound identification. |

| Analyte | Retention Time (min) |
|---|---|
| DMDS | 1.47 ± 0.04 |
| OXAT | 4.40 ± 0.13 |
| DITH | 6.45 ± 0.19 |
| CPMS | 7.65 ± 0.23 |
| BTZ | 7.92 ± 0.24 |
| CPMSO | 10.5 ± 0.32 |
| CPMSO2 | 11.87 ± 0.36 |

## C. ANALYTES

| Analyte | Abstract Service (CAS Number) | Boiling Point (°C) |
|---|---|---|
| DMDS | 624-92-0 | --- |
| OXAT | 15890-15-1 | 147 |
| DITH | 505-29-3 | 200 |
| CPMS | 123-09-1 | --- |
| BTZ | 95-16-9 | 231 |
| CPMSO | 934-73-6 | --- |
| CPMSO2 | 98-57-7 | --- |


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## D. REAGENTS AND STANDARD ANALYTICAL REFERENCE MATERIALS (SARMs)

The standards used for the target compound certification and calibration are all USATHAMA standard analytical reference materials (SARMs) with the exception of BTZ, which was not available as a SARM at the time of this certification. Equivalent standards may be used as long as they have been characterized according to Section VI.E.3 of the USATHAMA Quality Assurance (QA) Plan (2nd Edition, March 1987). The BTZ standard was from Aldrich Chemical Company, Milwaukee, WI. A copy of the mass spectrum of this BTZ standard, the reference mass spectrum from the combined Wiley/National Bureau of Standards library of mass spectra, and a chromatogram of the standard analyzed by gas chromatography with flame ionization detector (GC/FID) are presented in Attachment 1. The SARM and Aldrich lot numbers for the target analytes are as follows:

| Analyte | SARM or Aldrich Lot Number |
|---|---|
| DMDS | SARM - 1378 |
| OXAT | SARM - 2119 |
| DITH | SARM - 2111 |
| CPMS | SARM - 1105 |
| BTZ | Aldrich - 91928DP |
| CPMSO | SARM - 3107 |
| CPMSO2 | SARM - 1106 |

Other chemicals and materials include: methylene chloride (pesticide grade); acetone (pesticide grade); sodium sulfate (400 C for 4 hours in muffle furnace); and, "Standard Soil" (certification spikes used an uncontaminated natural soil from Rocky Mountain Arsenal, rather than the USATHAMA Standard soil).


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# IV. CALIBRATION

## A. INITIAL CALIBRATION

### 1. Preparation of Standards

a. Individual primary stock calibration standards are prepared by weighing approximately 25 milligrams (mg) of each of the six target analytes into separate 25-mL volumetric flasks, then diluting to volume with methylene chloride. The nominal concentrations of each of the primary stock calibration standards prepared this way for each analyte is 1,000 ug/mL.

b. A composite primary stock calibration standard is prepared by adding 5 mL of each individual stock calibration standard to methylene chloride in a 50-mL volumetric flask and then diluting to volume with methylene chloride. The nominal concentrations in the composite primary stock calibration standard for each analyte is 100 ug/mL.

c. A composite secondary calibration stock is prepared by placing 5 mL of the composite primary calibration stock into a 50-mL volumetric flask and diluting to volume with methylene chloride (a 1:10 dilution of the composite primary calibration stock). The nominal concentration in the composite secondary calibration stock for each analyte is 10.0 ug/mL.

d. Composite working calibration standards are prepared using methylene chloride, the composite primary and secondary stock calibration standards and volumetric pipettes as shown in Table IV-1.

### 2. Instrument Calibration

To calibrate the instrument, 7 uL of each standard in Table IV-1 is injected into the instrument in the same manner as a sample extract. Duplicate composite calibration standards are analyzed during precertification calibration, and the single dilutions of the composite standards are analyzed during initial calibration.


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Currently an independent reference standard is not available for organosulfur compounds in water. Meanwhile an independent stock will be prepared to serve as a reference standard. The reference must be analyzed along with the initial and precertification calibration standards, and the results must be within ± 25% of the true value for the calibration to be considered valid. If the analysis of the reference standard fails, the source of the problem must be identified and corrected. The initial calibration and analysis of the independent reference standard must be repeated. The result of the second analysis of the independent reference standard must be within the acceptable limits, as specified by the source of the standard, before the analysis of samples may proceed. Since initial calibrations are performed daily, a reference is required at least weekly.

3. Analysis of Calibration Data

After analyzing the standards (i.e., one blank and nine standards) in duplicate, the data are tabulated and graphed. Data are analyzed using the lack of fit (LOF) and zero intercept (ZI) tests (USATHAMA QA Plan, 2nd Edition, March 1987). All pre-certification calibration data passed the LOF-ZI tests, therefore calibrations are linear. Not all data from the standards analyzed in Table IV-1 are used for the regressions (i.e. Standard A would be used for CPMSO and CPMSO2 only, while Standards G-J would not be used for CPMSO and CPMSO2).

4. Calibration Checks

At the end of the daily instrumental analysis, the highest working calibration standard is injected into the GC. The response of the recovery of this end-of-day analysis should be ± 25% of the response or recovery obtained from the analysis of the same working calibration standard curve analyzed that day. If the first calibration check does not pass this criterion, then the standard may be reinjected and compared to the initial response. The post run standard data is documented and the percent drift is calculated on the chromatographic logsheet. An explanation must be provided if the calibration check is not acceptable for the data to be reported, otherwise the samples are rerun.



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5. Retention Time Windows

Retention time windows for a method are established based on historical data of daily performance of the method or by using retention windows from a reference method. If a reference method with established windows is not available the windows are calculated by calculating the relative standard deviation of the retention variation throughout the run for all standards, control samples and continuing calibration standards and multiplying the result by three and rounded to 1 significant figure. The windows are expressed in absolute minutes where the retention variation is minimal throughout the chromatographic run and as a percentage of the retention time in methods whose variation is proportional to retention time. The retention window is applied to retention times from a specified standard during initial or daily calibration. These retention window values are entered into the method used for processing of the chromatographic data for computerized identification/rejection of detected peaks. The analyst can override the identification/rejection of a peak by providing documentation of his decision.

## B. DAILY CALIBRATION

For daily calibration an initial calibration curve and QC checks will be performed as stated in Section IV.A.

# V. CERTIFICATION TESTING

## A. PREPARATION OF CERTIFICATION SPIKES

1. Individual primary stock control spiking solutions are prepared by weighing approximately 50 mg of each of the analytes into separate 50-mL volumetric flasks, then diluting each to volume with acetone. The nominal concentrations in the individual primary stock spiking solutions were 1,000 ug/mL.

2. A composite primary stock control spiking solution is prepared (in a 50-mL volumetric flask) by diluting 5 mL of each of the individual primary stock control spiking solutions (2.5 mL for BTZ during certification only) with acetone to the final volume of 50 mL. The nominal concentrations in the composite primary stock control spiking solution prepared as above were 100 ug/mL (50 ug/mL for BTZ during certification only).


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3. The spiking procedure in Table V-1 was implemented to prepare control spike samples to determine the accuracy and reporting limits for each analyte. In each case, 10 g of "standard soil" in separate, 60-mL amber vials were spiked with the appropriate volume of the composite primary control spiking solution and allowed to dry for 1 hour prior to extraction. Actual concentrations during certification are presented in Table V-1.

### B. ANALYSIS OF CERTIFICATION SPIKES

Certification control spikes are analyzed by the procedures outlined is Section VII.

## VI. SAMPLE HANDLING STORAGE

### A. SAMPLING PROCEDURE

There are no special considerations required due to the nature of organosulfur compounds. Soil samples may be collected as grab samples or cores. The samples need to be chilled to 4 deg. C immediately following sampling.

### B. CONTAINERS

Sampling containers used are 1.2 Liter glass amber jars with a teflon-lined cap for grab samples, or polybutyrate tubes for core samples.

### C. STORAGE CONDITIONS

Samples are to be kept on ice in coolers until shipped to the analytical lab. Upon arrival, the samples are stored at 4 deg. C in a walk-in refrigerator.


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Table IV-1. Preparation of Initial Calibration Standards.

| Standard | Composite Standard Used | Vol. Used (mL) | Final Vol. (mL) | Nominal Concentration (ug/L) | Nominal Extract Concentration (ug/g) |
|---|---|---|---|---|---|
| Blank | - | 0 | 25 | 0.0 | 0.0 |
| A | 1 | 5.0 | 25 | 20,000 | 40.0 |
| B | 1 | 4.0 | 50 | 8,000 | 16.0 |
| C | 1 | 2.0 | 50 | 4,000 | 8.00 |
| D | 1 | 1.0 | 50 | 2,000 | 4.00 |
| E | 2 | 4.0 | 50 | 800 | 1.60 |
| F | 2 | 5.0 | 100 | 500 | 1.00 |
| G | 2 | 2.0 | 50 | 400 | 0.80 |
| H | 2 | 2.5 | 100 | 250 | 0.50 |
| I | 2 | 1.0 | 50 | 200 | 0.40 |
| J | 2 | 0.8 | 50 | 160 | 0.32 |

Source: ESE, 1988.

Table IV-2. Standards Used for Regression for Daily Calibration.

| Compound | Standards |
|---|---|
| DMDS | B through J |
| OXAT | B through J |
| DITH | B through J |
| CPMS | B through J |
| BTZ | B through J |
| CPMSO | A through F |
| CPMSO2 | A through F |

Source: ESE



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Table V-1: Preparation of Certification Control Spike Samples

| Control Spike Samples Prepared | Volume of Composite Primary Control Spiking Solution Used (mL) | Concentration of Control Spike Samples (ug/g) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | DMDS | OXAT | DITH | CPMS | BTZ | CPMSO2 | CPMSO |
| Blank | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 0.05 | 0.692 | 0.856 | 0.571 | 0.540 | 0.264 | 0.562* | 0.593* |
| 3 | 0.10 | 1.38 | 1.71 | 1.14 | 1.08 | 0.528 | 1.12* | 1.19* |
| 4 | 0.20 | 2.77 | 3.42 | 2.28 | 2.16 | 1.06 | 2.25 | 2.37 |
| 5 | 0.50 | 6.92 | 8.56 | 5.71 | 5.40 | 2.64 | 5.62 | 5.93 |
| 6 | 1.00 | 13.8 | 17.1 | 11.4 | 10.8 | 5.28 | 11.2 | 11.9 |
| 7 | 2.00 | NA+ | NA | NA | 21.6 | 10.6 | 22.5 | 23.7 |
| 8 | 4.00 | NA | NA | NA | NA | NA | 45.0 | 47.4 |

Spikes prepared with 10 g of Standard Soil.

* Not detected during certification and, consequently, data are not presented in Att. 1.

+NA = not analyzed. Spikes were made at higher concentrations to supplement the non-detected data (see * above) for some analytes; only selected higher concentration spikes were analyzed since only selected data needed to be supplemented.

Source: ESE, 1988.



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### D. HOLDING TIME LIMITS

The holding time limit is seven days from the time of sampling to extraction of the sample and 40 days from extraction to analysis.

### E. SOLUTION VERIFICATION

When fresh control spiking solutions are prepared. They must be verified to determine that:

1. The previous spiking solution had or had not deteriorated.

2. The new solution was correctly prepared.

Therefore, dilute working spike solutions will be checked against working standards before initial use and again within seven days before subsequent use.

## VII. PROCEDURE

Daily quality control spikes (see Section IX.A) and environmental soil samples are prepared for analysis and analyzed as follows:

### A. SEPARATIONS

1. Transfer 10 g of each soil sample into separate 60-mL amber vials and mix with 10 g of anhydrous sodium sulfate (Prepare daily control spikes as specified in Section IX.A). Environmental samples are to be extracted within 7 days of sample collection.

2. Add 20 mL of methylene chloride with a volumetric pipette, and cap tightly.

3. Shake the culture tube in a horizontal position for 4 hours on a wrist-action shaker.

4. Allow the particulate to settle or centrifuge, if necessary.

5. With a disposable pipette, transfer approximately 1 to 2 mL of the extract to an autosampler vial. Before sealing, add several granules of anhydrous sodium sulfate to remove moisture. The extract is now ready for instrumental analysis.



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6. Transfer the remainder of the extract to an 8-mL amber glass vial and cap tightly. Save as a backup sample.

7. Store samples at 4 degrees C until analysis. Samples must be analyzed within 40 days of sample extraction.

### B. CHEMICAL REACTIONS

No chemical reactions are required by this method.

### C. INSTRUMENTAL ANALYSIS

1. Perform daily instrument calibration as described in Section IV.B. Use the instrument conditions listed in Section III.B.

2. Place the sample extracts in the autosampler tray and inject 7 uL of each sample extract into the instrument under exactly the same conditions as those under which it was calibrated.

## VIII. CALCULATIONS

A linear calibration curve is constructed from the calibration data by plotting the response versus the concentration of each standard. The calibration curve slope and intercept are determined by linear regression. The concentration of a target compound in the extract is calculated by substituting the response into the calibration curve equation. The concentration of each analyte in the original soil sample is determined by the following formula:

$$\text{Concentration (ug/g)} = \frac{\text{Extract Conc. (ug/L)} \times 20\ \text{mL}}{V_w\ (\text{g}) \times 1000\ \text{mL/L}}$$

where: Extract Conc. = Calculated concentration in the extract in nanograms per milliliter (ng/mL)
20 mL = Final extract volume in milliliters;
Vw = Sample weight in grams (nominally 10.0 g).

Percent moisture and dilution factors are entered as separate parameters. The Installation Restoration Data Management System Computer calculates for dry-weight basis.


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# IX. DAILY QUALITY CONTROL

## A. CONTROL SAMPLES

The daily control spikes required are a Standard Matrix Method Blank, one low level Standard Matrix Spike at approximately twice the CRL, and two high-level Standard Matrix Spikes at five times the low level spike. Individual stock spiking solutions are prepared as discussed in Section. V.A.1.

A composite daily control spike solution is prepared (in a 50-mL volumetric flask) by diluting the following volumes of the individual primary stock control spiking solutions to volume with acetone:

| Analyte | mLs Stock | Concentration (ug/mL) |
|---|---|---|
| DMDS | 5.0 | 100 |
| OXAT | 5.0 | 100 |
| DITH | 4.0 | 80 |
| CPMS | 5.0 | 100 |
| BTZ | 5.0 | 100 |
| CPMSO | 10.0 | 200 |
| CPMSO2 | 10.0 | 200 |

The spike solution is verified when needed as described in Section VI.E. From the combined daily control spike solution, the daily control spikes are prepared as shown in Table IX-1 by adding the prescribed amount of stock to 10 g of standard soil. Allow the spike to dry on the soil for 1 hour before extraction.

## B. CONTROL CHARTS

In the initial construction of the control charts, data from the laboratory certification analysis will be used. Data from spiked QC samples, within a lot, will be compared to control chart limits to demonstrate that analysis of the lot is under control and will be used to update the charts. X-R control charts will be used in the Quality Assurance (QA) Program.

Control charts are prepared for each target analyte using percent recovery (found concentration - method blank/ spiked concentration x 100) data from the duplicate spiked QC samples variations in spiking solution concentrations.


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To prepare control charts, the analyst should have access to the following data:

1. Average (x) percent recovery for the two high concentration spiked QC samples in each lot,
2. Difference (R) between the percent recoveries for the two high concentration spiked QC samples in each lot,
3. Three-point moving average (x) spike recovery of the low-concentration spike QC sample, and
4. Three-point moving difference (R) between the percent recoveries for the low-concentration spike QC sample.

The initial control chart shall be prepared using the four pairs of certification data closest to the spiking concentration used during analysis. The average (x), average range (R), and control limits for x and R shall be updates after each lot for the first 20 lots. Limits established after Lot 20 will be used for the next 20 lots. Control charts will be updated after each 20 lots thereafter using the most recent 40 lots of QC data. In updating the control charts, the new data must be combined with the individual values of previous percent recoveries and not the mean of all previous data.

Initial control chart limits are presented in Table IX-2.

## X. REFERENCES

U.S. Army Toxic and Hazardous Materials Agency, (USATHAMA). 1987. QA Plan (December 1985, 2nd Edition, March 1987).

## XI. DATA

### A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION

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### B. PRE-CERTIFICATION CALIBRATION

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### C. DAILY CALIBRATION AND CHROMATOGRAM

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Environmental Science & Engineering, Inc.
Gainesville Laboratory
Gainesville, Florida

**TITLE: DETERMINATION OF ORGANOSULFUR COMPOUNDS IN WATER BY GAS CHROMATOGRAPHY (METHOD UL04)**

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**TITLE: DETERMINATION OF ORGANOSULFUR COMPOUNDS IN WATER BY GAS CHROMATOGRAPHY (METHOD UL04)**

## I. SUMMARY

### A. ANALYTES

This method is applicable to the analysis of the following organosulfur compounds in environmental water samples.

Dimethyldisulfide (DMDS)
1,4-Oxathiane (OXAT)
1,4-Dithiane (DITH)
p-Chlorophenylmethylsulfide (CPMS)
Benzothiazole (BTZ)
p-Chlorophenylmethylsulfoxide (CPMSO)
p-Chlorophenylmethylsulfone (CPMSO2)

### B. MATRIX

This method is applicable to all environmental water matrices.

### C. GENERAL METHOD

This method employs extraction of the water matrix with methylene chloride, solvent concentration using standard Kuderna-Danish (KD) techniques, and analysis by gas chromatography (GC) using flame-photometric detection (FPD) in the sulfur mode.



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## II. APPLICATION

### A. TESTED CONCENTRATION RANGE

The tested concentrations in "Standard water" samples are:

| Analyte | Tested Concentration Range(ug/L) |
|---|---|
| DMDS | 1.14 to 22.8 |
| OXAT | 1.98 to 39.5 |
| DITH | 1.11 to 22.2 |
| CPMS | 1.26 to 25.3 |
| BTZ | 2.11 to 42.2 |
| CPMSO | 4.23 to 106 |
| CPMSO2 | 4.22 to 106 |

### B. SENSITIVITY

The instrumental responses for each compound, reported in peak area units, at the certified reporting limits for the calibration curve were determined by the Hubaux and Vos detection limits from the calibration curve data.

| Analyte | Calibration Reporting Limit (ug/L) | Area Counts |
|---|---|---|
| DMDS | 1.14 | 57,000 |
| OXAT | 1.98 | 23,200 |
| DITH | 1.11 | 32,600 |
| CPMS | 1.26 | 15,600 |
| BTZ | 2.11 | 26,600 |
| CPMSO | 4.23 | 37,000 |
| CPMSO2 | 4.72 | 33,400 |



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## C. REPORTING LIMITS

The corresponding upper certified range and reporting limits for each analyte in environmental water samples are:

| Analyte | Certified Reporting Limit (ug/L) | Upper Range (ug/L) |
|---|---|---|
| DMDS | 1.14 | 22.8 |
| OXAT | 1.98 | 39.5 |
| DITH | 1.11 | 22.2 |
| CPMS | 1.26 | 25.3 |
| BTZ | 2.11 | 42.2 |
| CPMSO | 4.23 | 106 |
| CPMSO2 | 4.72 | 106 |

## D. INTERFERENCES

Solvents, reagents, glassware, and other sample processing equipment may yield chromatograms with interfering peaks. All reagents, glassware, and sample handling equipment must be free from interferences that have retention times equal to the retention times of the compounds of interest.

## E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 sample extracts in an 8-hour day. One analyst can perform approximately 10 extractions in an 8-hour day.

## F. SAFETY INFORMATION

The target compounds in this method are toxic. The preparation of all standards should be performed in a laboratory hood. Adequate dermal protection must be used when handling samples and standards.



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## III. APPARATUS AND CHEMICALS

### A. GLASSWARE/HARDWARE

1. 1,000-milliliter (mL) separatory funnel with Teflon stopcock.
2. 500-mL KD concentrator.
3. Class A volumetric flasks (5-, 25-, 50- and 100-mL).
4. Class A volumetric pipettes (0.5- to 25-mL).
5. Microsyringes (250- and 1,000-uL).
6. Pasteur pipettes (disposable).
7. Graduated cylinder (1,000-mL).
8. Amber-glass vials (9-mL with Teflon -lined crimp caps).
9. Snyder columns (KD, 3-ball macro- and modified micro-Snyder).
10. Concentrator tubes (KD, 25-mL graduated, with ground-glass stoppers).
11. Glass funnels [58-millimeter (mm) short-stem].
12. Hengar boiling chips (10/40 mesh, pre-extracted with methylene chloride; available from Hengar Co., Philadelphia, PA, catalog number 136CC).
13. Glass vials (2-mL with Teflon -lined crimp-seal caps for use with an automatic sampler).
14. Amber bottles (60-mL with Teflon -lined screw caps).
15. Hot water bath.
16. Stainless-steel spatulas.
17. Analytical balance [Mettler AE160 or equivalent, with 0.0001-gram (g) sensitivity].



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18. Glass wool (silanized).

19. Tipit (50-mL).

## B. INSTRUMENTATION

1. Gas chromatograph with an FPD (Varian 3400 or equivalent), equipped with an automatic sampler (Varian 8100 or equivalent) and integrator (Spectra-Physics 4270 or equivalent).

2. Chromatographic conditions:

   a. Column: 5-percent SP-1000 on Chromosorb [6-foot (ft) by 2-mm inside diameter (ID) by 6-mm outside diameter (OD) column].

   b. Injector temperature: 200 degrees Celsius (°C).

   c. Temperature program: 80°C, hold 3 minutes (min), heat at 32 degrees Celsius per minute (°C/min) to 240°C, hold 7 min.

   d. Detector temperature: 300°C.

   e. Gas flow: Helium at 30 milliliters per minute (mL/min), hydrogen at 140 mL/min, Air 1 at 80 mL/min, air 2 at 170 mL/min.

   f. Injection volume: 6 microliters (uL).

   g. Retention times: A retention time window of ± 0.02 min. of the retention time of the high standard for each analyte will be used for compound identification. Retention times at time of certification are:

| Analyte | Retention Time (min) |
|---|---|
| DMDS | 1.29 ± 0.04 |
| OXAT | 4.02 ± 0.12 |
| DITH | 6.17 ± 0.19 |
| CPMS | 7.42 ± 0.22 |
| BTZ | 7.72 ± 0.24 |
| CPMSO | 10.07 ± 0.30 |
| CPMSO2 | 11.20 ± 0.34 |



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## C. ANALYTES

| Analytes | Chemical Abstract Service (CAS) Number | Boiling Point (°C) |
|---|---|---|
| DMDS | 624-92-0 | -- |
| OXAT | 15890-15-1 | 147 |
| DITH | 505-29-3 | 200 |
| CPMS | 123-09-1 | -- |
| BTZ | 95-16-9 | 231 |
| CPMSO | 934-73-6 | -- |
| CPMSO2 | 98-57-7 | -- |

## D. REAGENTS AND STANDARD ANALYTICAL REFERENCE MATERIALS (SARMS)

1. Methylene chloride (pesticide-grade).

2. Acetone (pesticide-grade).

3. Sodium chloride (reagent grade).

4. Sodium sulfate [American Chemical Society (ACS), granular, anhydrous, heated at 400°C for 4 hours in a muffle furnace].

5. "Standard water" [distilled water containing 100 milligrams per liter (mg/L) each of sulfate and chloride].

6. DMDS [Standard Analytical Reference Material (SARM) ID No. 1378, obtained from USAEC].

7. OXAT (SARM ID No. PA2340, from USAEC).

8. DITH (SARM ID No. PA2318, from USAEC).

9. CPMS (SARM ID No. PA2320, from USAEC).

10. BTZ (from Aldrich Chemical Co. Milwaukee, WI 91928DP).



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11. CPMSO (SARM ID No. PA2321, from USAEC).

12. CPMSO2 (SARM ID No. PA2322, from USAEC).

## IV. CALIBRATION

### A. INITIAL CALIBRATION

#### 1. PREPARATION OF STANDARDS

Individual primary stock calibration standards are prepared by weighing approximately 32 milligrams (mg) of each of the six target analytes into separate 25-mL volumetric flasks, then diluting to volume with methylene chloride. The nominal concentrations of each of the primary stock calibration standards prepared this way for each analyte is 1,280 ug/mL.

A composite primary stock calibration standard is prepared by adding 5 mL of each individual stock calibration standard to methylene chloride in a 50-mL volumetric flask and then diluting to volume with methylene chloride. The nominal concentrations in the composite primary stock calibration standard for each analyte is 128 ug/mL.

A composite secondary calibration stock is prepared by placing 5 mL of the composite primary calibration stock into a 50-mL volumetric flask and diluting to volume with methylene chloride (a 1:10 dilution of the composite primary calibration stock). The nominal concentration in the composite secondary calibration stock for each analyte is 12.8 ug/mL.

Composite working calibration standards are prepared using methylene chloride, the composite primary and secondary stock calibration standards and volumetric pipettes as shown in Table IV-1.

#### 2. INSTRUMENT CALIBRATION

To calibrate the instrument, 6 uL of each standard in Table IV-1 is injected into the instrument in the same manner as a sample extract. Duplicate composite calibration standards are analyzed during precertification calibration, and the single dilutions of the composite standards are analyzed during initial calibration.



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Currently an independent reference standard is not available for organosulfur compounds in water. Meanwhile an independent stock will be prepared to serve as a reference standard. The reference must be analyzed along with the initial and precertification calibration standards, and the results must be within ± 25% of the true value for the calibration to be considered valid. If the analysis of the reference standard fails, the source of the problem must be identified and corrected. The initial calibration and analysis of the independent reference standard must be repeated. The result of the second analysis of the independent reference standard must be within the acceptable limits, as specified by the source of the standard, before the analysis of samples may proceed. Since initial calibrations are performed daily, a reference is required at least weekly.

Response of the FPD to sulfur when operated in the sulfur mode is logarithmic and increases as the square of the concentration. The square root of the response is plotted against the concentration for determination of the concentration of analyte in solution.

## 3. CALIBRATION CHECKS

At the end of the daily instrumental analysis, the highest working calibration standard is injected into the GC. The response of the recovery of this end-of-day analysis should be ± 25% of the response or recovery obtained from the analysis of the same working calibration standard curve analyzed that day. If the first calibration check does not pass this criterion, then the standard may be reinjected and compared to the initial response. The post run standard data is documented and the percent drift is calculated on the chromatographic logsheet. An explanation must be provided if the calibration check is not acceptable for the data to be reported, otherwise the samples are rerun.

## 4. RETENTION TIME WINDOWS

Retention time windows for a method are established based on historical data of daily performance of the method or by using retention windows from a reference method. If a reference method with established windows is not available the windows are calculated by calculating the relative standard deviation of the retention variation throughout the run for all standards, control samples and continuing calibration standards and



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multiplying the result by three and rounded to 1 significant figure. The windows are expressed in absolute minutes where the retention variation is minimal throughout the chromatographic run and as a percentage of the retention time in methods whose variation is proportional to retention time. The retention window is applied to retention times from a specified standard during initial or daily calibration. These retention window values are entered into the method used for processing of the chromatographic data for computerized identification/rejection of detected peaks. The analyst can override the identification/rejection of a peak by providing documentation of his decision.

## B. DAILY CALIBRATION

For daily calibration an initial calibration curve and QC checks will be performed as stated in Section IV.A.



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Table IV-1. Preparation of Initial Calibration Standards.

| Standard | Composite Standard Used (ug/mL) | Volume Used (mL) | Final Volume (mL) | Nominal Concentration (ug/mL) |
|---|---|---|---|---|
| Blank | - | 0 | 25 | 0.0 |
| A | 128 | 5.0 | 25 | 25.6 |
| B | 128 | 4.0 | 50 | 10.2 |
| C | 128 | 2.0 | 50 | 5.12 |
| D | 128 | 1.0 | 50 | 2.56 |
| E | 12.8 | 4.0 | 50 | 1.02 |
| F | 12.8 | 5.0 | 100 | 0.64 |
| G | 12.8 | 2.0 | 50 | 0.51 |
| H | 12.8 | 2.5 | 100 | 0.32 |
| I | 12.8 | 1.0 | 50 | 0.26 |
| J | 12.8 | 0.8 | 50 | 0.21 |

Source: ESE

Table IV-2. Standards Used for Regression for Daily Calibration.

| Compound | Standards |
|---|---|
| DMDS | B through J |
| OXAT | B through J |
| DITH | B through J |
| CPMS | B through J |
| BTZ | B through J |
| CPMSO | A through F |
| CPMSO2 | A through F |

Source: ESE


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## V. CERTIFICATION TESTING

See Attachment A.

## VI. SAMPLE HANDLING STORAGE

### A. SAMPLING PROCEDURE

There are no special considerations required due to the nature of organosulfur compounds. The samples need to be chilled to 4°C immediately following sampling.

### B. CONTAINERS

Sampling containers used are 1.2 Liter glass amber jars with a teflon-lined cap.

### C. STORAGE CONDITIONS

Samples are to be kept on ice in coolers until shipped to the analytical lab. Upon arrival, the samples are stored at 4°C in a walk-in refrigerator.

### D. HOLDING TIME LIMITS

The holding time limit is seven days from the time of sampling to extraction of the sample and 40 days from extraction to analysis.

### E. SOLUTION VERIFICATION

When fresh control spiking solutions are prepared, they must be verified to determine that:

1. The previous spiking solution had or had not deteriorated.

2. The new solution was correctly prepared. Therefore, dilute working spike solutions will be checked against working standards before initial use and again within seven days before subsequent use. The spike solutions should use the same instrumentation used on the samples.



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## VII. PROCEDURE

Daily quality control spikes (Section IX.A) and environmental samples are prepared for analysis and analyzed as follows:

### A. SEPARATIONS

1. Using a 1-liter graduated cylinder, transfer 800 mL of sample (or standard water for daily control spikes) to a 1-liter separatory funnel.

2. Daily control spikes are spiked as specified in Table IX-1.

3. Add 50 mL of methylene chloride to each separatory funnel.

4. Extract the sample by shaking the funnel for 2 min, with periodic venting to release solvent vapor pressure.

5. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must mechanically complete the phase separation. The optimum technique depends on the sample but may include either sonication or stirring, filtering, and filtration of the emulsion through glass wool.

6. Pass the methylene chloride extract through unwashed, baked (400°C, 4 hours) sodium sulfate into a 500-mL KD apparatus. Rinse the sodium sulfate with methylene chloride after the extract has dried.

7. Add a second 50-mL volume of methylene chloride to the separatory funnel and complete the extraction procedure a second time, combining the extracts in the KD apparatus.

8. Perform a third extraction in the same manner.

9. Add 1 to 2 clean boiling chips to the KD apparatus and attach a 3-ball Snyder column. Prewet the Snyder column by adding approximately 1 mL of methylene chloride to the top.



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10. Place the KD apparatus on the hot water bath (70°C to 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches approximately 5 mL, remove the KD apparatus and allow it to drain for at least 10 min while cooling.

11. Rinse the Snyder column, remove the Snyder column, and rinse the flask into the concentrator tube with approximately 5 mL of methylene chloride. Rinse the lower joint into the concentrator tube when separating it from the flask.

12. Attach a modified micro-Snyder column to the concentrator tube in the hot water bath. When the apparent volume of the liquid reaches 2 mL, remove the concentrator tube and allow it to cool for approximately 10 min.

13. Adjust the final volume to 5 mL by adding methylene chloride. Transfer approximately 1 mL to an autosampler vial and seal. Save the remaining extract at 4°C as a backup. Sample extracts must be analyzed within 40 days of extraction.

## B. CHEMICAL REACTIONS

No chemical reactions are required by this method.

## C. INSTRUMENTAL ANALYSIS

1. Perform daily instrument calibration as described in Section IV.B. Use the instrument conditions listed in Section III.B.

2. Place the sample extracts in the autosampler tray and inject 5 uL of each sample extract into the instrument under exactly the same conditions as those under which it was calibrated.



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## VIII. CALCULATIONS

A linear or quadratic calibration curve is constructed from the calibration data by plotting the square root of the response versus the concentration of each standard. The calibration curve slope and intercept are determined by regression. The concentration of a target compound in the extract is calculated by substituting the square root of the response into the calibration curve equation. The concentration of each analyte in the original water sample is determined by the following formula:

$$\text{Concentration (ug/L)} = \frac{\text{Extract Conc. (ng/L)} \times 5\ \text{mL}}{V_s\ \text{(L)} \times 1{,}000\ \text{mL/L}}$$

where: Extract Conc. = Calculated concentration in the extract in nanograms per milliliter (ng/mL);
5 mL = Final extract volume in milliliters;
$V_s$ = Sample volume in liters (nominally 0.8 L).

Dilution factors, if any, are reported separately.

## IX. DAILY QUALITY CONTROL

### A. CONTROL SAMPLES

The daily control spikes required are a Standard Matrix Method Blank, one low level Standard Matrix Spike at approximately twice the CRL, and two high-level Standard Matrix Spikes at five times the low level spike. Individual stock spiking solutions are prepared as discussed in Section V.A.1. A composite daily control spike solution is prepared (in a 100-mL volumetric flask) by diluting the following volumes of the individual primary stock control spiking solutions to volume with acetone:

| Analyte | mLs Stock | Concentration (ug/mL) |
|---|---|---|
| DMDS | 1.5 | 15.0 |
| OXAT | 2.0 | 20.0 |
| DITH | 1.5 | 15.0 |
| CPMS | 1.5 | 15.0 |
| BTZ | 2.0 | 20.0 |
| CPMSO | 4.0 | 40.0 |
| CPMSO2 | 5.0 | 50.0 |



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The spike solution is verified when needed as described in Section VI.E. From the composite daily control spike solution, the daily control spikes are prepared as shown in Table IX-1.

## B. CONTROL CHARTS

In the initial construction of the control charts, data from the laboratory certification analysis will be used. Data from spiked QC samples, within a lot, will be compared to control chart limits to demonstrate that analysis of the lot are under control and will be used to update the charts. X-R control charts will be used in the Quality Assurance (QA) Program. Control charts are prepared for each target analyte using percent recovery (found concentration - method blank/ spiked concentration x 100) data from the duplicate spiked QC samples variations in spiking solution concentrations.

To prepare control charts, the analyst should have access to the following data:

1. Average (x) percent recovery for the two high concentration spiked QC samples in each lot.

2. Difference (R) between the percent recoveries for the two high concentration spiked QC samples in each lot.

3. Three-point moving average (x) spike recovery of the low-concentration spike QC sample, and

4. Three-point moving difference (R) between the percent recoveries for the low-concentration spike QC sample.

The initial control chart shall be prepared using the four pairs of certification data closest to the spiking concentration used during analysis. The average (x), average range (R), and control limits for x and R shall be updates after each lot for the first 20 lots. Limits established after Lot 20 will be used for the next 20 lots. Control charts will be updated after each 20 lots thereafter using the most recent 40 lots of QC data. In updating the control charts, the new data must be combined with the individual values of previous percent recoveries and not the mean of all previous data. Initial control chart limits are presented in Table IX-2.



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## X. REFERENCES

U.S. Army Toxic and Hazardous Materials Agency, (USATHAMA). 1987. QA Plan (December 1985, 2nd Edition, March 1987).

## XI. DATA

### A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION

Appendix 1

### B. PRE-CERTIFICATION CALIBRATION

Appendix 2

### C. DAILY CALIBRATION AND CHROMATOGRAM

Appendix 3
(Reference check sample not required during time of original certification)

### D. CERTIFICATION DATA

Appendix 4

## XII. ATTACHMENT

### A. DATA

### B. SECTION V - CERTIFICATION TESTING

### C. UL04 CHECKLIST



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# APPENDIX E

# APPENDIX E
# ESE LABORATORY STATEMENT OF QUALIFICATIONS AND QUALITY ASSURANCE MANUAL

Control No. 024

LCQAP August 1994

# Laboratory Comprehensive Quality Assurance Plan

Prepared by:

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ESE, 1994

# APPROVALS

This Quality Assurance/Quality Control (QA/QC) Manual has been read and understood and approved for use in the Gainesville Laboratory of Environmental Science & Engineering, Inc. (ESE).

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## LIST OF ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| AAS | atomic absorption spectrophotometry |
| AIHA | American Industrial Hygiene Association |
| ASTM | American Society for Testing and Materials |
| BFB | bromofluorobenzene |
| BOD | biochemical oxygen demand |
| °C | degrees Celsius |
| CCC | calibration check compounds |
| CCV | continuing calibration verification |
| CLASS | Chemical Laboratory Analysis and Scheduling System |
| COD | chemical oxygen demand |
| D | detection limit |
| DBCP | 1,2-Dibromo-3-chloropropane |
| DFTPP | decafluorotriphenylphosphine |
| DHRS | Department of Health and Rehabilitative Services |
| DI | deionized |
| DO | dissolved oxygen |
| DOT | Department of Transportation |
| ECD | electron capture detector |
| EDB | 1,2-Dibromoethane |
| ELAP | Environmental Laboratory Approval Program |
| EPA | U.S. Environmental Protection Agency |
| ESE | Environmental Science & Engineering, Inc. |
| eV | electronvolt |
| FDER | Florida Department of Environmental Regulation |
| FID | flame ionization detector |
| FPD | flame photometric detector |
| FR | fraction code |

## LIST OF ACRONYMS AND ABBREVIATIONS
(Continued, Page 2 of 5)

| | |
|---|---|
| FRN | frame reference number |
| ft | foot |
| g | gram |
| g/kg | grams per kilogram |
| GC | gas chromatography |
| GC/FID | GC employing flame ionization detection |
| GC/FPD | GC employing flame photometric detection |
| GC/HPLC | gas chromatography/high performance liquid chromatography |
| GC/MS | gas chromatograph/mass spectrometer |
| GC/MS/DS | gas chromatography/mass spectrometry/data system |
| GC/NPD | GC employing nitrogen-phosphorus detection |
| GLP | Good Laboratory Practice |
| HCL | hydrochloric acid |
| $HNO_3$ | nitric acid |
| HPLC | high performance liquid chromatography |
| $H_2SO_4$ | sulfuric acid |
| ICAP | inductively coupled argon plasma |
| ICS | interference check solution |
| ICV | initial calibration verification |
| ID | identification |
| IR | infrared |
| KCl | potassium chloride |
| kg | kilogram |
| KOH | potassium hydroxide |
| L | liter |

LIST OF ACRONYMS AND ABBREVIATIONS
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| | |
|---|---|
| LC50 | 50 percent lethal concentration |
| MBAS | methylene blue active substances |
| MDL | method detection limits |
| mg/kg | milligrams per kilogram |
| mg/L | milligrams per liter |
| mL | milliliter |
| mm | millimeter |
| $mm^2$ | square millimeter |
| MSC | matrix spike compound |
| NaOH | sodium hydroxide |
| $Na_2S_2O_3$ | sodium thiosulfate |
| ng | nanogram |
| NIOSH | National Institute of Occupational Safety and Health |
| NIST | National Institute of Standards and Technology |
| NTU | nephelometric turbidity unit |
| PAT | Proficiency Analytical Testing Program |
| PCB | polychlorinated biphenyl |
| PCP | pentachlorophenol |
| PCU | platinum-cobalt unit |
| % RSD | percent relative standard deviation |
| PFS | prefield setup |
| PID | photoionization device |
| PNA | polynuclear aromatic hydrocarbon |
| ppb | parts per billion |

## LIST OF ACRONYMS AND ABBREVIATIONS
(Continued, Page 4 of 5)

| | |
|---|---|
| ppt | parts per thousand |
| psi | pounds per square inch |
| PVC | polyvinyl chloride |
| QA | quality assurance |
| QA/QC | quality assurance/quality control |
| QC | quality control |
| QCC | quality control check |
| RF | response factor |
| RPD | relative percent difference |
| S | surrogate |
| SCT | salinity, conductivity, and temperature |
| SDS | sodium dodecyl sulfate |
| SOP | standard operating procedure |
| STORET | storage and retrieval |
| THMS | trihalomethanes |
| TIC | tentatively identified compound |
| TOC | total organic carbon |
| TOX | total organic halides |
| TRPH | total recoverable petroleum hydrocarbons |
| µg/g | micrograms per gram |
| µg/L | micrograms per liter |
| µL | microliter |
| µmho/cm | micromhos per centimeter |
| UPS | United Parcel Service |
| USACE | U.S. Army Corps of Engineers |
| USGS | U.S. Geological Survey |

## LIST OF ACRONYMS AND ABBREVIATIONS

(Continued, Page 5 of 5)

| | |
|---|---|
| UV | ultraviolet |
| VOC | volatile organic compound |
| YSI | Yellow Springs Instruments |

# 3.0 STATEMENT OF POLICY

## 3.1 QUALITY ASSURANCE (QA) STATEMENT OF POLICY

It is the policy of Environmental Science & Engineering, Inc. (ESE), Gainesville Laboratory, to maintain an active quality assurance/quality control (QA/QC) program to provide the highest quality information and ensure the highest professional standards in deliverables for every project undertaken. An established QA/QC philosophy and program are essential for any organization to consistently produce valid laboratory data. To be valid, data must be generated under controlled conditions which do not adversely affect data quality. Data must also be interpreted by capable professionals who are trained in appropriate scientific disciplines, maintain a current knowledge of their field, and are experts in the applications for which the data will be used. The objectives of the QA/QC program are to estimate the quality of each analytical system including precision, accuracy, and sensitivity sufficient for each project. The QA/QC program also assists in the early recognition of deficiencies which might affect data quality, validate data, and define data usability.

ESE's commitment to the QA/QC process is evidenced by the establishment of a separate QA/Safety Division which is responsible for overseeing QA activities and an internal QA/QC department which is responsible for overseeing QA and QC activities within the Gainesville Laboratory. QA is a management system which ensures the completion of predetermined activities. All activities are recorded including traceability, completeness, and document security. QC refers to specific actions taken to ensure that system performance is consistent with established limits. These actions ensure precision, accuracy, comparability, and completeness of analytical data.

ESE supports a corporate-wide Quality Education System (QES). All employees are trained in the quality improvement process. This training is supplemented at the departmental level by instructing employees on the importance of QA/QC and the price of nonconformance.

## 3.2 SCOPE

All major environmental studies and analyses conducted by ESE Gainesville Laboratory for projects under the guidance of the Department of Energy (DOE), the Naval Energy And Environmental Support Activity (NEESA), the Hazardous Waste Remedial Actions Program (HAZWRAP), the Florida Department of Environmental Regulations (FDER) or any state and federal government agency must be performed in accordance with this Laboratory Comprehensive Quality Assurance Plan (LCQAP).

This LCQAP will be the basis for all project-specific QA plans except where alternate QA plans are required. When appropriate, this LCQAP will be filed with a client and/or regulatory agency and, once approved, may be referenced in lieu of repetitive submission of plans in which only a portion of the information is changed.

## 3.3 DOCUMENT CONTROL

This LCQAP will be revised periodically as procedural changes become necessary. Changes will be documented by the date and revision number of each section. ESE's QA/QC Department will keep a distribution list and assign a unique number to each copy of the LCQAP. When a section is revised, the revision date will replace the original date in the heading code, the revision number will be changed, and the table of contents will be updated. Copies of the revised sections will be provided to each individual on the distribution list.

These procedures will apply once the plan has been finalized and implemented; these procedures will not apply to draft documents.

# 4.0 ORGANIZATION AND RESPONSIBILITIES

## 4.1 LABORATORY OPERATIONS CAPABILITIES

ESE laboratory operations include the following capabilities:

1. Groundwater and surface water analysis,
2. Soil and sediment analysis,
3. Wastewater analysis,
4. Air monitoring screening,
5. Drum analysis, and
6. Underground storage tank analysis.

## 4.2 KEY PERSONNEL

This section includes ESE's Gainesville Laboratory key personnel identified by title and a brief summary of each individuals responsibilities. An organization chart of the Gainesville Laboratory is presented in Figure 4-1.

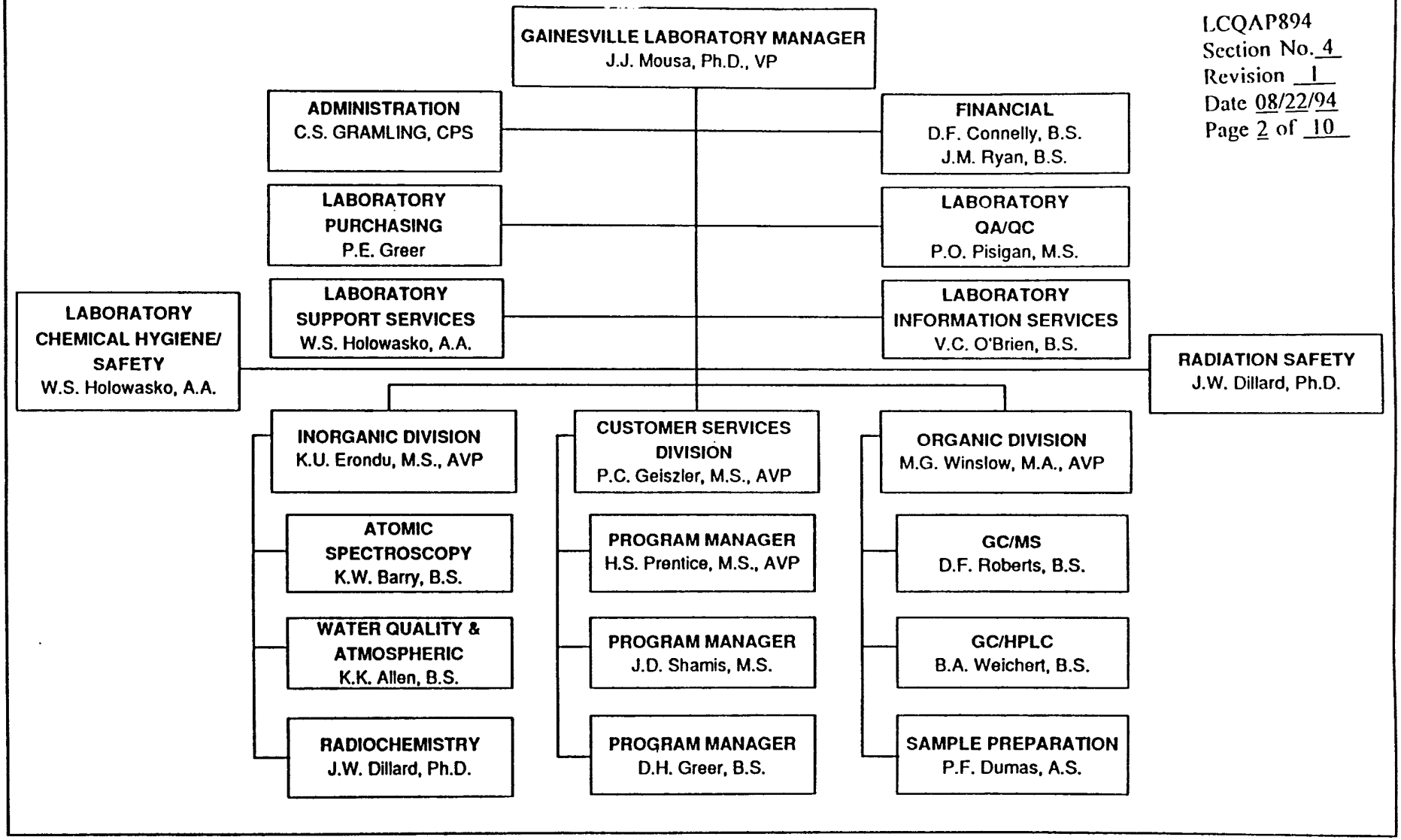
### 4.2.1 LABORATORY OPERATIONS PERSONNEL

#### 4.2.1.1 Laboratory Director

The Laboratory Director is responsible for the overall management of the analytical laboratory, including the appointment and supervision of the Laboratory Information Services Manager, Laboratory Division Managers, Customer Service Manager, and Laboratory QA/QC Manager. He is responsible for approving all analytical procedures and associated QA/QC procedures.

#### 4.2.1.2 Laboratory QA/QC Manager

The Laboratory QA/QC Manager is responsible for the overall management of the laboratory QA/QC operations, including the appointment and supervision of the Laboratory QA/QC Coordinators.




**ADMINISTRATIVE ORGANIZATION**
**ANALYTICAL SERVICES LABORATORY,**
**GAINESVILLE, FLORIDA**

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

#### 4.2.1.3 Laboratory QA/QC Coordinator

The Laboratory QA/QC Coordinator is responsible for coordinating the certification programs of the Gainesville Laboratory. The Laboratory QA/QC Coordinator also performs laboratory and data audits, and maintains QC records for inspection by ESE project management and the Project QA Coordinator. The Laboratory QA/QC Coordinator provides guidance and coordination to resolve any QA/QC deficiencies and reviews precision, accuracy, and blind samples for projects to ensure completeness of the QC data.

#### 4.2.1.4 Laboratory Information Services Manager

The Laboratory Information Services Manager oversees ESE's computerized data management system and is responsible for the following:

1. Maintaining ESE's Chemical Laboratory Analysis Scheduling System (CLASS™) (refer to Section 10.1);
2. Approval of all changes made to CLASS™; and
3. Storage of chain-of-custody logsheets, analytical batch folders for all departments in one central location, and all other computerized data.

#### 4.2.1.5 Laboratory Division Managers

The Analytical Laboratory Division Managers are responsible for the overall management of their respective analytical inorganic and organic sections or including the appointment and supervision of their Department Managers. The Customer Service Manager is responsible for the overall management of the project operations within the laboratory including the appointment and supervision of the Laboratory Program Manager.

#### 4.2.1.6 Laboratory Program Manager

The Laboratory Program Manager is responsible for the overall management of the project operations within the Gainesville Laboratory, including the appointment and supervision of the Laboratory Coordinators and Sample Custodian.

#### 4.2.1.7 Laboratory Coordinator

The Laboratory Coordinator acts as liaison between field and laboratory operations and is responsible for the following:

1. Coordination of sample analyses to meet project or client objectives;
2. Preparation of analytical reports, including coordination with the QA Division or Laboratory QA/QC Manager and laboratory management to ensure that the data are validated prior to release to the client;
3. Review of any QA/QC deficiencies reported by the Laboratory Department Manager; and
4. Coordination of any data changes resulting from review by the Laboratory QA/QC Coordinator, and/or Project Manager.

#### 4.2.1.8 Sample Custodian

The Sample Custodian checks in the samples from the field upon receipt by the laboratory. The Sample Custodian compares all samples contained in the shipment to the logsheet(s) to ensure that all samples designated on the logsheet have been received. The Sample Custodian will note any special remarks concerning the shipment and deliver the logsheet to the Laboratory Information Services Manager. The Sample Custodian places samples in appropriate storage areas and notifies the appropriate Laboratory Coordinators and Laboratory Department Managers, or designee.

#### 4.2.1.9 Laboratory Department Managers

The Laboratory Department Managers are responsible for providing consistent and accurate laboratory data and technical reports produced by their analysts. These individuals are responsible to the Project Manager to ensure that all personnel under their direction are knowledgeable of the QA/QC requirements of the project and that all QC and technical review procedures are followed and documentation is provided.

#### 4.2.1.10 Laboratory Analyst

Laboratory Analysts must perform preliminary QC checks to ensure that each batch of data being generated passes all the required QC criteria.

#### 4.2.1.11 Radiation Safety Officer

The Radiation Safety Officer is responsible for enforcing company policy in handling radioactive materials in order to ensure protection of all ESE employees and facilities from exposure to radiation. He is responsible for evaluating new requirements by Florida DHRS and/or NRC to ensure that ESE's Radioactive Materials License is in compliance and reviewing and evaluating standard operating procedures and sections of the Laboratory Safety Manual involving the use of radioactive materials for appropriateness. He is also responsible for monitoring spills and leaks in areas where radioactive materials are used, leak testing and documenting all sealed and unsealed radiation sources, and issuing, collecting and shipping personal monitoring devices. He conducts radiation safety training of all personnel prior to their handling of radioactive materials, and maintains an inventory of all radioactive materials. He will remove and maintain documentation for the removal of radioactive samples as necessary to a temporary storage area within the company facility.

#### 4.2.1.12 Laboratory Chemical Hygiene Officer

The Chemical Hygiene Officer (CHO) will assist laboratory supervisors in implementing the Chemical Hygiene Program. He will provide training (in accordance with Health and Safety (H&S) SOP 112- Chemical Hygiene Program Training), review laboratory safety manuals and SOPs, and perform safety audits of laboratory procedures and safety inspections of laboratory protective equipment to determine compliance. Areas of non-compliance will be reported to the appropriate supervisor or manager. The CHO will make this Program readily available to laboratory workers and supervisors. He will evaluate worker chemical exposure (in accordance with H&S SOP 140) and will provide a written report of each exposure assessment or determination to the Laboratory Director for action

as necessary. Any over exposure shall be reported to the Regional Health and Safety Coordinator (RHSC).

## 4.3 ANALYTICAL CHEMISTRY FACILITIES

ESE's Gainesville laboratory currently comprises 41,000 $ft^2$ of laboratory and office space. In order to facilitate the movement of samples through the laboratory and maintain uncompromised chain-of-custody, the laboratory has separate functional areas dedicated to sample receipt, sample storage, sample preparation, and sample analysis.

The laboratory has approximately 500 $ft^2$ of space dedicated to sample check-in, where samples are unpacked and logged into the laboratory tracking system. Large walk-in cold rooms are used for storage of environmental samples at 4 $\pm$ °C, and approximately 1,100 $ft^3$ of walk-in and reach-in freezers are available for storing biota samples at -10°C or lower. Throughout the laboratory facilities, numerous smaller refrigerator and freezer units are maintained for storage of standards, spiking solutions, and sample extracts.

The laboratory has dedicated areas for organic extractions, inorganic preparation, metals digestion, GC/MS and HPLC/MS analysis, inductively coupled plasma (ICP) and AAS analysis, classical water quality analysis, radiological analysis, asbestos analysis, toxic chemicals handling, special projects, and additional support areas housing ovens, analytical balances, glassware washing, kit preparation, chemicals storage, waste storage, etc.

The laboratory is supplied with demineralized water for glassware washing and other functions. Demineralized-distilled water is available for reagent preparation, and supplies of organic-free water are maintained at all times for use in trace organic analysis.

The laboratory facility is equipped throughout with a full range of safety equipment, including fume hoods, eye washes, emergency showers, emergency lights, fire extinguishers, spill cleanup kits, emergency breathing equipment, fire pull boxes, smoke alarms, warning signs, lighted exit signs, safety glasses, and fire blankets.

## 4.4 ANALYTICAL CHEMISTRY INSTRUMENTATION

ESE's Gainesville laboratory utilizes state-of-the-art analytical instrumentation for multi-matrix chemical analyses. Table 4-1 lists the laboratory's major analytical instrumentation.

Currently, 27 GCs with FIDs, nitrogen-phosphorus detectors (NPD), FPDs, PIDs, HECDs, and ECDs are available for project use. In addition, there are twelve HPLCs with UV, fluorometric, conductivity, electrochemical, and radiochemical detectors. Several units are equipped with post-column derivitization modules and fluorescence spectrometers. A photodiode array spectrometer is also available for simultaneous multi-wavelength monitoring. Autosamplers are used with the GCs and HPLCs for sample handling efficiency, increased sample throughput, and greater precision and accuracy. Automated data acquisition systems are dedicated to each chromatographic unit.

ESE's Gainesville laboratory has ten GC/MSs: four are dedicated to volatile organic analyses, four to semivolatile organic analyses, and two to air analysis. All GC/MS systems are equipped with automatic samples and state-of-the-art computer data systems. The mass spectral libraries are the combined Wiley/NIST library with 77,000 reference spectra and the EPA/NIST library with 42,000 reference spectra. The GC/MS facilities also have standalone GC/FIDs for sample screening. In addition, the laboratory has an HPLC/MS for specialized analysis of thermally labile, polar/water soluble, and macro-molecular organic species.

Table 4-1. Major Analytical Instrumentation

| Instrument | Total | Manufacturer | Model | Year Purchased |
|---|---|---|---|---|
| Organics | | | | |
| GC/MS | 10 | Finnigan (2) | Incos 50, 500 | 1987 |
| | | Hewlett-Packard (7) | 5970 (3) | 1985, 88, 90 |
| | | | 5987 (2) | 1977, 78 |
| | | | 5988 | 1984 |
| | | | 5996 | 1984 |
| LC/MS | 1 | Hewlett-Packard | 59980 | 1990 |
| GC | 27 | Hewlett-Packard (18) | 5730 (3) | 1978, 79, 89 |
| | | | 5880 | 1982 |
| | | | 5890 (14) | 1984 (3), 85 (2), 86 (5), 91, 93 (2), 94 (1) |
| | | Tracor (5) | 540 | 1986 (3), 87 (2) |
| | | Varian (2) | 3400 | 1985 |
| | | Shimadzu (2) | GC-14 | 1991 |
| HPLC | 12 | Altex (2) | 332 | 1979 |
| | | | 420 | 1980 |
| | | Shimadzu (6) | SCL-6A (6) | 1988, 89, 90 (3), 91 |
| | | Hewlett-Packard (4) | 1090 | 1992 |
| | | | 1050 | 1992, 93, 94 |
| Metals | | | | |
| ICP/MS | 1 | Perkin Elmer | Elan 5000 | 1992 |
| ICP | 3 | Jarrell-Ash (2) | 1100, 61 | 1981, 89 |
| | | Perkin-Elmer | P2000 | 1987 |
| AAS | 9 | Buck Scientific (2) | 400 | 1987, 89 |
| | | Perkin-Elmer (6) | 4100 (2) | 1992, 93 |
| | | | 3100 | 1991 |
| | | | 5100 (3) | 1986, 87, 90 |

Table 4-1. Major Analytical Instrumentation (Continued, Page 2 of 2)

| Instrument | Total | Manufacturer | Model | Year Purchased |
|---|---|---|---|---|
| Metals | | | | |
| AAS | | Questron | Merlin | 1993 |
| Water Quality | | | | |
| TOC | 1 | Dohrman | DC-190 | 1991 |
| TOX | 1 | MCI | TOX-10 | 1989 |
| IC | 4 | Dionex | 2120 (2)<br>4000 (2) | 1983<br>1986 |
| Autoanalyzer | 6 | Technicon | II (5)<br>TRAACS 800 | 1980, 83, 85<br>1986 |
| IR | 1 | Perkin-Elmer | 1420 | 1983 |
| UV-VIS | 3 | Bausch & Lomb (2)<br>Perkin-Elmer | 20<br>552 | 1982, 84<br>1984 |
| Radiochemistry | | | | |
| A-B Proportional Counter | 2 | Berthold | LB-770<br>LB-700-2 | 1983<br>1991 |
| Alpha Spectrometer | 20 | Tennelec (2) | TC256 | 1989, 1992 |
| Alpha Scintillation Counter | 12 | Ludlum | 1000 | 1976 |
| Liquid Scintillation Counter | 1 | Beckman | LS-1801 | 1987 |
| GeLi Detector | 2 | EG&G Ortec | GEM20180 | 1987 |
| Nal Dectector | 1 | Bicron | P-14-W | 1987 |
| Biological Oxidizer | 1 | R.J. Harvey | OX-600 | 1990 |

Source: ESE.

Extensive capability for inorganic metals analysis is provided by three ICAPs, both simultaneous and sequential, nine AASs, and one ICP/MS. The bulk of the metals work is acomplished with the ICAPs. The AASs are available with flame, graphite furnace, hydride, and cold vapor capabilities as needed. The ICAPs, ICP/MS, and AASs are equipped with automated sample introduction and data handling systems.

For water quality and air/industrial hygiene studies, a total of six autoanalyzers are available for the routine analysis of sulfur dioxide, nitrite, nitrate, phosphate, sulfate, nitrogen, and silica. Four ICs are also available to enhance ESE's capabilities to analyze for trace level anionic species; munition and chemical agent degradation products; and for a variety of ionic materials, including organic salts and acids.

ESE maintains a state-of-the-art radiation counting laboratory and a wet laboratory reserved specifically for radiochemical analysis. The available instrumentation includes a Gamma-Ray Spectrometer consisting of a 3-inch by 3-inch sodium-iodide scintillation detector housed in a 4-inch steel shield and coupled to a 1,024-channel pulse height analyzer, two Berthod Gas-Flow Proportional Low Background Alpha/Beta Counting System with a cosmic ray guard detector surrounded by a 4-inch shield of low background lead bricks, a Radon Gas Counting System consisting of a Ludlum Radon Flash Photomultiplier Tube coupled to an amplifier scaler-timer, a Thermoluminescence Dosimeter, and Geiger counters. Other instrumentation, including a GeLi Spectrometer, a liquid scintillation counter, and a tissue oxidizer are also available in the radiochemistry laboratory.

Supporting equipment available includes GPC autoprep units for extract cleanup, sonicators with sonaboxes for soil extractions, centrifuges, shaking devices, blenders, tissuemisers, balances, ovens, etc., and a full range of laboratory glassware necessary for all aspects of environmental analytical chemistry.

# 5.0 QA OBJECTIVES FOR MEASUREMENT DATA

## 5.1 LABORATORY ANALYSIS

Analyses are performed according to standard U.S. Environmental Protection Agency (EPA) analytical procedures for analysis of water and soil/sediment unless otherwise specified (Tables 5-1 through 5-65). EPA precision and accuracy data and historic ESE data were used as the basis for developing criteria to assess laboratory method performance and the precision and accuracy of sample data, as noted. These limits are subject to change based on actual historic and current performance; updates will be provided for insertion into all control copies of our site-specific QAPPs, as appropriate. Specific compounds are used for controlling purposes in multianalyte methods and are identified in Tables 5-2 through 5-65. Laboratory method performance is evaluated using calibration checks, blanks, and QC check samples; sample accuracy and precision are evaluated using matrix spike and matrix spike duplicate data.

The reporting limits (RLs) achievable for all parameters are listed in Tables 5-3 through 5-65 (odd numbered tables). These RLs were developed in accordance with specific procedures referenced for each analytical method. The RLs for waters and calculated for solids are typically reported as RLs, if no matrix and/or other interferences (i.e., instrument noise) are found to be present (subject to adjustment for dilutions and/or moisture contents).

The following is a brief explanation of the terms that appear in Tables 5-2 through 5-65. Items that are not applicable are denoted by NA.

Reference: The reference of the standard analytical methodology used for each procedure.

Precision: Evaluated based on the relative percent difference (RPD) of duplicate spikes (see Section 11.0 for definitions).

Accuracy: Evaluated based on the percent recovery of each spike (see Section 11.0 for definition).

Units: Volume in liters (L) [e.g., micrograms per liter ($\mu$g/L)] indicates a water matrix; control spikes are added to either sample matrices or to organic-free laboratory water. Mass in grams (g) or kilograms (kg) [e.g., milligrams per kilogram (mg/kg)] indicates a soil/sediment matrix; control spikes are added to sample matrices, soil, or blank water, depending on the analytical procedure.

Table 5-1. Sample Preparation Methods

| Sample Preparation Method Number | Description | Matrix | Sample Preparation for Methods |
|---|---|---|---|
| EPA 3005 | Acid Digestion | Aqueous | EPA 6010, 7020, 7200, 7210, 7450, 7480, 7610, 7770 |
| EPA 3010 | Acid Digestion | Aqueous | EPA 6010, 7020, 7200, 7210, 7450, 7480, 7610, 7770 |
| EPA 3015 | Acid Digestion | Aqueous | EPA 6020 |
| EPA 3020 | Acid Digestion | Aqueous | EPA 7041, 7060, 7091, 7131, 7191, 7201, 7210, 7421, 7470, 7471, 7481, 7740, 7841, 7911 |
| EPA 3050 | Acid Digestion | Solid | EPA 6010, 7020, 7200, 7210, 7450, 7480, 7610, 7770, 7041, 7060, 7091, 7131, 7191, 7201, 7210, 7421, 7470, 7471, 7481, 7740, 7841, 7911 |
| EPA 3051 | Acid Digestion | Solid | EPA 6020 |
| EPA 3510 | Separator Funnel Liquid-Liquid Extraction | Aqueous | EPA 8040, 8060, 8080, 8120, 8140, 8270, 8310 |
| EPA 3520 | Continuous Liquid-Liquid Extraction | Aqueous | EPA 8040, 8060, 8080, 8120, 8140, 8270, 8310 |
| EPA 3540 | Soxhlet Extraction | Solid | EPA 8040, 8060, 8080, 8120, 8140, 8270, 8310 |
| EPA 3550 | Sonication Extraction | Solid | EPA 8040, 8060, 8080, 8120, 8140, 8270, 8310 |
| EPA 5030 | Purge-And-Trap | Aqueous, Solid | EPA 8010, 8020, 8240, 8260 |
| EPA 3630 | Silica Gel Cleanup | Aqueous, Solid | EPA 8080 |
| EPA 3640 | Gel-Permeation Cleanup | Aqueous, Solid | EPA 8080, 8140, 8270* |
| EPA 3660 | Sulfur Cleanup | Aqueous, Solid | EPA 8080, 8120 |

*Only used for fish, oily, or highly contaminated samples.
Source: ESE.

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, Oil and Grease, TRPH, TOX, and Radiochemical Analysis

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Method Criterion[b,l] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Aluminum, Total | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Aluminum, Total | µg/L | EPA 200.7, 202.2, 3005, 3010, 6010 | 16 | 81-113 |
| Aluminum, Solid | mg/kg | EPA 3050, 7020, 6010[a] | 21[e] | 75-117[e] |
| Aluminum, Solid | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Antimony, Total[j] | µg/L | EPA 204.2, 3020, 7041 | 25[d] | 75-125[d] |
| Antimony, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 15 | 79-109 |
| Antimony, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Antimony, Solid[j] | mg/kg | EPA 3050, 6010 | 15 | 79-109 |
| Antimony, Solid[j] | mg/kg | EPA 3050, 7041 | 25[d] | 75-125[d] |
| Antimony, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Arsenic, Total[j] | µg/L | EPA 206.2, 3005, 3010, 6010[a], 3020, 7060 | 24 | 72-120 |
| Arsenic, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Arsenic, Solid[j] | mg/kg | EPA 3050, 7060, 6010[a] | 24 | 72-120 |
| Arsenic, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Barium, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Barium, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 10 | 86-106 |
| Barium, Solid[j] | mg/kg | EPA 3050, 6010 | 10 | 86-106 |
| Barium, Solid[j] | mg/kg | EPA 3051, 6010 | 25[d] | 75-125[d] |
| Beryllium, Total[j] | µg/L | EPA 210.2, 3020, 7091 | 25[d] | 75-125[d] |
| Beryllium, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 15 | 78-108 |
| Beryllium, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Beryllium, Solid[j] | mg/kg | EPA 3050, 6010 | 15 | 78-108 |
| Beryllium, Solid[j] | mg/kg | EPA 3050, 7091 | 25[d] | 75-125[d] |
| Beryllium, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Cadmium, Total[j] | µg/L | EPA 213.2, 3020, 7131 | 25[d] | 75-125[d] |
| Cadmium, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 14 | 80-108 |
| Cadmium, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Cadmium, Solid[j] | mg/kg | EPA 3050, 6010 | 14 | 80-108 |
| Cadmium, Solid[j] | mg/kg | EPA 3050, 7131 | 25[d] | 75-125[d] |
| Cadmium, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Calcium, Total[j] | mg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 19 | 78-116 |
| Calcium, Solid[j] | mg/kg | EPA 3050, 6010[a] | 28[e] | 60-116[e] |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 2 of 12)

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Method Criterion[b,l] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Chromium, Total[j] | µg/L | EPA 218.1, 3020, 7191 | 16 | 80-112 |
| Chromium, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 15 | 79-109 |
| Chromium, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Chromium, Solid[j] | mg/kg | EPA 3050, 6010 | 15 | 79-109 |
| Chromium, Solid[j] | mg/kg | EPA 3050, 7191 | 16 | 80-112 |
| Chromium, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Cobalt, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Cobalt, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 10 | 85-105 |
| Cobalt, Solid[j] | mg/kg | EPA 3050, 6010 | 10 | 85-105 |
| Cobalt, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Copper, Total[j] | µg/L | EPA 220.1, 220.2, 3020, 7210 | 25[d] | 75-125[d] |
| Copper, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 12 | 84-108 |
| Copper, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Copper, Solid[j] | mg/kg | EPA 3050, 6010 | 12 | 84-108 |
| Copper, Solid[j] | mg/kg | EPA 3050, 7210 | 25 | 75-125 |
| Copper, Solid[j] | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |
| Iron, Total | µg/L | EPA 200.7, 3005, 3010, 6010 | 18 | 77-113 |
| Iron, Solid | mg/kg | EPA 3050, 6010[a] | 11[e] | 86-108[e] |
| Lead, Total[j] | µg/L | EPA 239.2, 3020, 7421 | 27 | 71-125 |
| Lead, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 15 | 79-109 |
| Lead, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Lead, Solid[j] | mg/kg | EPA 3050, 6010, | 15 | 79-109 |
| Lead, Solid[j] | mg/kg | EPA 3050, 7421 | 27 | 71-125 |
| Lead, Solid[j] | mg/kg | EPA 3051, 6020, | 25[d] | 75-125[d] |
| Magnesium, Total | mg/L | EPA 200.7[a], 242.1, 3005, 3010, 6010[a] | 10 | 86-106 |
| Magnesium, Solid | mg/kg | EPA 3050, 7450, 6010[a] | 53[e] | 28-134[e] |
| Manganese, Total | µg/L | EPA 243.2 | 25[d] | 75-125[d] |
| Manganese, Total | µg/L | EPA 200.7, 3005, 3010, 6010 | 12 | 83-107 |
| Manganese, Total | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Manganese, Solid | mg/kg | EPA 3050, 6010 | 12 | 83-107 |
| Manganese, Solid | mg/kg | EPA 3050, 7460 | 25[d] | 75-125[d] |
| Manganese, Solid | mg/kg | EPA 3051, 6020 | 25[d] | 75-125[d] |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 3 of 12)

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Method Criterion[b,l] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Mercury, Total[j] | µg/L | EPA 245.1, 7470 | 21 | 83-125 |
| Mercury, Solid[j] | mg/kg | EPA 7471 | 21 | 83-125 |
| Mercury, Solid[j] | mg/kg | EPA 7471 (Modified) | 21 | 83-125 |
| Molybdenum, Total | µg/L | EPA 200.7[a], 246.2, 3005, 3010, 6010[a] | 25[d] | 75-125[d] |
| Molybdenum, Solid | mg/kg | EPA 3050, 6010[a], 7480, 7481 | 25[d] | 75-125[d] |
| Nickel, Total | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Nickel, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 14 | 78-106 |
| Nickel, Solid[j] | mg/kg | EPA 3050, 6010[a] | 14 | 78-106 |
| Nickel, Solid[j] | mg/kg | EPA 3051, 6020[a] | 25[d] | 75-125[d] |
| Potassium, Total | mg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 19 | 75-113 |
| Potassium, Solid | mg/kg | EPA 3050, 6010[a] | 19 | 75-113 |
| Potassium, Solid | mg/kg | EPA 3050, 7610 | 14 | 81-109 |
| Selenium, Total[j] | µg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 12 | 85-109 |
| Selenium, Total[j] | µg/L | EPA 270.2, 3020, 7740 | 29 | 71-129 |
| Selenium, Solid[j] | mg/kg | EPA 3050, 7740 | 29 | 71-129 |
| Selenium, Solid[j] | mg/kg | EPA 3050, 6010[a] | 12 | 85-109 |
| Silicon, Total | µg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 25 | 75-125 |
| Silicon, Solid | mg/kg | EPA 3050, 6010[a] | 25 | 75-125 |
| Silver, Total | µg/L | EPA 272.2 | 24 | 68-116 |
| Silver, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 17 | 73-107 |
| Silver, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Silver, Solid[j] | mg/kg | EPA 3050, 6010 | 17 | 73-107 |
| Silver, Solid[j] | mg/kg | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Sodium, Total | mg/L | EPA 273.1 | 25[d] | 75-125[d] |
| Sodium, Total | mg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 15 | 82-112 |
| Sodium, Solid | mg/kg | EPA 3050, 6010[a], 7770 | 51[e] | 29-131[e] |
| Strontium, Total | µg/L | EPA 200.7[a], 3005, 3010, 6010[a,c] | 12[f] | 95-115[f] |
| Strontium, Solid | mg/kg | EPA 3050, 6010[a] | N/A | N/A |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 4 of 12)

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Method Criterion[b,l] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Thallium, Total[j] | µg/L | EPA 279.2, 3020, 7841 | 27 | 74-128 |
| Thallium, Total[j] | µg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 18 | 75-111 |
| Thallium, Total[j] | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Thallium, Solid[j] | mg/kg | EPA 3050, 6010[a] | 18 | 75-111 |
| Thallium, Solid[j] | mg/kg | EPA 3050, 7841 | 27 | 74-128 |
| Thallium, Solid[j] | mg/kg | EPA 3051, 6020[a] | 25[d] | 75-125[d] |
| Titanium, Total | µg/L | EPA 200.7[a], 283.2[a], 3005, 3010, 6010[a,c] | 24 | 68-116 |
| Titanium, Solid | mg/kg | EPA 3050, 6010[a] | 24 | 68-116 |
| Tin, Total[j] | µg/L | EPA 200.7[a], 3005, 3010, 6010[a] | 25 | 75-125 |
| Tin, Solid[j] | mg/kg | EPA 3050, 6010[a] | 25 | 75-125 |
| Uranium, Total | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Uranium, Solid | mg/kg | EPA 3051, 6020[a] | 25[d] | 75-125[d] |
| Vanadium, Total[j] | µg/L | EPA 286.2, 3020, 7911 | 25[d] | 75-125[d] |
| Vanadium, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 9 | 87-105 |
| Vanadium, Solid[j] | mg/kg | EPA 3050, 6010 | 9 | 87-105 |
| Vanadium, Solid[j] | mg/kg | EPA 3050, 7911 | 25[d] | 75-125[d] |
| Zinc, Total | µg/L | EPA 3015, 6020 | 25[d] | 75-125[d] |
| Zinc, Total[j] | µg/L | EPA 200.7, 3005, 3010, 6010 | 18 | 76-112 |
| Zinc, Solid[j] | mg/kg | EPA 3050, 6010 | 18 | 76-112 |
| Zinc, Solid[j] | mg/kg | EPA 3050, 6010 | 25[d] | 75-125[d] |
| Alkalinity, Total | mg/L-$CaCO_3$ | EPA 310.1 | 13 | 86-112 |
| COD, high-level | mg/L | HACH 8000 | 10 | 93-113 |
| COD, low-level | mg/L | HACH 8000 | 18 | 82-118 |
| Cyanide[j] | mg/L | EPA 335.3, 9010 | 26 | 71-123 |
| Cyanide, Solid[j] | µg/g | EPA 9010 (Mod) | 22 | 75-119 |
| Moisture | % Wet Weight | ASTMD 2216-71 | 23 | NA |
| Bromide | mg/L | EPA 300, 9056 | 6 | 95-107 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 5 of 12)

| Parameter | Units | Reference | Method Criterion[b] | |
|---|---|---|---|---|
| | | | Precision (Max RPD) | Accuracy (Percent Recovery) |
| Chloride | mg/L | EPA 325.3 | 12 | 88-112 |
| Chloride | mg/L | EPA 300, 9056 | 8 | 92-108 |
| Chloride | mg/kg | EPA 9056 | 8 | 93-109 |
| Nitrogen, $NO_2$ + $NO_3$ | mg/L-as N | EPA 353.2 | 10 | 88-108 |
| Nitrogen, $NO_2$ + $NO_3$ | mg/kg-as N | EPA 353.2 (Modified) | 10 | 87-107 |
| Nitrogen, $NO_3$[h] | mg/L-as N | EPA 353.2, 9200 | 5 | 95-105 |
| Nitrogen, $NO_3$ | mg/L-as N | EPA 300, 9056 | 9 | 92-110 |
| Nitrogen, $NO_2$ | mg/L-as N | EPA 353.2 | 8 | 93-109 |
| Nitrogen, $NO_2$ | mg/L-as N | EPA 300, 9056 | 8 | 93-109 |
| Nitrogen, $NH_3$ + $NH_4$ | mg/L-as N | EPA 350.1 | 26 | 76-128 |
| Nitrogen, TKN | mg/L-as N | EPA 351.2 | 23 | 78-124 |
| Nitrogen, TKN, Solid | mg/kg | CE-81-1, p. 3-201, Method 1 | 40 | 57-137 |
| Phosphorus, T | mg/L-as P | EPA 365.1 | 12 | 88-112 |
| Phosphorus, Ortho | mg/L-as P | EPA 300, 9056 | 13 | 86-112 |
| Phosphorus, Ortho | mg/L-as P | EPA 365.1 | 10 | 90-110 |
| Silica | mg/L | EPA 370.1 | 10 | 91-111 |
| Sulfate | mg/L | EPA 300, 9056 | 7 | 93-107 |
| Sulfate | mg/L | EPA 375.4 | 13 | 87-113 |
| Sulfide[j] | mg/L | EPA 376.2, 9030 | 33 | 60-126 |
| Sulfide, Solid[j] | mg/kg | EPA 9030 | 33 | 60-126 |
| Acidity, Total | mg/L-$CaCO_3$ | EPA 305.1 | 8 | 92-108 |
| BOD, 5-day | mg/L | EPA 405.1 | 40 | 63-143 |
| BOD, 14-day | mg/L | EPA 405.1 | 40 | 63-143 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 6 of 12)

| Parameter | Units | Reference | Method Criterion[b] Precision (Max RPD) | Method Criterion[b] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Carbon, Total | mg/L | EPA 415.1, 9060 | 13 | 87-113 |
| Carbon, TOC | mg/L | EPA 415.1, 9060 | 13 | 87-113 |
| Carbon, TOC, Solid | g/kg | EPA 9060 (mod) | 17 | 82-116 |
| Carbon, TOC, Solid | % Organic Content | ASTM-D 2974[i] | 20 | NA |
| Chromium (+6) | µg/L | EPA 7196 | 15 | 83-113 |
| Chromium (+6), Solid | mg/kg | EPA 3060, 7196 (Mod) | 15 | 83-113 |
| Color, True | PCU | EPA 110.2 | NA | NA |
| Corrosivity | mm/yr | EPA 1110, SM 2330 | NA | NA |
| Dissolved Oxygen (DO) | mg/L | EPA 360.1 | 20 | NA |
| Fluoride | mg/L | EPA 340.2 | 17 | 81-115 |
| Fluoride | mg/L | EPA 300, 9056 | 6 | 95-107 |
| Hardness | mg/L-$CaCO_3$ | EPA 130.2 | 25 | 85-115 |
| Ignitability | °C | EPA 1010 | NA | NA |
| Odor, 25°C | Thrsh No | EPA 140.1 | NA | NA |
| Oil and Grease, Grav | mg/L | EPA 413.1 | 20 | 79-119 |
| Oil and Grease, IR | mg/L | EPA 9073[g, 413.2] | 37 | 54-128 |
| Oil and Grease, IR, Solid | µg/g | EPA 9071, 9073[g] | 22 | 78-122 |
| pH | Std Units | EPA 150.1, 9040 | 4 | NA |
| pH, Solid | Std Units | EPA 9045 | 8 | NA |
| Phenols | µg/L | EPA 420.2, 9066 | 20 | 73-112 |
| Phenols, Solid | µg/g | CE-81-1, p. 3-555 | 25 | 72-122 |
| Residue, Susp. (TSS) | mg/L | EPA 160.2 | 34 | NA |
| Residue, Diss., Total (TDS) 105 Deg | mg/L | EPA 160.1 | 19 | NA |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 7 of 12)

| Parameter | Units | Reference | Method Criterion[b] Precision (Max RPD) | Method Criterion[b] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Residue, Total (TS) | mg/L | EPA 160.3 | 19 | NA |
| MBAS (foaming agents) | mg/L | EPA 425.1 | 18 | 75-111 |
| Petroleum hydrocarbons (TRPH) | mg/L | EPA 418.1 | 37 | 54-128 |
| Petroleum hydrocarbons, Solid | µg/g | EPA 9073[g] | 23 | 76-122 |
| Petroleum hydrocarbons, Solid | µg/g | EPA 3550, 418.1 (Mod) | 34 | 67-135 |
| Specific Conductivity | µmhos/cm | EPA 120.1, 9050 | 15 | NA |
| Temperature | °C | EPA 170.1 | NA | NA |
| TOX | µg/L-Cl | EPA 9020A | 26 | 73-125 |
| TOX, Solid | µg/kg | EPA 9020A (Mod) | 31 | 66-128 |
| Turbidity | NTU | EPA 180.1 | 16 | NA |
| TCLP | -- | EPA 1311 | NA | NA |
| Americium-241 | pCi/L | ER 120, HASL G-03 | 25 | 85-115 |
| Americium-241, Solid | pCi/g | ER 120, HASL G-03 | 25 | 85-115 |
| Lead-210 | pCi/L | EPA-EERF | 25 | 80-120 |
| Lead-210, Solid | pCi/g | EPA-EERF | 25 | 80-120 |
| Gamma, gross | pCi/L | ER 150 | 25 | 80-120 |
| Gamma, gross, Solid | pCi/g | ER 150 | 25 | 80-120 |
| Plutonium-238 | pCi/L | ER 160, HASL G-03 | 25 | 85-115 |
| Plutonium-238, Solid | pCi/g | ER 160, HASL G-03 | 25 | 85-115 |
| Plutonium-239 | pCi/L | ER 160, HASL G-03 | 25 | 85-115 |
| Plutonium-239, Solid | pCi/g | ER 160, HASL G-03 | 25 | 85-115 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 8 of 12)

| Parameter | Units | Reference | Method Criterion[b] | |
|---|---|---|---|---|
| | | | Precision (Max RPD) | Accuracy (Percent Recovery) |
| Plutonium-240 | pCi/L | ER 160, HASL G-03 | 25 | 85-115 |
| Plutonium-240, Solid | pCi/g | ER 160, HASL G-03 | 25 | 85-115 |
| Plutonium-241 | pCi/L | ER 160, HASL G-03 | 25 | 75-125 |
| Plutonium-241, Solid | pCi/g | ER 160, HASL G-03 | 25 | 75-125 |
| Thorium-234 | pCi/L | ER 130 | 25 | 85-115 |
| Thorium-234, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Thorium-227 | pCi/L | EPA-EERF | 25 | 85-115 |
| Thorium-227, Solid | pCi/g | EPA-EERF | 25 | 85-115 |
| Thorium-228 | pCi/L | EPA-EERF | 25 | 85-115 |
| Thorium-228, Solid | pCi/g | EPA-EERF | 25 | 85-115 |
| Thorium-230 | pCi/L | EPA-EERF | 25 | 85-115 |
| Thorium-230, Solid | pCi/g | EPA-EERF | 25 | 85-115 |
| Thorium-232 | pCi/L | EPA-EERF | 25 | 85-115 |
| Thorium-232, Solid | pCi/g | EPA-EERF | 25 | 85-115 |
| Strontium-90 | pCi/L | EML Sr-01 | 25 | 80-120 |
| Strontium-90, Solid | pCi/g | EML Sr-01 | 25 | 80-120 |
| Strontium 90 | pCi/L | EPA 905.0 | 16 | 81-113 |
| Strontium 89 | pCi/L | EPA 905.0 | 14 | 83-111 |
| Uranium-233 | pCi/L | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-233, Solid | pCi/g | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-234 | pCi/L | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-234, Solid | pCi/g | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-235 | pCi/L | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-235, Solid | pCi/g | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-236 | pCi/L | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-236, Solid | pCi/g | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-238 | pCi/L | ER 310, HASL G-03 | 25 | 85-115 |
| Uranium-238, Solid | pCi/g | ER 310, HASL G-03 | 25 | 85-115 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 9 of 12)

| Parameter | Units | Reference | Method Criterion[b] Precision (Max RPD) | Method Criterion[b] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Uranium, Natural | pCi/L | EPA 908.0 | 15 | 85-115 |
| Uranium | pCi/L | EPA 908.0 | 25 | 75-125 |
| Uranium, Total, Solid | pCi/g | EPA 3050/908.0 | 25 | 75-125 |
| Tritium | pCi/L | ER 210 | 25 | 80-120 |
| Tritium, Solid | pCi/g | ER 210 | 25 | 80-120 |
| Tritium, Total | pCi/L | EPA 906.0 | 29 | 71-129 |
| Cadmium-109 | pCi/L | ER 130 | 25 | 80-120 |
| Cadmium-109, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Cobalt-57 | pCi/L | ER 130 | 25 | 80-120 |
| Cobalt-57, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Cerium-139 | pCi/L | ER 130 | 25 | 80-120 |
| Cerium-139, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Yttrium-88 | pCi/L | ER 130 | 25 | 80-120 |
| Yttrium-88, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Cobalt-60 | pCi/L | ER 130 | 25 | 80-120 |
| Cobalt-60, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Cesium-137 | pCi/L | ER 130 | 25 | 85-115 |
| Cesium-137, Solid | pCi/g | ER 130 | 25 | 80-120 |
| Neptunium-237 | pCi/L | EPA 907.0 | 25 | 85-115 |
| Neptunium-237, Solid | pCi/g | EPA 3050(Mod),907.0 | 25 | 85-115 |
| Polonium-210 | pCi/L | HASL Po-01 | 25 | 85-115 |
| Polonium-210, Solid | pCi/g | HASL Po-01 | 25 | 85-115 |
| Technetium-99 | pCi/L | HASL Tc-01 | 25 | 70-130 |
| Technetium-99, Solid | pCi/g | HASL Tc-01 | 25 | 70-130 |
| Alpha, Gross | pCi/L | EPA 900.0, 9310 | 38 | 58-134 |
| Alpha, Gross, Solid | pCi/g | EPA 3050 (Mod), 9310 | 31 | 67-129 |
| Beta, Gross | pCi/L | EPA 900.0, 9310 | 36 | 58-130 |
| Beta, Gross, Solid | pCi/g | EPA 3050 (Mod), 9310 | 36 | 58-130 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 10 of 12)

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Method Criterion[b,l] Accuracy (Percent Recovery) |
|---|---|---|---|---|
| Radium Alpha, Gross | pCi/L | EPA 900.1 | 25 | 75-125 |
| Radium, 226 | pCi/L | EPA 903.1, 9320 | 25 | 72-122 |
| Radium, 226, Alpha emit | pCi/L | EPA 903.0, 9315 | 17 | 83-117 |
| Radium, 226, Solid | pCi/g | EPA 3050 (Mod), 9320 | 29 | 70-128 |
| Radium, 226, Alpha emit, Solid | pCi/g | EPA 3050 (Mod), 9315 | 17 | 83-117 |
| Radium 228 | pCi/L | Brooks and Blanchard | 25 | 75-125 |
| Radium 228 | pCi/L | EPA 904.0, 9320 | 15 | 84-114 |
| Radium 228, Solid | pCi/g | EPA 3050 (Mod), 9320 | 15 | 84-114 |
| Radium, Total | pCi/L | EPA 903.0, 9315 | 45[k] | 55-145[k] |
| Actinium-227 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Actinium-227, Solid | pCi/g | ER 1301 | 25 | 85-115 |
| Actinium-228 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Actinium-228, Solid | pCi/g | ER 1301 | 25 | 85-115 |
| Beryllium-7 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Beryllium-7, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Bismuth-214 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Bismuth-214, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Lead-212 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Lead-212, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Lead-214 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Lead-214, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Palladium-234 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Palladium-234, Solid | pCi/g | ER 130 | 25 | 85-115 |
| Potassium-40 | pCi/L | ER 130, EPA 901.1 | 25 | 85-115 |
| Potassium-40, Solid | pCi/L | ER 130 | 25 | 85-115 |

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 11 of 12)

| Parameter | Units | Reference | Method Criterion[b,l] Precision (Max RPD) | Accuracy (Percent Recovery) | |
|---|---|---|---|---|---|
| Gamma Emitters* | pCi/L | ER-130, EPA 901.1 | | 25 | NA |
| Gamma Emitters* | pCi/g | ER-130, | | 25 | NA |

*The gamma emitters are Barium-133 (Ba-133), Beryllium-7 (Be-7), Potassium-40 (K-40), Cesuim-134 (Cs-134), Cesium-137 (Cs-137), Cobalt-60 (Co-60), Lead-212 (Pb-212), Lead-214 (Pb-214), Bismuth-214 (Bi-214), Actinium-227 (Ac-227), Actinium-228 (Ac-228), Palladium-234 (Pa-234), Thorium-234 (Th-234), Ruthenium-106 (Ru-106), and Zinc-65 (Zn-65).

Note:

CLP = EPA Contract Laboratory Program.
MAS = million asbestos structures.
NA = not applicable.
N/A = spiking and recovery information are not available.
SOW = statement of work.
TCLP = toxicity characteristics leaching procedure.
TOX = total organic halides.
TRPH = total recoverable petroleum hydrocarbons.

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, TOX, and Radiochemical Analysis (Continued, Page 12 of 12)

References:

ASTM D2974--American Society for Testing and Materials Designation: D2974-87, July 1987.
EPA 100-400--Methods for Chemical Analyses of Water and Waste. EPA 600/4-79-20--Revised March 1983.
EPA 900--Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA 600/4-80-032, August 1980.
EPA 1310-9073--Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition (Method 9073, draft 1989: oil and grease methods exclude 7.8 and 7.10).
CE-81-1--Procedures for Handling and Chemical Analysis of Sediment and Water Samples, EPA/USACE, May 1981.
EPA CLM--Chemistry Laboratory Manual of Bottom Sediments, PB-215192, Federal Water Quality Administration, December 1969.
Hach 8000--Hach Handbook of Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
EPA 600 M4-82-020--Interim Method for Determination of Asbestos in Bulk Insulation Samples.
NIOSH--National Institute of Occupational Safety and Health (NIOSH) Manual of Analytical Methods, 3rd Edition, Volume 1, 1984.
SM 4500-N--Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.
EML Sr-01 = Environmental Measurements Laboratory- Health and Safety Laboratory - 300 (HASL-300), 27th Edition.
EPA-EERF = Eastern Environmental Radiation Facility Radiochemistry Procedures Manual (EPA 520/5-84-006).
ER 100-310 = Environmental Analytical Procedures- Radiochemical (ER), Los Alamos-10300-Manual, Health and Environmental Chemistry; Analytical Techniques, Data Management, and Quality Assurance.
HASL G-03 = Health and Safety Laboratory Manual, Environmental Measurements Laboratory, Department of Energy, New York, New York.

[a]The parameter may be analyzed by the referenced methodology, but it is not a control parameter for this referenced method.
[b]ESE historical data.
[c]SOP derived from referenced method is used for this unlisted analyte.
[d]Criteria from EPA CLP SOW 3/90.
[e]Data from ESE method certification.
[f]ESE performance evaluation data for EPA's Water Supply Studies.
[g]EPA Method 9073, draft method, EPA 1989, for oil and grease exclude steps 7.8 and 7.10.
[h]$NO_3$ (as N) by EPA 353.2 is calculation of $(NO_2 + NO_3) - (NO_2)$; also, method criteria do not apply.
[i]Percent organic carbon is calculated from the percent organic content data obtained from the ASTMD method using the equation: % Organic Carbon = $\frac{\%\ \text{Total Organic Content}}{1.724}$
[j]Appendix IX compounds.
[k]Criteria from EPA Method 903.0--Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA 600/4-80-032, August 1980.
[l]CLP precision and accuracy acceptance criteria for metals will be used for Los Alamos projects.

Source: ESE.

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses

| Parameter | Reference | Reporting Limit Aqueous* (µg/L) | Reporting Limit Solid* (mg/kg) |
|---|---|---|---|
| Aluminum | EPA 200.7, 3005, 3010, 3050, 6010 | 50 | 10 |
| Aluminum | EPA 202.2, 3050, 7020 | 5.0** | 0.5** |
| Aluminum | EPA 3015, 3051, 6020 | 25 | 10 |
| Antimony | EPA 200.7, 3005, 3010, 3050, 6010 | 50 | 5.0 |
| Antimony | EPA 204.2, 3050, 7041 | 3.0** | 0.5** |
| Antimony | EPA 3015, 3051, 6020 | 0.5 | 0.005 |
| Arsenic | EPA 200.7, 3005, 3010, 3050, 6010 | 100 | 10 |
| Arsenic | EPA 206.2, 3020, 3050, 7060 | 2.5 | 0.25 |
| Arsenic | EPA 3015, 3051, 6020 | 1.0 | 0.10 |
| Barium | EPA 200.7, 3005, 3010, 3050, 6010 | 20 | 2.0 |
| Barium | EPA 3015, 3051, 6020 | 0.5 | 0.20 |
| Beryllium | EPA 200.7, 3015, 3010, 3050, 6010 | 5.0 | 0.5 |
| Beryllium | EPA 210.2, 3020, 3050, 7091 | 0.20 | 0.02 |
| Beryllium | EPA 3015, 3051, 6020 | 0.20 | 0.02 |
| Cadmium | EPA 200.7, 3005, 3010, 3050, 6010 | 3.9 | 0.39 |
| Cadmium | EPA 213.2, 3050, 7131 | 0.20 | 0.02 |
| Cadmium | EPA 3015, 3051, 6020 | 0.20 | 0.05 |
| Calcium | EPA 200.7, 3005, 3010, 3050, 6010 | 100 | 75 |
| Chromium | EPA 200.7, 3005, 3010, 3050, 6010 | 10 | 1.0 |
| Chromium | EPA 218.2 | 2.5 | 0.5 |
| Chromium | EPA 3015, 3051, 6020 | 1.0 | 0.10 |
| Cobalt | EPA 200.7, 3005, 3010, 3050, 6010 | 20 | 2.0 |
| Cobalt | EPA 3015, 3051, 6020 | 0.5 | 0.20 |
| Copper | EPA 200.7, 3005, 3010, 3050, 6010 | 5 | 0.5 |
| Copper | EPA 3015, 3051, 6020 | 0.5 | 0.10 |
| Iron | EPA 200.7, 3005, 3010, 3050, 6010 | 45 | 25 |
| Lead | EPA 200.7, 3005, 3010, 3050, 6010 | 50 | 10 |
| Lead | EPA 239.2, 3020, 3050, 7421 | 2.0 | 0.5 |
| Lead | EPA 3015, 3051, 6020 | 0.5 | 0.10 |
| Magnesium | EPA 200.7, 3005, 3010, 3050, 6010 | 50 | 25 |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 2 of 8)

| Parameter | Reference | Reporting Limit Aqueous* (µg/L) | Reporting Limit Solid* (mg/kg) |
|---|---|---|---|
| Manganese | EPA 200.7, 3005, 3010, 3050, 6010 | 5.0 | 2.5 |
| Manganese | EPA 3015, 3051, 6020 | 0.5 | 5.0 |
| Mercury | EPA 245.1, 7470, 7471 | 0.2 | 0.1 |
| Mercury, Low Level | EPA 245.1, 7471 | 0.01 | 0.001 |
| Molybdenum | EPA 200.7, 3005, 3010, 3050, 6010 | 10 | 1.0 |
| Molybdenum | EPA 3050, 7480, 7481 | 5.0** | 0.5** |
| Nickel | EPA 200.7, 3005, 3010, 3050, 6010 | 15 | 2.0 |
| Nickel | EPA 3015, 3051, 6020 | 1.0 | 0.10 |
| Potassium | EPA 200.7, 3005, 3010, 3050, 6010 | 600 | 100 |
| Potassium | EPA 3010, 3050, 7610 | 5.0** | 0.5** |
| Selenium | EPA 270.2, 6010, 200.7, 3005, 3010, 3050 | 100 | 10 |
| Selenium | EPA 200.7, 3050, 7740 | 2.5 | 0.25 |
| Silicon | EPA 200.7, 3005, 3010, 3050, 6010 | 200 | 20 |
| Silver | EPA 200.7, 3005, 3010, 3050, 6010 | 5.0 | 0.50 |
| Silver | EPA 272.2 | 0.25 | 0.05 |
| Silver | EPA 6020 | 0.1 | 0.02 |
| Sodium | EPA 200.7, 3005, 3010, 3050, 6010 | 100 | 50 |
| Sodium | EPA 273.1, 3050, 7770 | 100** | 10.0** |
| Strontium | EPA 200.7, 6010, 3005, 3010, 3050 | 2.5 | 2.0 |
| Thallium | EPA 200.7, 3005, 3010, 3050, 6010 | 200 | 20 |
| Thallium | EPA 279.2, 3050, 7841 | 5.0 | 0.5 |
| Thallium | EPA 3015, 3051, 6020 | 0.5 | 0.10 |
| Tin | EPA 200.7, 3010, 3050, 6010 | 50 | 5 |
| Titanium | EPA 200.7, 3005, 3010, 6010 | 10 | 2.0 |
| Uranium | EPA 3015, 3051, 6020 | 0.1 | 0.1 |
| Vanadium | EPA 200.7, 3005, 3010, 3050, 6010 | 10 | 1.0 |
| Vanadium | EPA 286.2, 3050, 7911 | 5.0 | 0.5 |
| Zinc | EPA 200.7, 3005, 3010, 3050, 6010 | 30 | 5.0 |
| Zinc | EPA 3015, 3051, 6020 | 1.0 | 0.20 |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 3 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Alkalinity, Total | mg/L-$CACO_3$ | EPA 310.1 | 5.0*** |
| COD, high-level | mg/L | HACH 8000 | 50** |
| COD, low-level | mg/L | HACH 8000 | 5.0** |
| Cyanide | mg/L | EPA 335.3 | 0.005** |
| Cyanide, Solid | µg/g | EPA 335.3. 9010 | 0.25** |
| Moisture | % Wet Wt | ASTM-D 2216-71 | 0.5** |
| Bromide | mg/L | EPA 9056 | 0.2** |
| Bromide | mg/L | EPA 300 | 1.0** |
| Chloride | mg/L | EPA 325.3 | 1.0*** |
| Chloride | mg/L | EPA 300, 9056 | 0.5** |
| Nitrogen, $NO_2$ + $NO_3$ | mg/L-as N | EPA 353.2 | 0.010** |
| Nitrogen, $NO_3$ | mg/L-as N | EPA 300, 353.2, 9056 | 0.010** |
| Nitrogen, $NO_2$ | mg/L-as N | EPA 300, 353.2, 9056 | 0.010** |
| Nitrogen, $NH_3$ + $NH_4$ | mg/L-as N | EPA 350.1 | 0.05** |
| Nitrogen, TKN | mg/L-as N | EPA 351.2 | 0.10** |
| Nitrogen, TKN, Solid | mg/kg | CE-81-1, p. 3-201, Method 1 | 10** |
| Phosphorus, T | mg/L-as P | EPA 300, 9056 | 0.01** |
| Phosphorus, T | mg/L-as P | EPA 365.1 | 0.01** |
| Phosphorus, T Solid | mg/kg-as P | EPA 365.1 | 2.5** |
| Phosphorus, Ortho, D | mg/L-as P | EPA 365.1 | 0.01** |
| Phosphorus, Ortho, D | mg/L-as P | EPA 300, 9056 | 0.01** |
| Total O-$PO_4$ | mg/L-as P | EPA 365.1 | 0.01** |
| Silica, Diss | mg/L | EPA 370.1 | 2.0** |
| Sulfate | mg/L | EPA 375.4 | 5.0** |
| Sulfate | mg/L | EPA 300, 9056 | 0.50** |
| Sulfide | mg/L | EPA 376.2 | 0.05** |
| Sulfide, Solid | mg.kg | EPA 9030 | 0.25** |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 4 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Acidity, Total | mg/L-$CaCO_3$ | EPA 305.1 | 5** |
| BOD, 5-day | mg/L | EPA 405.1 | 1.0** |
| BOD, 14-day | mg/L | EPA 405.1 | 1.0*** |
| Carbon, Total | mg/L | EPA 415.1, 9060 | 1.0** |
| Carbon, TOC | mg/L | EPA 415.1, 9060 | 1.0** |
| Carbon, TOC, Solid | g/kg | EPA 9060 (Mod) | 360***** |
| Carbon, TOC, Solid | % Organic Content | ASTM- D 2974 | 0.1** |
| Chromium (+6) | µg/L | EPA 7196 | 2.0** |
| Chromium (+6), Solid | mg/kg | EPA 3060, 7196 (Mod) | 0.2** |
| Color, true | PCU | EPA 110.2 | 5.0** |
| Fluoride | mg/L | EPA 340.2 | 0.10** |
| Fluoride | mg/L | EPA 300, 9056 | 0.50** |
| Hardness | mg/L-$CaCO_3$ | EPA 130.2 | 1.0††† |
| Odor, 25°C | Thrsh No. | EPA 140.1 | 1.0††† |
| Corrosivity | mm/yr | EPA 1110, SM2330 | |
| Dissolved Oxygen | mg/L | EPA 160.1 | |
| Turbidity | NTU | EPA 180.1 | 0.4** |
| MBAS (foaming agents, surfactants) | mg/L | EPA 425.1 | 0.1** |
| Oil and Grease, IR | mg/L | EPA 413.2 | 0.17** |
| Oil and Grease, Gravimetric | mg/L | EPA 413.1 | 5.0** |
| Oil and Grease, IR, Solid | µg/g | EPA 9071, 9073 | 21** |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 5 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Phenols | µg/L | EPA 420.2, 9056 | 5.0** |
| Phenols, Solid | µg/g | CE-81-1, p. 3-555 | 100** |
| Residue, Diss., Total (TDS) 105 Deg | mg/L | EPA 160.1 | 10*** |
| Residue, Susp. (TSS) | mg/L | EPA 160.2 | 4*** |
| Residue, Total (TS) | mg/L | EPA 160.3 | 10** |
| Petroleum Hydrocarbons (TRPH) | mg/L | EPA 418.1 | 0.17** |
| Petroleum Hydrocarbons, Solid | µg/g | EPA 9071, 9073 | 21** |
| Petroleum Hydrocarbons, Solid | µg/g | EPA 3550, 418.1 | 21** |
| Specific Conductivity | µmho/cm | EPA 120.1 | 10** |
| TOX | µg/L-Cl | EPA 9020A | 10** |
| TOX, Solid | µg/kg | EPA 9020A (Mod) | 30** |
| Americium-241 | pCi/L | EPA 120, AM-03 | 0.1 |
| Americium-241, Solid | pCi/g | EPA 120, AM-03 | 0.01 |
| Lead-210 | pCi/L | EPA-EERF | 1.0 |
| Lead-210, Solid | pCi/g | EPA-EERF | 1.0 |
| Gamma, gross | pCi/L | EPA 150 | 100 |
| Gamma, gross, Solid | pCi/g | EPA 150 | 2 |
| Plutonium-238 | pCi/L | ER 160 | 0.1 |
| Plutonium-238, Solid | pCi/g | ER 160 | 0.01 |
| Plutonium-239 | pCi/L | ER 160 | 0.1 |
| Plutonium-239, Solid | pCi/g | ER 160 | 0.01 |
| Plutonium-240 | pCi/L | ER 160 | 0.1 |
| Plutonium-240, Solid | pCi/g | ER 160 | 0.01 |
| Plutonium-241 | pCi/L | ER 160 | 2.0 |
| Plutonium-241 Solid | pCi/g | ER 160 | 1.0 |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 6 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Thorium-234 | pCi/L | ER 130 | 20 |
| Thorium-234, Solid | pCi/g | ER 130 | 1.0 |
| Thorium-227 | pCi/L | EPA-EERF | 0.1 |
| Thorium-227, Solid | pCi/g | EPA-EERF | 0.1 |
| Thorium-228 | pCi/L | EPA-EERF | 0.1 |
| Thorium-228, Solid | pCi/g | EPA-EERF | 0.1 |
| Thorium-230 | pCi/L | EPA-EERF | 0.1 |
| Thorium-230, Solid | pCi/g | EPA-EERF | 0.1 |
| Thorium-232 | pCi/L | EPA-EERF | 0.1 |
| Thorium-232, Solid | pCi/g | EPA-EERF | 0.1 |
| Strontium-90 | pCi/L | EML Sr-01 | 1.0 |
| Strontium-90, Solid | pCi/g | EML Sr-01 | 0.5 |
| Strontium 90 | pCi/L | EPA 905.0 | 2.0**** |
| Strontium 89 | pCi/L | EPA 905.0 | 2.0**** |
| Uranium-233 | pCi/L | ER 310 | 0.1 |
| Uranium-233, Solid | pCi/g | ER 310 | 0.1 |
| Uranium-234 | pCi/L | ER 310 | 0.1 |
| Uranium-234, Solid | pCi/g | ER 310 | 0.1 |
| Uranium-235 | pCi/L | ER 310 | 0.1 |
| Uranium-235, Solid | pCi/g | ER 310 | 0.1 |
| Uranium-236 | pCi/L | ER 310 | 0.1 |
| Uranium-236, Solid | pCi/g | ER 310 | 0.1 |
| Uranium-238 | pCi/L | ER 310 | 0.1 |
| Uranium-238, Solid | pCi/g | ER 310 | 0.1 |
| Uranium, Natural | pCi/L | EPA 908.0 | 1.0 |
| Uranium, Total | pCi/L | EPA 908.0 | 1.0 |
| Uranium, Total, Solid | pCi/g | EPA 3050/908.0 | 0.3 |
| Tritium | pCi/L | ER 210 | 300 |
| Tritium, Solid | pCi/g | ER 210 | 300 |
| Tritium | pCi/L | EPA 906.0 | 600 |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 7 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Cadmium-109 | pCi/L | ER 130 | 421 |
| Cadmium-109, Solid | pCi/g | ER 130 | 14 |
| Cobalt-57 | pCi/L | ER 130 | 12 |
| Cobalt-57, Solid | pCi/g | ER 130 | 0.5 |
| Cerium-139 | pCi/L | ER 130 | 10 |
| Cerium-139, Solid | pCi/g | ER 130 | 0.4 |
| Yttrium-88 | pCi/L | ER 130 | 21 |
| Yttrium-88, Solid | pCi/g | ER 130 | 0.9 |
| Cobalt-60 | pCi/L | ER 130 | 22 |
| Cobalt-60, Solid | pCi/g | ER 130 | 0.9 |
| Cesium-137 | pCi/L | ER 130 | 3.0 |
| Cesium-137, Solid | pCi/g | ER 130 | 20 |
| Neptunium-237 | pCi/L | EPA 907.0 | 0.5 |
| Neptunium-237 | pCi/g | EPA 3050(Mod). 907.0 | 0.2 |
| Polonium-210 | pCi/L | HASL Po-01 | 1.0 |
| Polonium-210 | pCi/g | HASL Po-02 | 0.5 |
| Technetium-99 | pCi/L | HASL Tc-01 | 2.0 |
| Technetium-99 | pCi/g | HASL Tc-01 | 1.0 |
| Alpha, Gross | pCi/L | EPA 900.0 | 1.0++++ |
| Alpha, Gross, Solid | pCi/g | EPA 3050, 9310 (Mod) | 1.0++++ |
| Beta, Gross | pCi/L | EPA 900.0, 9310 | 3.0++++ |
| Beta, Gross, Solid | pCi/g | EPA 3050, 9310 (Mod) | 3.0++++ |
| Radium Alpha, Gross | pCi/L | EPA 900.1 | 1.0++++ |
| Radium 226 | pCi/L | EPA 903.1, 903.0, 9315, 9320 | 0.1++++ |
| Radium 226, Alpha emit | pCi/L | EPA 903.0, 9315 | 1.0+++ |
| Radium 226, Solid | pCi/g | EPA 3050, 9315, 9320 (Mod) | 0.1++++ |
| Radium 226, Alpha emit, Solid | pCi/g | EPA 3050, 9315 (Mod) | 1.0+++ |
| Radium 228 | pCi/L | Brooks and Blanchard | |
| Radium 228 | pCi/L | EPA 904.0, 9320 | 2.0++++ |
| Radium 228, Solid | pCi/g | EPA 3050, 9320 (Mod) | 2.0++++ |

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, TOX, and Radiochemical Analyses (Continued, Page 8 of 8)

| Parameter | Units | Reference | Reporting Limit |
|---|---|---|---|
| Radium, Total | pCi/L | EPA 903.0, 9315 | 1.0†††† |
| Gamma Emitters | | | |
| Beryllium-7 | pCi/L | ER 130, EPA 901.1 | 20 |
| Beryllium-7 | pCi/g | ER 130 | 140 |
| Potassium-40 | pCi/L | ER 130, EPA 901.1 | 43 |
| Potassium-40 | pCi/g | ER 130 | 300 |
| Lead-212 | pCi/L | ER 130, EPA 901.1 | 7.0 |
| Lead-212 | pCi/g | ER 130 | 50 |
| Lead-214 | pCi/L | ER 130, EPA 901.1 | 7.0 |
| Lead 214 | pCi/g | ER 130 | 50 |
| Cesium-137 | pCi/L | ER 130, EPA 901.1 | 3.0 |
| Cesium-137 | pCi.g | ER 130 | 20 |
| Bismuth-214 | pCi/L | ER 130, EPA 901.1 | 8.0 |
| Bismuth-214 | pCi/g | ER 130 | 55 |
| Actinium-227 | pCi/L | ER 130, EPA 901.1 | 25 |
| Actinium-227 | pCi/g | ER 130 | 175 |
| Actinium-228 | pCi/L | ER 130, EPA 901.1 | 10 |
| Actinium-228 | pCi/g | ER 130 | 70 |
| Palladium-234 | pCi/L | ER 130, EPA 901.1 | 600 |
| Palladium-234 | pCi/g | ER 130 | 4200 |
| Thorium-234 | pCi/L | ER 130, EPA 901.1 | 100 |
| Thorium-234 | pCi/g | ER 130 | 700 |

Note:
µg/L = micrograms per liter.
mg/kg = milligrams per kilogram.
mg/L = milligrams per liter.
pCi/L = picocuries per liter.
pCi/g = picocuries per gram.
g/kg = grams per kilogram.
NTU = nephelometric turbidity unit.
PCU = platinum-cobalt unit.
µg/g = micrograms per gram.
µmho/cm = microhmos per centimeter.

*Based on ESE's instrument detection limit (IDL) studies unless indicated differently. The EPA Contract Laboratory Program (CLP) SOW 3/90 requirements are followed when the IDL studies are conducted.
†Based on aqueous IDL studies times a factor of 0.1 to take into account sample weight and final volume of digestate, unless indicated differently.

** Based on the lowest standard that ESE routinely uses. For solids, the reporting limits are adjusted for sample weight and final volume.

†† HACH instrument recommended detection limit, HACH Co., Box 389, Loveland, CO 80537.

††† Methods for Chemical Analyses of Water and Waste, EPA 600/4-79-020, Revised, March 1983.

†††† Obtained from Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989. The reporting limits for petroleum hydrocarbon and oil and grease already take into account sample volume or sample weight and final extract volume.

***** Based on one-half of the major increments of the titration.

††††† Based on EPA's detection limits calculation procedure recommended for radiochemical analyses. (Reference: Carbon-14 in Aqueous Samples, Environmental Measurements Laboratory Manual, 1981.)

***** Based on MDL Study.

Source: ESE.

Table 5-4. Analytes, Precision, and Accuracy Data For Trihalomethanes, EPA 501.2

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Chloroform | 33 | 77-143 |
| Bromodichloromethane | 33 | 79-137 |
| Dibromochloromethane | 33 | 23-125 |
| Bromoform | 33 | 43-106 |
| THMs, total* | 33† | 62-128† |

Reference: Accuracy: EPA Method 501.2--The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction, EPA, Environmental Monitoring and Support Laboratory, Cincinnati, OH, May 15, 1979.

Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method EPA 501.2.

*Matrix spike and QC check sample compound.
†Accuracy and precision criteria are based on ESE historical data.

Source: ESE.

Table 5-5. Reporting Limit Data for Trihalomethanes, EPA 501.2

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Chloroform | 0.6 |
| Bromodichloromethane | 0.2 |
| Dibromochloromethane | 0.1 |
| Bromoform | 0.1 |
| THMs, total | 1.0 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume.

Source: ESE.

Table 5-6. Analytes, Precision, and Accuracy Data For EDB and DBCP, EPA 504 and Modified 504*

| Parameter | Aqueous** | | Solid** | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 1,2-Dibromoethane (EDB)† | 20 | 80-120 | 33 | 65-131 |
| DBCP (nemagon)† | 30 | 67-127 | 45 | 54-144 |

*See Appendix B for Modified Method 504.
†Matrix spike and QC check sample compound.
**Accuracy and precision criteria are based on ESE historical data.

Source: ESE.

Table 5-7. Reporting Limit Data for EDB and DBCP, EPA 504 and Modified 504

| Parameter | Reporting Limits* Aqueous (µg/L) | Solid (µg/kg) |
|---|---|---|
| 1,2-Dibromoethane (EDB)† | 0.02 | 30 |
| DBCP (nemagon)† | 0.02 | 30 |

*Based on the lowest standard that ESE routinely uses.
The solid detection limits are expressed on a wet weight basis.
†Appendix IX compounds.

Source: ESE.

Table 5-8. Analytes, Precision, and Accuracy Data For Organohalide Pesticides and Arochlors in Drinking Water, EPA 508

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Aldrin* | 29 | 57-115 |
| Chlordane, alpha | 36 | 63-135 |
| Chlordane, gamma | 36 | 63-135 |
| Chlordane | 36 | 63-135 |
| 4,4'-DDD | 19 | 88-126 |
| 4,4'-DDE | 36 | 63-135 |
| 4,4'-DDT | 50 | 62-162 |
| Dieldrin* | 26 | 61-113 |
| Endosulfan I | 26 | 61-113 |
| Endosulfan II | 30 | 62-122 |
| Endosulfan Sulfate | 46 | 56-148 |
| Endrin* | 26 | 62-114 |
| Endrin Aldehyde | 24 | 64-112 |
| BHC-alpha | 30 | 62-114 |
| BHC-beta | 20 | 75-115 |
| BHC-delta | 34 | 68-136 |
| BHC-gamma (Lindane)* | 29 | 60-118 |
| Heptachlor* | 35 | 63-133 |
| Heptachlor Epoxide | 26 | 61-113 |
| Hexachlorobenzene | 65 | 34-164 |
| Methoxychlor | 41 | 64-146 |
| Propachlor | 28 | 75-131 |
| Trifluralin | 16 | 87-119 |

Table 5-8. Analytes, Precision, and Accuracy Data For Organohalide Pesticides and Arochlors in Drinking Water, EPA 505 (Continued, Page 2 of 2)

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Toxaphene | 41 | 73-155 |
| PCB-1016 | 23 | 74-120 |
| PCB-1221 | 29 | 63-121 |
| PCB-1232 | 22 | 64-108 |
| PCB-1242 | 22 | 74-118 |
| PCB 1248 | 30 | 54-114 |
| PCB-1254 | 35 | 50-120 |
| PCB-1260 | 59 | 29-147 |

Reference: Accuracy: EPA Method 508--Supplement to "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," EPA, Cincinnati, OH, September 1986. Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (EPA 508).

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-9. Reporting Limits Data for Organohalide Pesticides and Arochlors in Drinking Water, EPA 508

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| ALdrin | 0.10 |
| Chlordane, alpha | 0.5 |
| Chlordane, gamma | 0.5 |
| Chlordane | 0.5 |
| 4,4'-DDD | 0.05 |
| 4,4-DDE | 0.01 |
| 4,4'-DDT | 0.06 |
| Dieldrin | 0.10 |
| Endosulfan I | 0.2 |
| Endosulfan II | 0.2 |
| Endosulfan Sulfate | 0.5 |
| Endrin | 0.2 |
| Endrin Aldehyde | 0.2 |
| BHC-alpha | 0.1 |
| BHC-beta | 0.1 |
| BHC-delta | 0.1 |
| BHC-gamma | 0.1 |
| Heptachlor | 0.1 |
| Heptachlor Epoxide | 0.1 |
| Hexachlorobenzene | 0.05 |
| Methoxychlor | 0.05 |
| Propachlor | 0.5 |
| Trifluralin | 0.5 |
| Toxaphene | 10 |
| PCB-1016 | 2.0 |
| PCB-1221 | 2.0 |
| PCB-1232 | 2.0 |
| PCB-1242 | 2.0 |
| PCB 1248 | 2.0 |
| PCB-1254 | 2.0 |
| PCB-1260 | 2.0 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Method detection limits listed in EPA Method 508, Analysis of Organohalide Compounds in Drinking Water (supplement to "Methods for the Determination of Organic Compounds in Drinking Water," EPA/600/4-88/039, December 1988) times ten.

Source: ESE.

Table 5-10. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Alachlor | 33 | 62-128 |
| Ametryn | 30 | 61-121 |
| Atraton | 33 | 58-124 |
| Atrazine* | 24 | 68-116 |
| Bromacil | 27 | 64-118 |
| Butachlor | 12 | 84-108 |
| Butylate | 63 | 34-160 |
| Carboxin | 12 | 90-114 |
| Chlorpropham | 33 | 60-126 |
| Cycloate | 27 | 62-116 |
| Diazinon* | 21 | 94-136 |
| Dichlorvos | 18 | 79-115 |
| Diphenamid | 24 | 69-117 |
| Disulfoton | 30 | 59-119 |
| Disulfoton Sulfone | 30 | 68-128 |
| Disulfoton Sulfoxide | 33 | 54-120 |
| EPTC | 27 | 58-112 |

Table 5-10. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507,
(Continued, Page 2 of 3)

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Ethoprop | 15 | 88-118 |
| Fenamiphos | 24 | 66-114 |
| Fenarimol | 15 | 84-114 |
| Fluridone | 27 | 60-114 |
| Hexazinone | 21 | 69-111 |
| Merphos | 24 | 72-120 |
| Methyl Parathion | 30 | 68-128 |
| Metolachlor | 12 | 81-105 |
| Metribuzin | 15 | 86-116 |
| Mevinphos | 33 | 62-128 |
| Molinate | 54 | 44-152 |
| Napropamide | 18 | 83-119 |
| Norflurazon | 15 | 79-109 |
| Pebulate | 27 | 67-121 |
| Prometon | 27 | 51-105 |
| Prometryn | 24 | 69-117 |
| Pronamide | 30 | 61-121 |

Table 5-10. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507,
(Continued, Page 3 of 3)

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Propazine | 24 | 68-116 |
| Simazine* | 21 | 79-121 |
| Simetryn | 15 | 84-114 |
| Stirofos | 18 | 80-116 |
| Tebuthiuron | 27 | 57-111 |
| Terbacil | 18 | 79-115 |
| Terbufos | 12 | 85-109 |
| Terbutryn | 27 | 67-121 |
| Triademefon | 24 | 69-117 |
| Tricyclazole | 21 | 65-107 |
| Vernolate | 18 | 75-111 |

Reference: Accuracy: EPA Method 507--Methods for the Determination of Organic Compounds in Drinking Water, EPA 600/4-88/039, December 1988.
Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (EPA 507).

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-11. Reporting Limit Data for Nitrogen and Phosphorus Containing Pesticides, EPA 507

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Alachlor | 3.8 |
| Ametryn | 20 |
| Ametraton | 6.0 |
| Atrazine | 0.6+ |
| Bromacil | 25 |
| Butachlor | 3.8 |
| Butylate | 0.3+ |
| Carboxin | 6.0 |
| Chlorpropham | 5.0 |
| Cycloate | 2.5 |
| Diazinon | 0.3+ |
| Dichlorvos | 0.3+ |
| Diphenamid | 6 |
| Disulfoton | 0.3+ |
| Disulfoton Sulfone | 38 |
| Disulfoton Sulfoxide | 3.8 |
| EPTC | 0.3 |
| Ethoprop | 1.9 |
| Fenamiphos | 10 |
| Fenarimol | 3.8 |

Table 5-11. Reporting Limit Data for Nitrogen and Phosphorus Containing Pesticides, EPA 507 (Continued, Page 2 of 3)

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Fluridone | 38 |
| Hexazinone | 1.2* |
| Merphos | 0.3* |
| Methyl Parathion | 25 |
| Metolachlor | 7.5 |
| Metribuzin | 1.5 |
| Mevinphos | 2.9 |
| Molinate | 1.5 |
| Napropamide | 2.5 |
| Norflurazon | 5 |
| Pebulate | 0.3* |
| Prometon | 3 |
| Prometryn | 1.9 |
| Pronamide | 7.6 |
| Propazine | 1.3 |
| Simazine | 0.75 |
| Simetryn | 2.5 |
| Stirofos | 0.6* |
| Tebuthiuron | 13 |
| Terbacil | 45 |

Table 5-11. Reporting Limit Data for Nitrogen and Phosphorus Containing Pesticides, EPA 507 (Continued, Page 3 of 3)

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Terbufos | 0.3* |
| Terbutryn | 2.5 |
| Triademefon | 6.5 |
| Tricyclazole | 10 |
| Vernolate | 0.3 |

*Estimated detection limits listed in EPA Method 507, (Methods for the Determination of Organic Compounds in Drinking Water, EPA 600/4-88/039, December 1988) times ten.
*Based on the lowest standard that ESE routinely uses, taking into account sample volume and final extract volume. The lowest standard is chosen to be 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-12. Analytes, Precision, and Accuracy Data For Chlorinated Herbicides, EPA 515

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| 2,4-D* | 30 | 54-120 |
| 2,4,5-TP/Silvex+der.* | 30 | 48-126 |
| Dalapon | 30 | 64-118 |
| Picloram | 30 | 20-104 |
| Dinoseb | 30 | 0-113 |
| Dicamba (banvel)* | 30 | 54-120 |
| Pentachlorophenol | 30 | 30-96 |
| 2,4-DB | 30 | 0-159 |
| 2,4,5-T | 30 | 46-124 |

Reference: Accuracy: EPA Method 515--Supplement to "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," EPA, Cincinnati, OH, September 1986.
Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the EPA 515 method.

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-13. Reporting Limit Data for Chlorinated Herbicides, EPA 515.1

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| 2,4-D | 0.029 |
| 2,4,5-TP/Silvex+der. | 0.029 |
| Dalapon | 0.029 |
| Picloram | 0.029 |
| Dinoseb | 0.029 |
| Dicamba (banvel) | 0.029 |
| Pentachlorophenol | 0.029 |
| 2,4-DB | 0.029 |
| 2,4,5-T | 0.029 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-14. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD% Recovery) | Accuracy |
| Dichlorobenzene, total | 21 | 82-124 |
| 1,1-Dichloroethane | 16 | 80-112 |
| 1,2-Dichloroethane | 16 | 79-111 |
| 1,1-Dichloroethylene* | 14 | 61-145 |
| cis-1,2-Dichloroethene | 20 | 81-121 |
| trans-1,2-Dichloroethene | 17 | 76-110 |
| 1,2-Dichloropropane | 18 | 79-115 |
| 1,3-Dichloropropane | 18 | 78-114 |
| 2,2-Dichloropropane | 51 | 35-107 |
| 1,1-Dichloropropene | 27 | 71-125 |
| Ethylbenzene | 26 | 73-125 |
| Hexachlorobutadiene | 20 | 80-120 |
| Isopropylbenzene | 23 | 78-124 |
| cis-1,3-Dichloropropene | 20 | 0-227 |
| trans-1,3-Dichloropropene | 20 | 17-183 |
| p-Cymene (4-Isopropyltoluene | 15 | 81-111 |
| Naphthalene | 25 | 79-129 |
| Methylene chloride | 16 | 79-111 |
| n-Propylbenzene | 12 | 85-109 |
| Styrene | 21 | 81-123 |
| 1,1,1,2-Tetrachloroethane | 20 | 70-110 |
| 1,1,2,2-Tetrachloroethane | 19 | 72-110 |
| Tetrachloroethene | 20 | 69-109 |
| Toluene* | 13 | 76-125 |
| 1,2,3-Trichlorobenzene | 26 | 83-135 |
| 1,2,4-Trichlorobenzene | 25 | 83-135 |
| Benzene* | 11 | 76-127 |
| Bromobenzene | 17 | 83-117 |
| Bromochloromethane | 19 | 71-109 |

Table 5-14. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 2 of 3)

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Bromodichloromethane | 18 | 77-113 |
| Bromoform | 19 | 82-120 |
| Bromomethane | 25 | 70-120 |
| n-Butylbenzene | 23 | 77-123 |
| sec-Butylbenzene | 23 | 77-123 |
| tert-Butylbenzene | 22 | 80-124 |
| Carbon tetrachloride | 26 | 58-110 |
| Chlorobenzene* | 13 | 75-130 |
| Chloroform | 18 | 72-108 |
| Chloromethane | 27 | 66-120 |
| Chloroethane | 27 | 62-116 |
| 2-Chlorotoluene | 19 | 71-109 |
| 4-Chlorotoluene | 25 | 74-124 |
| Dibromochloromethane | 21 | 71-113 |
| 1,2-Dibromo-3-chloropropane | 60 | 23-143 |
| 1,2-Dibromoethane | 12 | 90-102 |
| Dibromomethane | 17 | 83-117 |
| 1,2-Dichlorobenzene | 19 | 74-112 |
| 1,3-Dichlorobenzene | 21 | 78-120 |
| 1,4-Dichlorobenzene | 19 | 84-122 |
| Dichlorodifluoromethane | 23 | 67-113 |
| 1,1,1-Trichloroethane | 24 | 74-122 |
| 1,1,2-Trichloroethane | 22 | 82-126 |
| Trichloroethene* | 14 | 71-120 |
| Trichlorofluoromethane | 24 | 65-113 |
| 1,2,3-Trichloropropane | 43 | 65-151 |
| 1,2,4-Trimethylbenzene | 24 | 75-123 |
| 1,3,5-Trimethylbenzene | 22 | 70-114 |

Table 5-14. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 3 of 3)

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Vinyl chloride | 20 | 78-118 |
| Xylene, total | 23 | 81-127 |
| Dichlorobenzene-D4† | NA | 87-107 |
| 4-Bromofluorobenzene† | NA | 86-115 |

Reference: Accuracy: EPA Method 524.2--Methods for the Determination of Organic Compounds in Drinking Water, EPA 600/4-88/039, December 1988 and CLP SOW 7/87 for the controlling analytes.
Precision: ESE (meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the EPA method 524.2) and CLP SOW 7/87 for controlling analytes.

Note: N/A = spiking and recovery information is not available.
NA = not applicable.

*Matrix spike and QC check sample compound.
†Surrogate; the surrogate is added to all environmental samples and quality control samples.

Source: ESE.

Table 5-15. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Dichlorobenzene, total | 1.0 |
| 1,1-Dichloroethane | 1.0 |
| 1,2-Dichloroethane | 1.0 |
| 1,1-Dichloroethylene | 1.0 |
| cis-1,2-Dichloroethene | 1.0 |
| trans-1,2-Dichloroethene | 1.0 |
| 1,2-Dichloropropane | 1.0 |
| 1,3-Dichloropropane | 1.0 |
| 2,2-Dichloropropane | 1.0 |
| 1,1-Dichloropropene | 1.0 |
| Ethylbenzene | 1.0 |
| Hexachlorobutadiene | 1.0 |
| Isopropylbenzene | 1.0 |
| cis-1,3-Dichloropropene | 1.0 |
| trans-1,3-Dichloropropene | 1.0 |
| p-Cymene | 1.0 |
| Naphthalene | 1.0 |
| Methylene chloride | 1.0 |
| Pentachloroethane | 1.0 |
| Styrene | 1.0 |
| 1,1,1,2-Tetrachloroethane | 1.0 |
| 1,1,2,2-Tetrachloroethane | 1.0 |
| Tetrachloroethene | 1.0 |
| Toluene | 1.0 |
| 1,2,3-Trichlorobenzene | 1.0 |
| 1,2,4-Trichlorobenzene | 1.0 |
| Benzene | 1.0 |
| Bromobenzene | 1.0 |
| Bromochloromethane | 1.0 |
| Bromodichloromethane | 1.0 |
| Bromoform | 1.0 |

Table 5-15. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2
(Continued, Page 2 of 3)

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Bromomethane | 1.0 |
| n-Butylbenzene | 1.0 |
| sec-Butylbenzene | 1.0 |
| tert-Butylbenzene | 1.0 |
| Carbon tetrachloride | 1.0 |
| Chlorobenzene | 1.0 |
| Chloroform | 1.0 |
| bis-2-Chloroisopropyl ether | 1.0 |
| Chloromethane | 1.0 |
| Chloroethane | 1.0 |
| 2-Chlorotoluene | 1.0 |
| 4-Chlorotoluene | 1.0 |
| Dibromochloromethane | 1.0 |
| 1,2-Dibromo-3-chloropropane | 3.0 |
| 1,2-Dibromoethane | 3.0 |
| Dibromomethane | 1.0 |
| 1,2-Dichlorobenzene | 1.0 |
| 1,3-Dichlorobenzene | 1.0 |
| 1,4-Dichlorobenzene | 1.0 |
| Dichlorodifluoromethane | 1.0 |
| 1,1,1-Trichloroethane | 1.0 |
| 1,1,2-Trichloroethane | 1.0 |
| Trichloroethene | 1.0 |
| Trichlorofluoromethane | 1.0 |
| 1,2,3-Trichloropropane | 1.0 |
| 1,2,4-Trimethylbenzene | 1.0 |
| 1,3,5-Trimethylbenzene | 1.0 |
| Vinyl chloride | 1.0 |
| Xylene, total | 1.0 |

Table 5-15. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2
(Continued, Page 3 of 3)

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*The reporting limits are based on the concentration that can be detected reliably according to ESE's past database and analytical experience performing
volatile organic analyses by gas chromatography/mass spectrometry.

Source: ESE.

Table 5-16. Analytes, Precision, and Accuracy Data For N-Methyl Carbamoxyloximes and N-Methyl Carbamates, EPA 531.1

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Aldicarb* | 17† | 83-117† |
| Aldicarb sulfone | 20 | 84-120 |
| Aldicarb sulfoxide | 20 | 72-128 |
| Carbaryl* | 26† | 63-115† |
| Carbofuran* | 18† | 81-117† |
| 3-Hydroxycarbofuran | 20 | 36-162 |
| Methomyl | 20 | 64-115 |
| Oxamyl | 20 | 34-148 |

Reference: Accuracy: EPA Method 531.1--Supplement to "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," EPA, Cincinnati, OH, September 1986.
Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (EPA 531.1).

*Matrix spike and QC check sample compound.
†Accuracy and precision criteria are based on ESE historical data.

Source: ESE.

Table 5-17. Reporting Limit Data for N-Methyl Carbamoxyloximes and N-Methyl Carbamates, EPA 531.1

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Aldicarb | 2.0 |
| Aldicarb sulfone | 2.0 |
| Aldicarb sulfoxide | 2.0 |
| Carbaryl | 2.0 |
| Carbofuran | 2.0 |
| 3-Hydroxycarbofuran | 2.0 |
| Methomyl | 2.0 |
| Oxamyl | 2.0 |

*Based on the lowest standard that ESE routinely uses. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-18. Analytes, Precision, and Accuracy Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Bromodichloromethane | 20 | 42-172 | 30 | 42-172 |
| Bromoform | 20 | 13-159 | 30 | 13-159 |
| Bromomethane | 20 | 0-144 | 30 | 0-144 |
| Carbon tetrachloride | 20 | 43-143 | 30 | 43-143 |
| Chlorobenzene* | 43† | 51-137† | 50 | 38-150 |
| Chloroethane | 20 | 46-137 | 30 | 46-137 |
| 2-Chloroethylvinylether | 20 | 14-186 | 30 | 14-186 |
| Chloroform | 20 | 49-133 | 30 | 49-133 |
| Chloromethane | 20 | 0-193 | 30 | 0-193 |
| Dibromochloromethane | 20 | 24-191 | 30 | 24-191 |
| Dichlorobenzene,tot. | 20 | 42-143 | 30 | 42-143 |
| 1,1-Dichloroethane | 20 | 47-132 | 30 | 47-132 |
| 1,2-Dichloroethane | 20 | 51-147 | 30 | 51-147 |
| 1,1-Dichloroethylene* | 38† | 51-127† | 30 | 28-167 |
| Trans-1,2-dichloroethene | 20 | 38-155 | 30 | 38-155 |
| 1,2-Dichloropropane | 20 | 44-156 | 30 | 44-156 |
| cis-1,3-Dichloropropene | 20 | 22-178 | 30 | 22-178 |

Table 5-18. Analytes, Precision, and Accuracy Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010 (Continued, Page 2 of 2)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Trans-1,3-dichloropropene | 20 | 22-178 | 30 | 22-178 |
| Methylene chloride | 20 | 25-162 | 30 | 25-162 |
| 1,1,2,2-Tetrachloroethane | 20 | 8-184 | 30 | 8-184 |
| Tetrachloroethene | 20 | 26-162 | 30 | 26-162 |
| 1,1,1-Trichloroethane | 20 | 41-138 | 30 | 41-138 |
| 1,1,2-Trichloroethane | 20 | 39-136 | 30 | 39-136 |
| Trichloroethene* | 40† | 54-134†† | 30 | 35-146 |
| Trichlorofluoromethane | 20 | 21-156 | 30 | 21-156 |
| Vinyl chloride | 20 | 28-163 | 30 | 28-163 |
| Freon 113** | 33 | 73-139 | N/A | N/A |
| Bromofluorobenzene (surrogate) | NA | 43-125 | NA | 69-113 |

Reference: Accuracy: EPA Method SW 8010--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986. Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8010).

Note: N/A - spiking and recovery information are not available.
NA - Not applicable

*Matrix spike and QC check sample compound.
†Accuracy and precision criteria are based on ESE historical data.
**Accuracy and precision criteria are based on ESE validation study (see Appendix U).
Source: ESE.

Table 5-19. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid* (µg/kg) |
|---|---|---|
| Bromodichloromethane** | 1.0 | 100 |
| Bromoform** | 1.0 | 100 |
| Bromomethane** | 1.0 | 100 |
| Carbon tetrachloride** | 1.0 | 100 |
| Chlorobenzene** | 1.0 | 100 |
| Chloroethane** | 1.0 | 100 |
| 2-Chloroethylvinylether** | 1.0 | 100 |
| Chloroform** | 1.0 | 100 |
| Chloromethane** | 1.0 | 100 |
| Dibromochloromethane** | 1.0 | 100 |
| Dichlorobenzene,tot.** | 1.0 | 100 |
| 1,1-Dichloroethane** | 1.0 | 100 |
| 1,2-Dichloroethane** | 1.0 | 100 |
| 1,1-Dichloroethylene** | 1.0 | 100 |
| Trans-1,2-dichloroethene** | 1.0 | 100 |
| 1,2-Dichloropropane** | 1.0 | 100 |
| cis-1,3-Dichloropropene | 1.0 | 100 |
| Trans-1,3-dichloropropene | 1.0 | 100 |
| Methylene chloride** | 1.0 | 100 |
| 1,1,2,2-Tetrachloroethane** | 1.0 | 100 |
| Dichlorodifluoromethane | 1.0 | 100 |

Table 5-19. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010 (Continued, Page 2 of 2)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid† (µg/kg) |
|---|---|---|
| Tetrachloroethene** | 1.0 | 100 |
| 1,1,1-Trichloroethane** | 1.0 | 100 |
| 1,1,2-Trichloroethane** | 1.0 | 100 |
| Trichloroethene** | 1.0 | 100 |
| Trichlorofluoromethane** | 1.0 | 100 |
| Vinyl chloride** | 1.0 | 100 |
| Freon 113 | 1.0 | N/A |

Note: N/A = Reporting limit information not available.

*Based on the lowest standard that ESE routinely uses.
†Based on the lowest standard that ESE routinely uses times a factor of 100. The solid reporting limits are expressed on a wet weight basis.
**Appendix IX compounds.

Source: ESE.

Table 5-20. Analytes, Precision, and Accuracy Data for Purgeable Aromatics, EPA 602 and SW 5030/8020

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Benzene*,†† | 44† | 44-132† | 28† | 74-130† |
| Chlorobenzene†† | 20 | 55-135 | 30 | 55-135 |
| Dichlorobenzene,total†† | 20 | 42-143 | 30 | 42-143 |
| Ethylbenzene†† | 20 | 32-160 | 30 | 32-160 |
| Toluene*,†† | 40† | 47-127† | 26† | 75-127† |
| Xylenes, total†† | 23† | 80-126† | 27† | 74-128† |
| MTBE | 17 | 13-147 | 47 | 90-184 |
| Total VOA (BTEX) | 42† | 46-130† | 27† | 75-129† |
| Bromofluorobenzene (surrogate) | NA | 76-124 | NA | 82-116 |
| Fluorobenzene (surrogate) | NA | 67-127 | NA‘ | 67-127 |

Reference: Accuracy: EPA Method SW 8020--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8020).

Note: Total VOA (BTEX) is calculated and defined as the arithmetic sum of the concentrations of benzene, toluene, ethylbenzene, and total xylenes.

MTBE = methyl tert-butyl ether.
N/A = spiking and recovery information are not available.
NA = not applicable

* Matrix spike and QC check sample compound.
† Accuracy and precision criteria are based on ESE historical data.
†† Appendix IX compounds.

Source: ESE.

Table 5-21. Reporting Limit for Purgeable Aromatics, EPA 602 and SW 5030/8020

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid† (µg/kg) |
|---|---|---|
| Benzene | 1.0 | 100 |
| Chlorobenzene | 1.0 | 100 |
| Dichlorobenzene, total | 1.0 | 100 |
| Ethylbenzene | 1.0 | 100 |
| Toluene | 1.0 | 100 |
| Xylenes, total | 1.0 | 100 |
| MTBE | 1.0 | 100 |
| Total VOA (BTEX) | 5.0 | 500 |

Note: N/A = Reporting limit information not available.

*Based on the lowest standard that ESE routinely uses.
†Based on the lowest standard that ESE routinely uses times a factor of 100. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-22. Analytes, Precision, and Accuracy Data for Phenols, EPA 604 and SW 3510/3520/3540/3550/8040

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 4-Chloro-3-methylphenol*,† | 30 | 39-130 | 50 | 39-130 |
| 2-Chlorophenol*,† | 30 | 38-126 | 50 | 38-126 |
| 2,4-Dichlorophenol† | 30 | 44-119 | 50 | 44-119 |
| 2,4-Dimethylphenol† | 30 | 24-118 | 50 | 24-118 |
| 2,4-Dinitrophenol† | 30 | 12-145 | 50 | 12-145 |
| 2-Methyl-4,6-dinitrophenol† | 30 | 30-136 | 50 | 30-136 |
| 2-Nitrophenol† | 30 | 43-117 | 50 | 43-117 |
| 4-Nitrophenol† | 30 | 13-110 | 50 | 13-110 |
| Pentachlorophenol*,† | 30 | 36-134 | 50 | 36-134 |
| Phenol*,† | 30 | 23-108 | 50 | 23-108 |
| 2,4,6-Trichlorophenol† | 30 | 53-119 | 50 | 53-119 |

Reference: Accuracy: EPA Method SW 8040--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8040).

*Matrix spike and QC check sample compound.
†Appendix IX compounds.

Source: ESE.

Table 5-23. Reporting Limit Data for Phenols, EPA 604 and SW 3510/3520/3540/3550/8040

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| 4-Chloro-3-methylphenol | 5.0 | 500 |
| 2-Chlorophenol | 5.0 | 500 |
| 2,4-Dichlorophenol | 5.0 | 500 |
| 2,4-Dimethylphenol | 5.0 | 500 |
| 2,4-Dinitrophenol | 20.0 | 2,000 |
| 2-Methyl-4,6-dinitrophenol | 5.0 | 500 |
| 2-Nitrophenol | 5.0 | 500 |
| 4-Nitrophenol | 25.0 | 2,500 |
| Pentachlorophenol | 10.0 | 1,000 |
| Phenol | 5.0 | 500 |
| 2,4,6-Trichlorophenol | 6.0 | 600 |

*Based on the lowest standard that ESE routinely uses taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid detection limits are expressed on a wet weight basis.

Source: ESE.

Table 5-24. Analytes, Precision, and Accuracy Data for Benzidines, EPA 605

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (%Recovery) |
| Benzidine* | 30 | 31-92 |
| 3,3'-Dichlorobenzidine | 30 | 35-93 |

Reference: Accuracy: EPA Method 605--Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (EPA 605).

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-25. Reporting Limit Data for Benzidines, EPA 605

| Parameter | Reporting Limit* Aqueous (µg/L) |
|---|---|
| Benzidine | 1.0 |
| 3,3'-Dichlorobenzidine | 1.5 |

*Ten times the method detection limit listed in Table 1 of EPA Method 605.

Source: ESE.

Table 5-26. Analytes, Precision, and Accuracy Data for Phthalate Esters, EPA 606 and SW 35010/3520/3540/3550/8060

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| bis(2-Ethylhexyl)phthlate | 30 | 0-158 | 50 | 0-158 |
| Butyl benzyl phthalate | 30 | 30-136 | 50 | 30-136 |
| di-n-Butylphthalate* | 30 | 23-136 | 50 | 23-136 |
| Diethylphthalate* | 30 | 0-149 | 50 | 0-149 |
| Dimethylpthalate | 30 | 0-156 | 50 | 0-156 |
| di-n-Octylphthalate* | 30 | 0-114 | 50 | 0-114 |

Reference: Accuracy: EPA Method SW 8060--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8060).

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-27. Reporting Limit Data for Phthalate Esters, EPA 606 and SW 3510/3520/3540/3550/8060

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid† (µg/kg) |
|---|---|---|
| bis(2-Ethylhexyl)phthlate | 0.15 | 25 |
| Butyl benzylphthalate | 0.15 | 25 |
| di-n-Butylphthalate | 0.15 | 25 |
| Diethylphthalate | 0.15 | 25 |
| Dimethylpthalate | 0.15 | 25 |
| di-n-Octylphthalate | 0.30 | 50 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. the solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-28. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608/617 and SW 3510/3520/3540/3550/8080

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Aldrin[a,g] | 45[b] | 37-127[b] | 40[b] | 53-133[b] |
| BHC,A[g] | 30 | 37-134 | 50 | 37-134 |
| BHC,B[g] | 30 | 17-147 | 50 | 17-147 |
| BHC,D[g] | 30 | 19-140 | 50 | 19-140 |
| BHC,G(lindane)[a,g] | 51[b] | 43-145[b] | 42[b] | 45-129[b] |
| Chlordane[g] | 30 | 45-119 | 50 | 45-119 |
| DDD,PP'[g] | 30 | 31-141 | 50 | 31-141 |
| DDE,PP'[g] | 30 | 30-145 | 50 | 30-145 |
| DDT,PP'[a,g] | 53[b] | 46-152[b] | 59[b] | 37-155[b] |
| Dieldrin[a,g] | 43[b] | 56-142[b] | 47[b] | 46-140[b] |
| Endosulfan,A[g] | 30 | 45-153 | 50 | 45-153 |
| Endosulfan,B[g] | 30 | 0-202 | 50 | 0-202 |
| Endosulfan sulfate[g] | 30 | 26-144 | 50 | 26-144 |
| Endrin[a,g] | 60[b] | 35-155[b] | 37[b] | 52-126[b] |
| Endrin aldehyde[g] | 40[e] | 58-138[e] | 53[e] | 56-162[e] |
| Heptachlor[a,g] | 38[b] | 48-124[b] | 59[b] | 30-148[b] |
| Heptachlor epoxide[g] | 30 | 37-142 | 50 | 37-142 |

Table 5-28. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608/617 and SW 3510/3520/3540/3550/8080 (Continued, Page 2 of 3)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Methoxychlor[g] | 44[e] | 78-162[e] | 53[e] | 61-167[e] |
| Toxaphene[g] | 30 | 41-126 | 50 | 41-126 |
| Isodrin[g,i] | 27[b] | 74-128[b] | 10[b] | 97-117[b] |
| Kepone[g,h] | 44[b] | 26-114[b] | 30[b] | 98-158[b] |
| Metolachlor | 28[f] | 74-130[f] | 24[f] | 62-110[f] |
| Kelthane (Dicofol) | 20[f] | 47-87[f] | 19[f] | 40-78[f] |
| PCB-1016[d,g] | 30 | 50-114 | 50 | 50-114 |
| PCB-1221[g] | 30 | 15-178 | 50 | 15-178 |
| PCB-1232[g] | 30 | 10-215 | 50 | 10-215 |
| PCB-1242[g] | 30 | 39-150 | 50 | 39-150 |
| PCB 1248[g] | 30 | 38-158 | 50 | 38-158 |
| PCB-1254[g] | 30 | 29-131 | 50 | 29-131 |
| PCB-1260[d,g] | 30 | 8-127 | 50 | 8-127 |
| Dibutylchlorendate[c] (surrogate) | NA | 46-146[b] | NA | 32-156[b] |
| Tetrachloro-m-xylene (surrogate) | NA | 60-150 | NA | 60-150 |
| Decachlorobiphenyl (surrogate) | NA | 60-150 | NA | 60-150 |

Table 5-28. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608/617 and SW 3510/3520/3540/3550/8080 (Continued, Page 3 of 3)

Reference: Accuracy: EPA Method SW 8080 and CLP SOW 7/87 for the controlling analytes.
Precision: ESE and CLP SOW 7/87 for the controlling analytes.

Note: NA = not applicable.

[a]Matrix spike and QC check sample compound. For Los Alamos project CLP SOW 12/90 precision and accuracy acceptance criteria will be used.
[b]Accuracy and precision criteria are based on ESE historical data and method detection limit data.
[c]Surrogate; the surrogate is added to all environmental samples and quality control samples.
[d]PCB 1016 and PCB 1260 are only used as matrix spike and QCC samples compounds when using EPA 608/8080 to evaluate PCBs only.
[e]Accuracy and precision data are from ESE method certification.
[f]Based on validation studies performed by ESE (see Appendices D and E).
[g]Appendix IX compounds.
[h]This compound is not included in EPA's parameter lists for Methods 608, 617, and 8080; however, it can be analyzed by EPA method 8080 and is reported if specifically requested by the client (see Appendices L and M for method validation data).
[i]See Appendices L and M for method validation data of isodrin by EPA Method 8050.

Source: ESE.

Table 5-29. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| Aldrin | 0.006 | 3 |
| BHC,A | 0.006 | 3 |
| BHC,B | 0.006 | 3 |
| BHC,D | 0.006 | 3 |
| BHC,G(lindane) | 0.006 | 3 |
| Chlordane | 0.030 | 20 |
| DDD,PP | 0.006 | 3 |
| DDE,PP' | 0.006 | 3 |
| DDT,PP' | 0.006 | 3 |
| Dieldrin | 0.006 | 3 |
| Endosulfan,A | 0.006 | 3 |
| Endosulfan,B | 0.006 | 3 |
| Endosulfan sulfate | 0.006 | 3 |
| Endrin | 0.006 | 3 |
| Endrin aldehyde | 0.006 | 3 |
| Heptachlor | 0.006 | 3 |
| Heptachlor epoxide | 0.006 | 3 |
| Methoxychlor | 0.006 | 3 |
| Toxaphene | 0.6 | 300 |
| PCB-1016 | 0.12 | 60 |
| Mirex | 0.2 | 100 |
| Trifluralin | 0.1 | 50 |
| PCNB | 0.02 | 1 |

Table 5-29. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080
(Continued, Page 2 of 2)

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| Isodrin | 0.05 | 8 |
| Kepone | 0.10 | 50 |
| Metolachlor | 0.06 | 10 |
| Kelthane (Dicofol) | 0.06 | 10 |
| PCB-1221 | 0.12 | 60 |
| PCB-1232 | 0.12 | 60 |
| PCB-1242 | 0.12 | 60 |
| PCB-1248 | 0.12 | 60 |
| PCB-1254 | 0.12 | 60 |
| PCB-1260 | 0.12 | 60 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-30. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Acenaphthene[•,••] | 30[†] | 49-109[†] | 50 | 0-124 |
| Acenaphthylene[•,••] | 25[†] | 53-103[†] | 50 | 0-139 |
| Anthracene[•,••] | 40[†] | 44-124[†] | 50 | 0-126 |
| Benzo(a)anthracene[••] | 30 | 12-135 | 50 | 12-135 |
| Benzo(a)pyrene[•,••] | 38[†] | 45-121[†] | 62[†] | 27-151[†] |
| Benzo(b)fluoranthene[••] | 30 | 6-150 | 50 | 6-150 |
| Benzo(ghi)perylene[••] | 30 | 0-116 | 50 | 0-116 |
| Benzo(k)fluoranthene[•,••] | 41[†] | 41-123[†] | 55[†] | 22-132[†] |
| Chrysene[••] | 30 | 0-199 | 50 | 0-199 |
| Dibenyl(a,h)anthracene[••] | 30 | 0-110[†] | 50 | 0-110 |
| Fluoranthene[••] | 30 | 14-123 | 50 | 14-123 |
| Fluorene[•,••] | 35[†] | 40-110[†] | 49[†] | 25-123[†] |
| Indeno(1,2,3-cd)pyrene[••] | 30 | 0-116 | 50 | 0-116 |
| Naphthalene[•,••] | 48[†] | 21-117[†] | 50 | 0-122 |
| Phenanthrene[•,••] | 32[†] | 52-116[†] | 50 | 0-155 |
| Pyrene[••] | 30 | 0-140 | 50 | 0-140 |
| 1-Methylnaphthalene[••] | 37[†] | 23-97[†] | 37[†] | 23-97[†] |

Table 5-30. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310 (Continued, Page 2 of 2)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 2-Methylnaphthalene** | 37† | 23-97† | 37† | 23-97† |
| Methylnaphthalene, total | 37† | 23-97† | 75† | 10-160† |
| Triphenylene (surrogate) | NA | 60-124† | NA | 30-124† |

Reference: Accuracy: EPA Method SW 8310--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8310).

*Matrix spike and QC check sample compound.
†Accuracy and precision criteria are based on ESE historical data.
**Appendix IX compounds.

Source: ESE.

Table 5-31. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| Acenaphthene | 2.5 | 84 |
| Acenaphthylene | 1.5 | 44 |
| Anthracene | 0.095 | 3.5 |
| Benzo(a)anthracene | 0.002 | 0.05 |
| Benzo(a)pyrene | 0.004 | 0.4 |
| Benzo(b)fluoranthene | 0.001 | 0.02 |
| Benzo(ghi)perylene | 0.006 | 0.20 |
| Benzo(k)fluoranthene | 0.001 | 0.01 |
| Chrysene | 0.03 | 0.80 |
| Dibenzo(a,h)anthracene | 0.004 | 0.07 |
| Fluoranthene | 0.003 | 0.08 |
| Fluorene | 0.25 | 10 |
| Indeno(1,2,3-cd)pyrene | 0.004 | 0.15 |
| Naphthalene | 0.90 | 23 |
| Phenanthrene | 0.07 | 4.0 |
| Pyrene | 0.03 | 0.6 |
| 1-Methylnaphthalene | 1.5 | 45 |
| 2-Methylnaphthalene | 1.2 | 36 |
| Methylnaphthalene, total | 3.0 | 63 |

Table 5-31. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310
(Continued, Page 2 of 2)

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*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account sample weight and final extract volume. The solid reporting limits are expressed on a wet weight basis. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-32. Analytes, Precision, and Accuracy Data for Chlorinated Herbicides, EPA 615 and SW 8150.

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 2,4-D*,** | 55† | 9-119† | 48† | 35-131† |
| 2,4-DB | 30 | 84-102 | 50 | 84-102 |
| 2,4,5-T** | 30 | 67-103 | 50 | 67-103 |
| 2,4,5-TP/Silvex†der.*,** | 51† | 33-135† | 41† | 61-143† |
| Dicamba (banvel)* | 47† | 21-115† | 32† | 57-121† |
| Dalapon | 30 | 42-90 | 50 | 42-90 |
| Dichlorprop | 30 | 91-103 | 50 | 91-103 |
| Dinoseb** | 30 | 74-100 | 50 | 74-100 |
| MCPA | 30 | 86-110 | 50 | 86-110 |
| MCPP | 30 | 82-106 | 50 | 82-106 |

Reference: Accuracy: EPA Method SW 8150--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8150).

*Matrix spike and QC check sample compound.
†Accuracy and precision criteria are based on ESE historical data.
**Appendix IX compounds.

Source: ESE.

Table 5-33. Reporting Limit Data for Chlorinated Herbicides, EPA 615 and SW 8150

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| 2,4-D | 0.03 | 3 |
| 2,4-DB | 0.03 | 3 |
| 2,4,5-T | 0.03 | 3 |
| 2,4,5-TP/Silvex+der. | 0.03 | 3 |
| Dicamba (banvel) | 0.03 | 3 |
| Dalapon | 0.03 | 3 |
| Dichlorprop | 0.03 | 3 |
| Dinoseb | 0.03 | 3 |
| MCPA | 0.60 | 50 |
| MCPP | 0.60 | 50 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-34. Analytes, Precision, and Accuracy Data for Organophosphorous Pesticides, EPA 622/614 and SW 3510/3520/3540/3550/8140

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Bolstar | 30 | 46-84 | 50 | 46-84 |
| Chlorpyrifos | 30 | 82-115 | 50 | 82-115 |
| Coumaphos | 30 | 71-147 | 50 | 71-147 |
| Demeton | 30 | 36-99 | 50 | 36-99 |
| Diazinon* | 47** | 61-155** | 294** | 75-133** |
| Dichlorvos | 30 | 49-95 | 50 | 49-95 |
| Disulfoton†† | 30 | 55-109 | 50 | 55-109 |
| Ethoprop | 30 | 88-113 | 50 | 88-113 |
| Fenthion | 30 | 9-128 | 50 | 9-128 |
| Fensulfothion | 30 | 43-145 | 50 | 43-145 |
| Guthion (methylazinphos)* | 51** | 44-146** | 51** | 59-161** |
| Malathion* | 35** | 64-134** | 31** | 66-128** |
| Merphos | 30 | 97-144 | 50 | 97-144 |
| Mevinphos | 30 | 33-80 | 50 | 33-80 |
| Naled | 30 | 54-102 | 50 | 54-102 |
| Ethylparathion*,†† | 43** | 65-151** | 49** | 61-159** |
| Methylparathion†† | 30 | 80-112 | 50 | 80-112 |

Table 5-34. Analytes, Precision, and Accuracy Data for Organophosphorous Pesticides, EPA 622/614 and SW 3510/3520/3540/3550/8140 (Continued, Page 2 of 3)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Phorate[††] | 30 | 36-89 | 50 | 36-89 |
| Ronnel | 30 | 82-116 | 50 | 82-116 |
| Stirofos | 30 | 48-84 | 50 | 48-84 |
| Tokuthion | 30 | 44-85 | 50 | 44-85 |
| Trichloronate | 30 | 49-161 | 50 | 49-161 |
| Alachlor[***] | N/A | N/A | 19 | 112-150 |
| Metribuzin[***] | N/A | N/A | 31 | 62-124 |
| EPTC[-] | 25 | 70-120 | 6 | 97-109 |
| Butylate[+] | 23 | 61-107 | 7 | 95-114 |
| Pebulate[+] | 22 | 69-113 | 11 | 89-111 |
| Vernolate[+] | 23 | 69-115 | 8 | 94-110 |
| Atrazine[+] | 32 | 79-143 | 32 | 61-125 |
| Terbufos[+] | 16 | 79-111 | 15 | 88-118 |
| Hexazinone[+] | 50 | 56-150 | 50 | 40-140 |
| Famphur[††] | 61 | 52-174 | 40 | 69-149 |
| 0,0,0-Triethyl Phosphorothioate[††,***,†††] | 44 | 41-129 | 14 | 107-135 |
| Sulfotepp[††,***,†††] | 28 | 50-106 | 13 | 99-125 |

Table 5-34. Analytes, Precision, and Accuracy Data for Organophosphorous Pesticides, EPA 622/614 and SW 3510/3520/3540/3550/8140 (Continued, Page 3 of 3)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Thionazin[††,***,†††] | 22 | 64-108 | 13 | 92-118 |

Reference: Accuracy: EPA Method SW 8140--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE; meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8140).

Note: N/A = spiking and recovery information not available.

[•]Matrix spike and QC check sample compound.
[*]Precision and accuracy criteria are based on validation studies performed by ESE (see Appendices F and G).
[**]Accuracy and precision criteria are based on ESE historical data.
[††]Appendix IX compounds.
[***]Precision and accuracy criteria are based on validation studies performed by ESE (see Appendices J and K).
[†††]This compound is not included in EPA's parameter list for Method 614, 622, and 8140; however, it can be analyzed by EPA Method 8140 and is reported if specifically requested by the client.

Source: ESE.

Table 5-35. Reporting Limit Data for Organophosphorous Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8140

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| Bolstar | 0.3 | 50 |
| Chlorpyrifos | 0.3 | 50 |
| Coumaphos | 3.0 | 500 |
| Demeton | 0.3 | 50 |
| Diazinon | 0.3 | 50 |
| Dichlorvos | 0.3 | 50 |
| Disulfoton | 0.3 | 50 |
| Ethoprop | 0.3 | 50 |
| Fenthion | 0.3 | 50 |
| Fensulfothion | 1.5 | 250 |
| Guthion (methylazinphos) | 3.0 | 500 |
| Malathion | 0.3 | 50 |
| Merphos | 0.3 | 50 |
| Mevinphos | 0.3 | 50 |
| Naled | 1.5 | 250 |
| Ethylparathion | 0.3 | 50 |
| Methylparathion | 0.3 | 50 |
| Phorate | 0.3 | 50 |
| Ronnel | 0.3 | 50 |
| Stirofos | 0.6 | 100 |

Table 5-35. Reporting Limit Data for Organophosphorous Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8140 (Continued, Page 2 of 2)

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| Tokuthion | 0.3 | 50 |
| Trichloronate | 0.3 | 50 |
| Alachlor | N/A | 100 |
| Metribuzin | N/A | 250 |
| EPTC | 0.3 | 50 |
| Butylate | 0.3 | 50 |
| Pebulate | 0.3 | 50 |
| Vernolate | 0.3 | 50 |
| Atrazine | 0.6 | 100 |
| Terbufos | 0.3 | 50 |
| Hexazinone | 1.5 | 250 |
| Famphur | 0.5 | 70 |
| 0,0,0-Triethyl Phosphorothioate | 0.5 | 70 |
| Sulfotepp | 0.5 | 70 |
| Thionazin | 0.5 | 70 |

Note: N/A = reporting limit not available.

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-36. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Acetone[d] | 53[c] | 56-162[c] | 53[c] | 52-158[c] |
| Benzene[a,d] | 11 | 76-127 | 21 | 66-142 |
| Bromodichloromethane[d] | 20 | 35-155 | 30 | 35-155 |
| Bromoform[d] | 20 | 45-169 | 30 | 45-169 |
| Bromomethane[d] | 20 | 0-242 | 30 | 0-242 |
| Carbon tetrachloride[d] | 20 | 70-140 | 30 | 70-140 |
| Chlorobenzene[a,d] | 13 | 75-130 | 21 | 60-133 |
| Chlorobenzene[e] | 9 | 89-107 | 37 | 76-150 |
| 2-Chloroethylvinyl ether | 20 | 0-305 | 30 | 0-305 |
| Chloroform[d] | 20 | 51-138 | 30 | 51-138 |
| Chloromethane[d] | 20 | 0-273 | 30 | 0-273 |
| Dibromochloromethane[d] | 20 | 53-149 | 30 | 53-149 |
| Dichlorobenzene,tot.[d] | 20 | 18-190 | 30 | 18-190 |
| 1,1-Dichloroethane[d] | 20 | 59-155 | 30 | 59-155 |
| 1,2-Dichloroethane[d] | 20 | 49-155 | 30 | 49-155 |
| 1,1-Dichloroethylene[a,d] | 14 | 61-145 | 22 | 59-172 |
| trans-1,2-Dichloroethene[d] | 20 | 54-156 | 30 | 54-156 |

Table 5-36. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 4)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 1,2-Dichloropropane[d] | 20 | 0-210 | 30 | 0-210 |
| cis-1,3-Dichloropropene[d] | 20 | 0-227 | 30 | 0-227 |
| trans-1,3-Dichloropropene[d] | 20 | 17-183 | 30 | 17-183 |
| Methylene chloride[d] | 20 | 0-221 | 30 | 0-221 |
| Methyl ethyl ketone[d] (MEK) | 42[e] | 61-145[e] | 79[e] | 12-172[e] |
| Methyl isobutyl ketone[d] (MIBK) | 46[e] | 50-146[e] | 41[e] | 78-160[e] |
| Styrene[d] | 13[e] | 91-117[e] | 13[e] | 89-115[e] |
| 1,1,2,2-Tetrachloroethane[d] | 20 | 46-157 | 30 | 46-157 |
| Tetrachloroethene[d] | 20 | 64-148 | 30 | 64-148 |
| Toluene[a,d] | 13 | 76-125 | 21 | 59-139 |
| 1,1,1-Trichloroethane[d] | 20 | 52-162 | 30 | 52-162 |
| 1,1,2-Trichloroethane[d] | 20 | 52-150 | 30 | 52-150 |
| Trichloroethene[a,d] | 14 | 71-120 | 24 | 62-137 |
| Trichlorofluoromethane[d] | 20 | 17-181 | 30 | 17-181 |
| Vinyl chloride[d] | 20 | 0-251 | 30 | 0-251 |
| Xylene, total[d] | 26[e] | 83-135[e] | 46[e] | 52-144[e] |
| Toluene-D8[f] | NA | 88-110 | NA | 81-117 |

Table 5-36. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 3 of 4)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 4-Bromofluorobenzene[†] | NA | 86-115 | NA | 74-121 |
| 1,2-Dichloroethane-D4[†] | NA | 76-114 | NA | 70-121 |
| Dibromofluoromethane [§] | NA | 86-118 | NA | 80-120 |
| Acetonitrile[d,f] | 48[e] | 120-216[e] | 49[e] | 91-188[e] |
| Acrolein[d] | 21[e] | 75-117[e] | 55[e] | 6-116[e] |
| Acrylonitrile[d] | 52[e] | 25-129[e] | 10[e] | 72-92[e] |
| Carbon Disulfide[d] | 14[e] | 81-109[e] | 47[e] | 97-191[e] |
| Chloroprene[d,f] | 8[e] | 93-109[e] | 8[e] | 89-105[e] |
| 3-Chloropropene[d,f] | 8[e] | 87-103[e] | 21[e] | 116-158[e] |
| Dichlorodifluoromethane[d] | 32[e] | 67-131[e] | 50[e] | 37-137[e] |
| trans- 1,4-Dichloro-2-butene[d,f] | 20[e] | 69-109[e] | 63[e] | 0-121[e] |
| 1,4-Dioxane[d,f] | 8[e] | 95-111[e] | 31[e] | 83-145[e] |
| Ethyl Methacrylate[d] | 12[e] | 112-136[e] | 19[e] | 100-138[e] |
| 2-Hexanone[d] | 41[e] | 100-182[e] | 98[e] | 45-241[e] |
| Iodomethane[d] | 15[e] | 84-114[e] | 53[e] | 61-167[e] |
| Isobutanol[d,f] | 31[e] | 71-133[e] | 37[e] | 74-148[e] |
| Methacrylonitrile[d,f] | 19[e] | 52-90[e] | 11[e] | 87-109[e] |

Table 5-36. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 4 of 4)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Methyl Methacrylate[d,f] | 16[e] | 66-98[e] | 33[e] | 56-122[e] |
| Pentachloroethane[d,f] | 36[e] | 48-120[e] | 59[e] | 20-138[e] |
| Propionitrile[d,f] | 53[e] | 114-220[e] | 60[e] | 25-145[e] |
| 1,1,1,2-Tetrachloroethane[d,f] | 6[e] | 88-100[e] | 23[e] | 75-121[e] |
| 1,2,3-Trichloropropane[d] | 8[e] | 121-137[e] | 27[e] | 89-143[e] |
| Vinyl Acetate[d] | 37[e] | 57-131[e] | 33[e] | 46-110[e] |

Reference: Accuracy: EPA Method SW 8240--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986 and CLP SOW 7/87 for the controlling analytes.
Precision: ESE (meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8240) and CLP SOW 7/87 for controlling analytes.

Note: NA = not applicable.

[a]Matrix spike and QC check sample compound.
[b]Surrogate; the surrogate is added to all environmental samples and quality control samples.
[c]Accuracy and precision data are from ESE method certification.
[d]Appendix IX compounds.
[e]Accuracy and precision data are from ESE method detection limit study.
[f]This compound is not included in EPA's lists of compounds (EPA 624, 8240, and 8260) methods; however, it can be analyzed by this method and is reported if specifically requested by the client (see Appendix N for method validation data).
[g]This surrogate will be used only for Los Alamos project.
Source: ESE.

Table 5-37. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid (µg/kg)** Low Level | Medium Level |
|---|---|---|---|
| Acetone | 9.0 | 10 | 5500† |
| Benzene | 2.2 | 5 | 220† |
| Bromodichloromethane | 2.2 | 5 | 110† |
| Bromoform | 2.6 | 5 | 235† |
| Bromomethane | 3.5 | 10 | 290† |
| Carbon tetrachloride | 2.6 | 5 | 220 |
| Chlorobenzene | 1.4 | 5 | 300† |
| Chloroethane | 8.2 | 10 | 410 |
| 2-Chloroethylvinyl ether | 3.1 | 5 | 500† |
| Chloroform | 2.5 | 5 | 110 |
| Chloromethane | 4.4 | 10 | 310 |
| Dibromochloromethane | 2.3 | 5 | 155† |
| Dichlorobenzene, total | 4.0 | 10 | 350† |
| 1,1-Dichloroethane | 2.5 | 5 | 235† |
| 1,2-Dichloroethane | 2.5 | 5 | 170 |
| 1,1-Dichloroethylene | 3.2 | 5 | 200 |
| trans-1,2-Dichloroethene | 2.4 | 5 | 120 |
| 1,2-Dichloropropane | 2.0 | 5 | 300† |
| cis-1,3-Dichloropropene | 2.0 | 5 | 250† |
| trans-1,3-Dichloropropene | 1.6 | 5 | 320† |
| Ethylbenzene | 1.3 | 5 | 360† |

Table 5-37. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 3)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid (µg/kg)** Low Level | Medium Level |
|---|---|---|---|
| Methylene chloride | 6.4 | 5.0 | 1000† |
| Methyl ethyl ketone | 10 | 10 | 2400† |
| Methyl isobutyl ketone | 12 | 10 | 600† |
| 2-Nitropropane | 50 | 50† | NA |
| Styrene | 0.5 | 5.0 | 355† |
| 1,1,2,2-Tetrachloroethane | 1.5 | 5.0 | 205† |
| Tetrachloroethene | 1.9 | 5.0 | 205† |
| Toluene | 1.7 | 5.0 | 300† |
| 1,1,1-Trichloroethane | 2.5 | 5.0 | 190† |
| 1,1,2-Trichloroethane | 2.8 | 5.0 | 250† |
| Trichloroethene | 3.0 | 5.0 | 120 |
| Trichlorofluoromethane | 4.6 | 5.0 | 300 |
| Vinyl chloride | 4.6 | 10.0 | 360 |
| Xylene, total | 3.72 | 5.0 | 770 |
| Acetonitrile | 200 | 50 | NA |
| Acrolein | 100 | 100 | NA |
| Acrylonitrile | 100† | 100† | NA |
| Carbon Disulfide | 5† | 5† | NA |
| Chloroprene | 10† | 10† | NA |
| 3-Chloropropene | 50† | 50† | NA |
| Dichlorodifluoromethane | 200† | 200† | NA |
| trans-1,4-Dichloro-2-butene | 50† | 5† | NA |
| 1,4-Dioxane | 200† | 50 | 200† |

Table 5-37. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 3 of 3)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid (µg/kg)** Low Level | Medium Level |
|---|---|---|---|
| Ethyl Methacrylate | 5† | 5† | NA |
| 2-Hexanone | 21† | 10† | NA |
| Iodomethane | 5† | 5† | NA |
| Isobutanol | 1000† | 1000† | NA |
| Methacrylonitrile | 5† | 5† | NA |
| Methyl Methacrylate | 5† | 5† | NA |
| Pentachloroethane | 5† | 5† | NA |
| Propionitrile | 5† | 5† | NA |
| 1,1,1,2-Tetrachloroethane | 5† | 5† | NA |
| 1,2,3-Trichloropropane | 5† | 5† | NA |
| Vinyl Acetate | 10† | 10† | NA |

Note: NA = Not available.

*Based on ESE's MDL studies conducted according to 40 CFR 136 Appendix B protocols, except for total dichlorobenzene, methyl ethyl ketone, and methyl isobutyl ketone. These compounds are difficult to analyze and do not respond well, therefore, the reporting limits have been adjusted to a concentration that is detected more reliably.

†Based on ESE's MDL studies but the reporting limits have been adjusted to a concentration that is detected more reliably.

**Based on ESE's MDL studies conducted according to 40 CFR 136 Appendix B protocols.

Source: ESE.

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Acenaphthene[a,f] | 31 | 46-118 | 19 | 31-137 |
| Acenaphthylene[f] | 30 | 33-146 | 50 | 33-146 |
| Anthracene[f] | 30 | 27-133 | 50 | 27-133 |
| Benzidine | 27[e] | 4-58[e] | 27[e] | 4-58[e] |
| Benzo(a)anthracene[f] | 30 | 33-143 | 30 | 33-143 |
| Benzo(b)fluoranthene[f] | 30 | 24-159 | 50 | 24-159 |
| Benzo(k)fluoranthene[f] | 30 | 11-162 | 50 | 11-162 |
| Benzo(a)pyrene[f] | 30 | 17-163 | 50 | 17-163 |
| Benzo(ghi)perylene[f] | 30 | 0-219 | 50 | 0-219 |
| Benzyl alcohol[f] | 14 | 65-93 | 43 | 17-103 |
| Butylbenzylphthalate[f] | 30 | 0-152 | 50 | 0-152 |
| bis(2-Chloroethyl)ether[f] | 30 | 12-158 | 50 | 12-158 |
| bis(2-Chloroethoxy)-methane[f] | 30 | 33-184 | 50 | 33-184 |
| bis(2-Ethylhexyl)-phthalate[f] | 30 | 8-158 | 50 | 8-158 |
| bis(2-Chloroisopropyl)-ether[f] | 30 | 36-166 | 50 | 36-166 |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 2 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 4-Bromophenylphenyl-ether[f] | 30 | 53-127 | 50 | 53-127 |
| Chloronaphthalene[f] | 30 | 60-118 | 50 | 60-118 |
| 2-Chlorophenol[a,f] | 40 | 27-123 | 50 | 25-102 |
| 4-Chloro-3-methylphenol[a,f] | 42 | 23-97 | 33 | 26-103 |
| 4-Chlorophenylphenylether[f] | 30 | 25-158 | 50 | 25-158 |
| Chrysene[f] | 30 | 17-168 | 50 | 17-168 |
| Dibenzo(a,h)anthracene[f] | 30 | 0-227 | 50 | 0-227 |
| di-n-Butylphthalate[f] | 30 | 1-118 | 50 | 1-118 |
| 1,3-Dichlorobenzene[f] | 30 | 0-172 | 50 | 0-172 |
| 1,2-Dichlorobenzene[f] | 30 | 32-129 | 50 | 32-129 |
| 1,4-Dichlorobenzene[a,f] | 28 | 36-97 | 27 | 28-104 |
| 3,3'-Dichlorobenzidine[f] | 30 | 0-262 | 50 | 0-262 |
| 2,4-Dichlorophenol[f] | 30 | 39-135 | 50 | 39-135 |
| Diethylphthalate[f] | 30 | 0-114 | 50 | 0-114 |
| 2,4-Dimethylphenol[f] | 30 | 32-119 | 50 | 32-119 |
| Dimethylpthalate[f] | 30 | 0-112 | 50 | 0-112 |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 3 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 2,4-Dinitrophenol[f] | 30 | 0-191 | 50 | 0-191 |
| 2,4-Dinitrotoluene[a,f] | 38 | 24-96 | 47 | 28-89 |
| 2,6-Dinitrotoluene[f] | 30 | 50-158 | 50 | 50-158 |
| di-n-Octylphthalate[f] | 30 | 4-146 | 50 | 4-146 |
| Fluoranthene[f] | 30 | 26-137 | 50 | 26-137 |
| Fluorene[f] | 30 | 59-121 | 50 | 59-121 |
| Hexachlorobenzene[f] | 30 | 0-152 | 50 | 0-152 |
| Hexachlorobutadiene[f] | 30 | 24-116 | 50 | 24-116 |
| Hexachlorocyclopentadiene[f] | 36[c] | 9-81[c] | 18[c] | 33-69[c] |
| Hexachloroethane[f] | 30 | 40-113 | 50 | 40-113 |
| Indeno(1,2,3-cd)pyrene[f] | 30 | 0-171 | 50 | 0-171 |
| Isophorone[f] | 30 | 21-196 | 50 | 21-196 |
| 2-Methyl-4,6-dinitrophenol[f] | 30 | 0-181 | 50 | 0-181 |
| Naphthalene[f] | 30 | 21-133 | 50 | 21-133 |
| Nitrobenzene [f] | 30 | 30-180 | 50 | 35-180 |
| 2-Nitrophenol[f] | 30 | 29-182 | 50 | 29-182 |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 4 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 4-Nitrophenol[a,f] | 50 | 10-80 | 50 | 11-114 |
| n-Nitrosodimethylamine[f] | 16[e] | 55-87[e] | 16[e] | 55-87[e] |
| n-Nitrosodi-n-propylamine[a,f] | 38 | 41-116 | 38 | 41-126 |
| n-Nitrosodiphenylamine[f] | 30 | 85-115 | 50 | 85-115 |
| Pentachlorophenol[a,f] | 50 | 9-103 | 47 | 17-109 |
| Phenanthrene[f] | 30 | 54-120 | 50 | 54-120 |
| Phenol[a,f] | 42 | 12-89 | 35 | 26-90 |
| Pyrene[a,f] | 31 | 26-127 | 36 | 35-142 |
| 1,2,4-Trichlorobenzene[a,f] | 28 | 39-98 | 23 | 38-107 |
| 2,4,6-Trichlorophenol[f] | 30 | 37-144 | 50 | 37-144 |
| Acetophenone[f] | 8[e] | 73-89[e] | 12[e] | 70-94[e] |
| 2-Acetylaminofluorene[f,g] | 10[e] | 23-43[e] | 11[e] | 15-37[e] |
| 4-Aminobiphenyl[f] | 14[e] | 44-72[e] | 48[e] | 5-101[e] |
| Aniline[f] | 34[e] | 51-119[e] | 74[e] | 6-154[e] |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 5 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Aramite[f,g] | 11[e] | 14-36[e] | 16[e] | 10-42[e] |
| 1,4-Benzenediamine[f,g] | 24[e] | 51-99[e] | 19 | 0-28 |
| p-Benzoquinone[f,g] | 29[e] | 19-77[e] | 49[e] | 64-162[e] |
| 4-Chloroaniline[f] | 20[e] | 48-87[e] | 89[e] | 0-149[e] |
| Chlorobenzilate[f,g] | 12[e] | 66-90[e] | 14[e] | 63-91[e] |
| 1-Chloronaphthalene[f] | 8[e] | 70-86[e] | 12[e] | 75-99[e] |
| Dibenz(a,j)acridine[f] | 19[e] | 69-107[e] | 40[e] | 0-58[e] |
| Diallate[f,g] | 8[e] | 41-57[e] | 8[e] | 43-59[e] |
| Dibenzofuran[f] | 14[e] | 71-99[e] | 42[e] | 40-124[e] |
| 2,6-Dichlorophenol[f] | 10[e] | 69-89[e] | 14[e] | 62-90[e] |
| Dimethoate[f,g] | 22[e] | 48-92[e] | 13[e] | 59-85[e] |
| p-(Dimethylamino)azo-benzene[f] | 8[e] | 57-73[e] | 15[e] | 46-76[e] |
| 7,12-Dimethylbenz(a) anthracene[f] | 12[e] | 43-67[e] | 11[e] | 12-34[e] |
| 3,3-Dimethylbenzidine[f,g] | 24[e] | 11-59[e] | 15[e] | 3-33[e] |
| m-Dinitrobenzene[f,g] | 8[e] | 32-48[e] | 15[e] | 38-68[e] |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 6 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Diphenylamine[f] | 7[e] | 68-82[e] | 14[e] | 56-85[e] |
| Diphenylhydrazine[f] | 8[e] | 67-83[e] | 16[e] | 65-97[e] |
| Ethylmethanesulfonate[f] | 7[e] | 72-86[e] | 16[e] | 64-96[e] |
| a,a-Dimethylphenylamine[f] | 93[e] | 0-149[e] | 31 | 0-41 |
| Hexachlorophene[f,g] | 51 | 138-240 | 73 | 0-71 |
| Hexachloropropene[f,g] | 17[e] | 45-79[e] | 14[e] | 54-82[e] |
| Isosafrole[f,g] | 12[e] | 64-88[e] | 28[e] | 50-106[e] |
| Methapyrilene[f,g] | 66[e] | 15-147[e] | 53 | 0-71 |
| 3-Methylcholanthrene[f] | 14[e] | 41-69[e] | 42[e] | 9-93[e] |
| Methylmethanesulfonate[f,g] | 36[e] | 35-107[e] | 24[e] | 52-100[e] |
| 2-Methylnaphthalene[f] | 17[e] | 55-89[e] | 28[e] | 49-105[e] |
| 2-Methylphenol(o-Cresol)[f] | 20[e] | 58-98[e] | 43[e] | 17-103[e] |
| 3-Methylphenol(m-Cresol)[f,g] | 8[e] | 64-80[e] | 10[e] | 59-79[e] |
| 4-Methylphenol(p-Cresol)[f] | 15[e] | 72-102[e] | 36[e] | 28-100[e] |
| 1-Naphthylamine[f] | 23[e] | 26-72[e] | 7[e] | 6-20[e] |
| 2-Naphthylamine[f] | 13[e] | 35-61[e] | 15[e] | 22-52[e] |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 7 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 2-Nitroaniline[f] | 13[e] | 45-71[e] | 18[e] | 48-84[e] |
| 3-Nitroaniline[f] | 25[e] | 28-78[e] | 22[e] | 40-84[e] |
| 4-Nitroaniline[f] | 33[e] | 63-91[e] | 45[e] | 75-165[e] |
| N-Nitrosodiethylamine[f,g] | 13[e] | 63-91[e] | 14[e] | 67-95[e] |
| N-Nitroso-di-n-butylamine[f] | 20[e] | 66-106[e] | 17[e] | 71-105[e] |
| N-Nitrosomethyl-ethylamine[f,g] | 79[e] | 13-171[e] | 23[e] | 29-75[e] |
| N-Nitrosomorpholine[f,g] | 18[e] | 54-90[e] | 22[e] | 49-93[e] |
| N-Nitrosopiperidine[f] | 15[e] | 64-94[e] | 17[e] | 57-91[e] |
| Nitroquinoline-1-oxide[f,g] | 57[e] | 0-113[e] | 53[e] | 11-117[e] |
| Nitrosopyrrolidine[f,g] | 25[e] | 52-102[e] | 16[e] | 53-85[e] |
| 1,4-Naphthoquinone[f,g] | 5[e] | 44-54[e] | 16[e] | 45-77[e] |
| 5-Nitro-o-toluidine[f,g] | 23[e] | 25-71[e] | 10[e] | 41-61[e] |
| Pentachlorobenzene[f] | 7[e] | 70-84[e] | 10[e] | 77-97[e] |
| Pentachloronitrobenzene[f] | 44[e] | 0-81[e] | 36[e] | 15-87[e] |
| Phenacetin[f] | 10[e] | 65-85[e] | 16[e] | 56-88[e] |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 8 of 9)

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| 2-Picoline[f] | 53[e] | 0-98[e] | 44[e] | 0-70[e] |
| Pronamide[f] | 15[e] | 51-81[e] | 13[e] | 57-83[e] |
| Pyridine[f,g] | 79[e] | 5-163[e] | 40[e] | 0-45[e] |
| Resorcinol[f,g] | 15[e] | 11-41[e] | 55[e] | 17-127[e] |
| 1,2,4,5-Tetrachloro-benzene[f] | 13[e] | 61-87[e] | 10[e] | 77-97[e] |
| 2,3,4,6-Tetrachlorophenol[f] | 16[e] | 48-80[e] | 12[e] | 48-72[e] |
| 2,4,5-Trichlorophenol[f] | 17[e] | 58-92[e] | 44[e] | 11-99[e] |
| 1,3,5-Trinitrobenzene[f,g] | 18[e] | 39-75[e] | 38[e] | 0-75[e] |
| o-Toluidine[f,g] | 8[e] | 71-87[e] | 13[e] | 60-86[e] |
| Safrole[f,g] | 25[e] | 50-100[e] | 23[e] | 58-104[e] |
| Nitrobenzene-D5[b] | NA | 35-114 | NA | 23-120[e] |
| 2-Fluorobiphenyl[b] | NA | 43-116 | NA | 30-115[e] |
| p-Terphenyl-D4[b] | NA | 33-141 | NA | 18-137 |
| Phenol-D6[b] | NA | 10-94 | NA | 24-113 |
| 2-Fluorophenol[b] | NA | 21-100 | NA | 25-121 |
| 2,4,6-Tribromophenol[b] | NA | 10-123 | NA | 19-122 |

Table 5-38. Analytes, Precision, and Accuracy Data for Semivolatile Orlganic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 9 of 9)

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Reference: Accuracy: EPA Method SW 8270--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986 and CLP SOW 7/87 for the controlling analytes. Precision: ESE (meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method (SW 8270) and CLP SOW7/87 for controlling analytes.

Note: N/A = spiking and recovery information is not available.
NA = not applicable.

[a]Matrix spike and QC check sample compound.
[b]Surrogate; the surrogate is added to all environmental samples and quality control samples.
[c]Accuracy and precision criteria taken from Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December, 1988.
[d]Accuracy and precision data are from ESE method certification.
[e]Accuracy and precision data are from ESE method detection limit study.
[f]Appendix IX compounds.
[g]This compound is not included in EPA's list of compounds (EPA 625, 8270) for these methods; however, it can be analyzed by this method and is reported if specifically requested by the client (see Appendix O for method validation data).

Source: ESE.

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid* (µg/kg) |
|---|---|---|
| Acenaphthene | 1.0 | 70 |
| Acenaphthylene | 1.0 | 70 |
| Anthracene | 1.0 | 70 |
| Benzidine | 20 | 2,000 |
| Benzo(a)anthracene | 1.5 | 100 |
| Benzo(b)fluoranthene | 1.5 | 100 |
| Benzo(k)fluoranthene | 1.5 | 100 |
| Benzo(a)pyrene | 2.0 | 140 |
| Benzo(ghi)perylene | 2.5 | 160 |
| Benzyl alcohol | 2.0 | 140 |
| Butylbenzylphthalate | 1.5 | 100 |
| bis(2-Chloroethyl)ether | 1.5 | 70 |
| bis(2-Chloroethoxy)methane | 1.0 | 70 |
| bis(2-Ethylhexyl)phthalate | 2.0 | 100 |
| bis(2-Chloroisopropyl)ether | 1.0 | 70 |
| 4-Bromophenylphenylether | 1.0 | 140 |
| 2-Chloronaphthalene | 1.0 | 70 |
| 2-Chlorophenol | 2.0 | 140 |
| 4-Chloro-3-methylphenol | 1.5 | 140 |
| 4-Chlorophenylphenylether | 1.0 | 100 |
| Chrysene | 1.5 | 100 |
| Dibenzo(a,h)anthracene | 2.5 | 160 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 2 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid* (µg/kg) |
|---|---|---|
| Dioxin (scan) | 2.0 | 140 |
| di-n-Butylphthalate | 1.0 | 70 |
| 1,3-Dichlorobenzene | 1.0 | 70 |
| 1,2-Dichlorobenzene | 1.0 | 70 |
| 1,4-Dichlorobenzene | 1.0 | 70 |
| 3,3'-Dichlorobenzidine | 5.0 | 500 |
| 2,4-Dichlorophenol | 2.0 | 140 |
| Diethylphthalate | 1.0 | 70 |
| 2,4-Dimethylphenol | 2.0 | 140 |
| Dimethylpthalate | 2.0 | 100 |
| 2,4-Dinitrophenol | 30.0 | 1,300 |
| 2,4-Dinitrotoluene | 2.0 | 140 |
| 2,6-Dinitrotoluene | 2.0 | 140 |
| di-n-Octylphthalate | 2.4 | 140 |
| Fluoranthene | 1.0 | 70 |
| Fluorene | 1.0 | 70 |
| Hexachlorobenzene | 2.0 | 100 |
| Hexachlorobutadiene | 2.0 | 140 |
| Hexachlorocyclopentadiene | 10 | 1000 |
| Hexachloroethane | 1.5 | 100 |
| Indeno(1,2,3-cd)pyrene | 2.5 | 160 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 3 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid* (µg/kg) |
|---|---|---|
| Isophorone | 1.0 | 140 |
| 2-Methyl-4,6-dinitrophenol | 20.0 | 1,000 |
| Naphthalene | 1.0 | 70 |
| Nitrobenzene | 1.0 | 70 |
| 2-Nitrophenol | 2.0 | 140 |
| 4-Nitrophenol | 10.0 | 500 |
| n-Nitrosodimethylamine | 1.0 | 100 |
| n-Nitrosodi-n-propylamine | 1.0 | 100 |
| n-Nitrosodiphenylamine | 1.0 | 70 |
| Pentachlorophenol | 10.0 | 500 |
| Phenanthrene | 1.0 | 70 |
| Phenol | 2.0 | 140 |
| Pyrene | 1.0 | 70 |
| 1,2,4-Trichlorobenzene | 1.0 | 100 |
| 2,4,6-Trichlorophenol | 4.5 | 300 |
| 2-Methylnaphthalene | 1.0 | 100 |
| 2,3,7,8-TCDD | 2.0 | 140 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 4 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid* (µg/kg) |
|---|---|---|
| Acetophenone | 1.0 | 70 |
| 2-Acetylaminofluorene | 2.0 | 140 |
| 4-Aminobiphenyl | 1.0 | 70 |
| Aniline | 2.0 | 140 |
| Aramite | 5.0 | 360 |
| 1,4-Benzenediamine | 30.0 | 2,000 |
| p-Benzoquinone | 10 | 300 |
| 4-Chloroaniline | 4.0 | 300 |
| Chlorobenzilate | 2.0 | 140 |
| 1-Chloronaphthalene | 1.0 | 70 |
| Dibenz(a,j)acridine | 2.0 | 140 |
| Diallate | 1.0 | 70 |
| Dibenzofuran | 1.0 | 70 |
| 2,6-Dichlorophenol | 1.0 | 70 |
| Dimethoate | 2.0 | 70 |
| p-Dimethylaminoazobenzene | 2.0 | 140 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 5 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid* (µg/kg) |
|---|---|---|
| 7,12-Dimethylbenz(a)anthracene | 2.0 | 140 |
| 3,3-Dimethylbenzidine | 2.0 | 140 |
| m-Dinitrobenzene | 2.5 | 200 |
| Diphenylamine | 1.0 | 70 |
| Diphenylhydrazine | 1.0 | 70 |
| Ethylmethanesulfonate | 1.0 | 70 |
| a,a-Dimethylphenethylamine | 11.0 | 500 |
| Hexachlorophene | 140 | 16,000 |
| Hexachloropropene | 4.0 | 140 |
| Isosafrole | 1.0 | 70 |
| Methapyrilene | 10 | 500 |
| 3-Methylcholanthrene | 2.0 | 140 |
| Methylmethanesulfonate | 1.0 | 70 |
| 2-Methynaphthalene | 1.0 | 100 |
| 2-Methylphenol (o-Cresol) | 2.0 | 140 |
| 3-Methylphenol (m-Cresol) | 2.0 | 140 |
| 4-Methylphenol (p-Cresol) | 2.0 | 140 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 6 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid* (µg/kg) |
|---|---|---|
| 1-Naphthylamine | 1.0 | 140 |
| 2-Naphthylamine | 1.0 | 140 |
| 2-Nitroaniline | 5.0 | 300 |
| 3-Nitroaniline | 5.0 | 300 |
| 4-Nitroaniline | 5.0 | 300 |
| N-Nitrosodiethylamine | 1.0 | 70 |
| N-Nitrosomethylethylamine | 2.0 | 70 |
| N-Nitroso-di-n-butylamine | 1.0 | 70 |
| N-Nitrosomorpholine | 1.0 | 70 |
| N-Nitrosopiperidine | 1.0 | 70 |
| 4-Nitroquinoline-1-oxide | 26.0 | 300 |
| Nitrosopyrrolidine | 1.0 | 70 |
| 1,4-Naphthoquinone | 2.0 | 140 |
| 5-Nitro-o-toluidine | 2.0 | 140 |
| Pentachlorobenzene | 1.0 | 70 |
| Pentachloronitrobenzene | 5.0 | 350 |

Table 5-39. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270
(Continued, Page 7 of 7)

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid* (µg/kg) |
|---|---|---|
| Phenacetin | 2.0 | 70 |
| 2-Picoline | 2.0 | 140 |
| Pronamide | 2.0 | 70 |
| Pyridine | 2.0 | 140 |
| Resorcinol | 20.0 | 700 |
| 1,2,4,5-Tetrachlorobenzene | 1.0 | 70 |
| 2,4,5-Trichlorophenol | 4.0 | 300 |
| 2,3,4,6-Tetrachlorophenol | 2.0 | 140 |
| 1,3,5-Trinitrobenzene | 2.0 | 700 |
| o-Toluidine | 1.0 | 70 |
| Safrole | 1.0 | 70 |

Note: ND = not detected.

*Based on ESE's MDL studies; however, the reporting limits of compounds that are difficult to analyze; were adjusted to a concentration that is detected more reliably. The MDL studies were conducted according to the 40 CFR 136 Appendix B protocols.

Source: ESE.

Table 5-40. Analytes, Precision, and Accuracy Data for Carbamates and Urea Pesticides, EPA 632 and Modified 632*

| Parameter | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD | Accuracy (%Recovery) | Precision (RPD) | Accuracy (%Recovery) |
| Aldicarb | NA | NA | 5 | 92-102 |
| Aldicarb sulfone | NA | NA | 4 | 64-72 |
| Aldicarb sulfoxide | NA | NA | 4 | 13-22 |
| Carbaryl (sevin)† | 55†† | 34-144†† | 43†† | 35-124†† |
| Carbofuran† | 37†† | 53-127†† | 34†† | 43-111†† |
| Diuron† | 29†† | 60-118†† | 19†† | 58-96†† |
| Linuron† | 26†† | 66-118†† | 37†† | 42-116†† |
| Methiocarb | 35 | 87-103 | 29 | 71-129 |
| Methomyl | 35 | 67-82 | 4 | 66-74 |
| Monuron | 35 | 92-102 | 23 | 82-128 |
| Oxamyl | 35 | 62-112 | 5 | 55-65 |
| Fluorometron† | 35 | 64-143 | 35 | 64-134 |
| Propoxur | 22 | 65-109 | 22 | 65-109 |
| Barban† | 12 | 86-110 | 12 | 86-110 |
| Chloropham | 12 | 83-107 | 12 | 83-107 |
| Neburon | 20 | 76-116 | 20 | 76-116 |
| Propham† | 18 | 70-106 | 18 | 70-106 |

Reference: Accuracy: EPA Method 632--Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA, January 1982.
Precision: ESE, meets or exceeds the RPD criteria that was calculated from the spiking and recovery information presented in the method (EPA 632).

Note: NA = method is not applicable.

* See Appendix A for Modified Method 632.
† Matrix spike and QC check sample compound.
** Based on the data validation study.
†† Accuracy and precision criteria are based on ESE historical data.

Source: ESE.

Table 5-41. Reporting Limit Data for Carbamates and Urea Pesticides, EPA 632 and Modified 632

| Parameter | Reporting Limits Aqueous* (µg/L) | Solid** (µg/kg) |
|---|---|---|
| Aldicarb | NA | 250 |
| Aldicarb sulfone | NA | 250 |
| Aldicarb sulfoxide | NA | 250 |
| Carbaryl (sevin) | 1.5 | 550 |
| Carbofuran | 2.5 | 1,000 |
| Diuron | 0.6 | 260 |
| Linuron | 0.65 | 270 |
| Methiocarb | 5.0 | 2,000 |
| Methomyl | 3.0 | 1,500 |
| Monuron | 0.65 | 260 |
| Oxamyl | 1.5 | 510 |
| Fluorometron | 0.5 | 200 |
| Propoxur | 4.0 | 1,700 |
| Barban | 2.0 | 940 |
| Chlorpropham | 1.5 | 700 |
| Neburon | 0.5 | 47 |
| Propham | 2.5 | 1,100 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

**Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a weight basis.

Source: ESE.

Table 5-42. Analytes, Precision, and Accuracy Data for Chlorinated Hydrocarbons, EPA 612 and SW 3510/3520/3540/3550/8120

| | Aqueous | | Solid | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| 2-Chloronaphthalene | 30 | 9-148 | 50 | 9-148 |
| 1,2-Dichlorobenzene* | 30 | 9-160 | 50 | 9-160 |
| 1,3-Dichlorobenzene | 30 | 0-150 | 50 | 0-150 |
| 1,4-Dichlorobenzene | 30 | 13-157 | 50 | 13-157 |
| Hexachlorobenzene* | 30 | 15-159 | 50 | 15-159 |
| Hexachlorobutadiene* | 30 | 0-139 | 50 | 0-139 |
| Hexachlorocyclopentadiene | 30 | 0-111 | 50 | 0-111 |
| Hexachloroethane* | 30 | 8-139 | 50 | 8-139 |
| 1,2,4-Trichlorobenzene* | 30 | 5-149 | 50 | 5-149 |

Reference: Accuracy: EPA Method SW8120--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.
Precision: ESE, meets or exceeds the RPD criteria that can be calculated from the spiking and recovery information presented in the method SW 8120.

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-43. Reporting Limit Data for Chlorinated Hydrocarbons, EPA 612 and SW 3510/3520/3540/3550/8120

| Parameter | Reporting Limits Aqueous* (µg/L) | Reporting Limits Solid† (µg/kg) |
|---|---|---|
| 2-Chloronaphthalene | 0.200 | 40.0 |
| 1,2-Dichlorobenzene | 0.100 | 20.0 |
| 1,3-Dichlorobenzene | 0.100 | 20.0 |
| 1,4-Dichlorobenzene | 0.100 | 20.0 |
| Hexachlorobenzene | 0.012 | 2.00 |
| Hexachlorobutadiene | 0.012 | 2.10 |
| Hexachlorocyclopentadiene | 0.012 | 2.40 |
| Hexachlorethane | 0.012 | 2.40 |
| 1,2,4-Trichlorobenzene | 0.15 | 2.60 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-44. Analytes, Precision, and Accuracy Data for Triazines, EPA 619

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Ametryne* | 30 | 74-134 |
| Atrazine | 30 | 78-138 |
| Prometon* | 17 | 50-84 |
| Prometryne | 51 | 59-161 |
| Propazine | 24 | 92-140 |
| Simatryn | 36 | 147-200 |
| Simazine* | 24 | 75-123 |
| Terbutylazine | 45 | 69-159 |
| Terbutryn | 36 | 47-119 |

Reference: Accuracy and Precision: EPA Method 619 -- Test Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-45. Reporting Limit Data for Triazines, EPA 619

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| Ametryn | 0.3 |
| Atrazine | 0.3 |
| Prometon | 1.2 |
| Prometryne | 0.6** |
| Propazine | 0.3** |
| Simatryn | 0.7** |
| Simazine | 0.6** |
| Terbuthylazine | 0.3** |
| Terbutryn | 0.6** |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.
**Ten times the detection limits specified in the Method EPA 619.

Source: ESE.

Table 5-46. Analytes, Precision, and Accuracy Data for Organonitrogen Pesticides, EPA 633

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Bromacil* | 40 | 82-162 |
| Deet | 6 | 99-111 |
| Hexazinone* | 23 | 78-124 |
| Metrabuzin* | 29 | 83-141 |
| Terbacil | 25 | 61-111 |
| Triadimefon | 13 | 84-110 |
| Tricyclazole | 22 | 56-100 |

Reference: Accuracy and Precision: EPA Method 633--Test Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-47. Reporting Limit Data for Organonitrogen Pesticides, EPA 633

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| Bromacil | 2.5 |
| Deet | 1.0** |
| Hexazinone | 2.5 |
| Metribuzin | 1.5 |
| Terbacil | 2.0** |
| Triadimefon | 7.0** |
| Tricyclazole | 1.0** |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.
**Ten times the detection limits listed in the Method EPA 633.

Source: ESE.

Table 5-48. Analytes, Precision, and Accuracy Data for Certain Amine Pesticides and Lethane, EPA 645

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Alachlor* | 40 | 64-144 |
| Butachlor | 25 | 68-118 |
| Diphenamid | 43 | 57-143 |
| Fluridone* | 35 | 57-127 |
| Lethane* | 60 | 33-153 |
| Norflurazon | 22 | 68-112 |

Reference: Accuracy and Precision: EPA Method 645 -- Test Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-49. Reporting Limit Data for Certain Amine Pesticides and Lethane, EPA 645

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| Alachlor | 2.0 |
| Butachlor | 3.0 |
| Diphenamid | 2.0 |
| Fluridone | 5.0 |
| Lethane | 1.0 |
| Norflurazon | 0.2 |

*Ten times the detection limits listed in the method, EPA 645.

Table 5-50. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), SW 8330

| | Aqueous† | | Solid† | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| HMX†† | 13 | 84-111 | 18 | 80-116 |
| RDX* | 30 | 51-111 | 18 | 71-107 |
| 1,3,5-Trinitrobenzene* | 28 | 46-102 | 25 | 65-115 |
| 1,3-Dinitrobenzene†† | 37 | 58-132 | 30 | 70-130 |
| Methyl-2,4,6-Trinitro-phenylnitramine (Tetryl)†† | 21 | 67-109 | 46 | 65-157 |
| Nitrobenzene* | 32 | 44-108 | 24 | 72-120 |
| 2,4,6-Trinitrotoluene* | 38 | 48-124 | 23 | 72-118 |
| 4-Amino-2,6-Dinitrotoluene†† | 26 | 66-118 | 21 | 87-126 |
| 2-Amino-4,6-Dinitrotoluene†† | 26 | 66-118 | 65 | 33-163 |
| 2,4-Dinitrotoluene* | 21 | 60-102 | 19 | 68-106 |
| 2,6-Dinitrotoluene†† | 26 | 67-119 | 44 | 58-146 |
| o-Nitrotoluene†† | 28 | 53-109 | 22 | 70-114 |
| m-Nitrotoluene†† | 48 | 40-136 | 48 | 40-136 |
| p-Nitrotoluene†† | 26 | 60-112 | 26 | 60-112 |
| 3,4-Dinitrotoluene (surrogate) | NA | 30-126 | NA | 71-143 |

*Matrix spike and QC check sample compound
†Accuracy and precision criteria based on ESE historical data, unless specified differently.
††Accuracy and precision criteria based on ESE method validation studies.

Source: ESE.

Table 5-51. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, SW 8330

| Parameter | Reporting Limit | |
|---|---|---|
| | Aqueous* (µg/L) | Solid† (µg/kg) |
| HMX | 0.25 | 500 |
| RDX | 0.25 | 500 |
| 1,3,5-Trinitrobenzene | 0.20 | 250 |
| 1,3-Dinitrobenzene | 0.10 | 200 |
| Methyl-2,4,6-Trinitro-phenylnitramine | 0.20 | 300 |
| Nitrobenzene | 0.20 | 300 |
| 4-Amino-2,6-Dinitrotoluene | 0.15 | 250 |
| 2,4,6-Trinitrotoluene | 0.15 | 250 |
| 2-Amino-4,6-Dinitrotoluene | 0.15 | 250 |
| 2,4-Dinitrotoluene | 0.15 | 150 |
| 2,6-Dinitrotoluene | 0.15 | 200 |
| o-Nitrotoluene | 0.25 | 500 |
| m-Nitrotoluene | 0.25 | 500 |
| p-Nitrotoluene | 0.30 | 600 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 50 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-52. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), AEC QA Plan 11/92**

| | Aqueous† | | Solid† | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| HMX†† | 13 | 84-111 | 18 | 80-116 |
| RDX* | 30 | 51-111 | 18 | 71-107 |
| 1,3,5-Trinitrobenzene* | 28 | 46-102 | 25 | 65-115 |
| 1,3-Dinitrobenzene†† | 37 | 58-132 | 30 | 70-130 |
| Methyl-2,4,6-Trinitro-phenylnitramine (Tetryl)†† | 21 | 67-109 | 46 | 65-157 |
| Nitrobenzene* | 32 | 44-108 | 24 | 72-120 |
| 2,4,6-Trinitrotoluene* | 38 | 48-124 | 23 | 72-118 |
| 2,4-Dinitrotoluene* | 21 | 60-102 | 19 | 68-106 |
| 2,6-Dinitrotoluene | 26 | 67-119 | 44 | 58-146 |
| o-Nitrotoluene* | 28 | 53-109 | 22 | 70-114 |
| m-Nitrotoluene†† | 48 | 40-136 | 48 | 40-136 |
| p-Nitrotoluene†† | 26 | 60-112 | 26 | 60-112 |
| 3,4-Dinitrotoluene (surrogate) | NA | 30-136 | NA | 71-145 |

*Matrix spike and QC check sample compound
†Accuracy and precision criteria based on ESE historical data, unless specified differently.
**UW32 and LW12 are ESE's USATHAMA approved methods (see Appendices H and I).
††Accuracy and precision criteria based on ESE method validation studies for Methods LW12 and UW32.

Source: ESE.

Table 5-53. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, AEC QA Plan 11/92

| Parameter | Reporting Limit Aqueous* (µg/L) | Reporting Limit Solid† (µg/kg) |
|---|---|---|
| HMX | 0.25 | 500 |
| RDX | 0.25 | 500 |
| 1,3,5-Trinitrobenzene | 0.20 | 250 |
| 1,3-Dinitrobenzene | 0.10 | 200 |
| Methyl-2,4,6-Trinitro-phenylnitramine | 0.20 | 300 |
| Nitrobenzene | 0.20 | 300 |
| 4-Amino-2,6-Dinitrotoluene | 0.15 | 250 |
| 2,4,6-Trinitrotoluene | 0.15 | 250 |
| 2-Amino-4,6-Dinitrotoluene | 0.15 | 250 |
| 2,4-Dinitrotoluene | 0.060 | 150 |
| 2,6-Dinitrotoluene | 0.070 | 200 |
| o-Nitrotoluene | 0.25 | 500 |
| m-Nitrotoluene | 0.25 | 500 |
| p-Nitrotoluene | 0.30 | 600 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 50 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-54. Analyte, Precision, and Accuracy Data for Glyphosate, EPA 547 (Modified)

| Parameter | Aqueous* Precision (RPD) | Aqueous* Accuracy (% Recovery) |
|---|---|---|
| Glyphosate | 18 | 90-126 |

*Precision and accuracy are based on method validation study performed by ESE (see Appendix V).

Source: ESE.

Table 5-55. Reporting Limit Data for Glyphosate, EPA 547 (Modified)

| Parameter | Reporting Limit Aqueous* (µg/L) |
|---|---|
| Glyphosate | 2.5 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-56. Analyte, Precision, and Accuracy Data for Ethylene-Bis-Dithiocarbamates (EBDC), EPA 630.1 (Modified)

| | Aqueous* | | Solid* | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| EBDC | 9 | 92-100 | 12 | 67-91 |

*Precision and accuracy are based on method validation studies performed by ESE (see Appendix W).

Source: ESE.

Table 5-57. Reporting Limit Data for Ethylene-Bis-Dithiocarbamates (EBDC), EPA 630.1 (Modified)

| Parameter | Reporting Limit | |
|---|---|---|
| | Aqueous* (µg/L) | Solid† (µg/kg) |
| EBDC | 5.5 | 100 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-58. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs in Water, EPA 617

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Aldrin[a] | 45[b] | 37 - 127[b] |
| BHC, A | 30 | 37 - 134 |
| BHC, B | 30 | 17 - 147 |
| BHC, D | 27 | 68 - 122 |
| BHC, G (lindane)[a] | 51[b] | 43 - 145[b] |
| Chlordane | 30 | 45 - 119 |
| DDD, PP' | 15 | 79 - 109 |
| DDE, PP' | 11 | 79 - 101 |
| DDT, PP' | 14 | 77 - 105 |
| Dieldrin | 15 | 83 - 113 |
| Endosulfan, A | 23 | 80 - 124 |
| Endosulfan, B | 14 | 79 - 107 |
| Endosulfan sulfate | 30 | 26 - 144 |
| Endrin[a] | 60[b] | 35 - 155[b] |
| Endrin aldehyde | 40[e] | 58 - 138[e] |
| Heptachlor[a] | 38[b] | 48 - 124[b] |
| Heptachlor epoxide | 12 | 82 - 106 |
| Methoxychlor | 20 | 77 - 117 |
| Toxaphene | 30 | 41 - 126 |
| Mirex | 14 | 75 - 103 |
| Trifluralin | 32 | 62 - 126 |
| PCNB | 19 | 64 - 102 |
| PCB-1061[d] | 30 | 50 - 114 |
| PCB-1221 | 30 | 15 - 178 |
| PCB-1232 | 30 | 10 - 215 |
| PCB-1242 | 30 | 39 - 150 |
| PCB-1248 | 30 | 38 - 158 |
| PCB-1254 | 30 | 29 - 131 |
| PCB-1260[d] | 30 | 8 - 127 |
| Dibutylchlorendate[c] | NA | 46 - 146[b] |
| Decachlorobiphenyl (surrogate) | NA | 12-140 |
| Tetrachlorobiphenyl (surrogate) | NA | 33-119 |

Table 5-58. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs in Water, EPA 617 (Continued, Page 2 of 2)

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Reference: Accuracy and Precision: EPA Methods 617 and SW 8080.

Note: NA = not applicable.

[a]Matrix spike and QC check sample compound.
[b]Accuracy and precision criteria are based on ESE historical data.
[c]Surrogate; the surrogate is added to all environmental samples and quality control samples.
[d]PCB 1016 and PCB 1260 are only used as matrix spike and QCC samples compounds when using EPA 608/8080 to evaluate PCBs only.

Source: ESE.

Table 5-59. Reporting Limit Data for Organochlorine Pesticides and PCBs in Water, EPA 617

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| Aldrin | 0.006 |
| BHC,A | 0.006 |
| BHC,B | 0.006 |
| BHC,D | 0.006 |
| BHC,G(lindane) | 0.006 |
| Chlordane | 0.030 |
| DDD,PP | 0.006 |
| DDE,PP' | 0.006 |
| DDT,PP' | 0.006 |
| Dieldrin | 0.006 |
| Endosulfan,A | 0.006 |
| Endosulfan,B | 0.006 |
| Endosulfan sulfate | 0.006 |
| Endrin | 0.006 |
| Endrin aldehyde | 0.006 |
| Heptachlor | 0.006 |
| Heptachlor epoxide | 0.006 |
| Methoxychlor | 0.006 |
| Toxaphene | 0.6 |
| PCB-1016 | 0.12 |
| Mirex | 0.2 |
| Trifluralin | 0.1 |
| PCNB | 0.02 |

Table 5-59. Reporting Limit Data for Organochlorine Pesticides and PCBs in Water, EPA 617 (Continued, Page 2 of 2)

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| PCB-1221 | 0.12 |
| PCB-1232 | 0.12 |
| PCB-1242 | 0.12 |
| PCB-1248 | 0.12 |
| PCB-1254 | 0.12 |
| PCB-1260 | 0.12 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-60. Analyte, Precision, and Accuracy Data for Tetrazene, EPA 8331

| Parameter | Aqueous* | | Solid* | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| Tetrazene | 10 | 94-114 | 25 | 35-85 |

*Precision and accuracy are based on EPA Method 8331--Test Methods for Evaluating Solid Waste, EPA SW-846, 3rd Edition, September 1986.

Source: ESE.

Table 5-61. Reporting Limit Data for Tetrazene, EPA 8331

| Parameter | Reporting Limit | |
|---|---|---|
| | Aqueous* (µg/L) | Solid (µg/kg) |
| Tetrazene | 7 | 1,000 |

Source: ESE.

Table 5-62. Analytes, Precision, and Accuracy Data for Non-Halogenated Volatile Organic Compounds, EPA 8015 Modified B

| Parameter | Aqueous | |
|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) |
| Isobutanol | 32 | 56-120 |
| 2-Ethoxyethanol | 17 | 85-109 |

*Accuracy and precision criteria are based on ESE historical data.

Source: ESE.

Table 5-63. Reporting Limit Data for Non-Halogenated Volatile Organic Compounds, EPA 8015 Modified B

| Parameter | Reporting Limits Aqueous* (µg/L) |
|---|---|
| Isobutanol | 500 |
| 2-Ethoxyethanol | 5,000 |

*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-64. Analytes, Precision, and Accuracy Data for Total Petroleum Hydrocarbons (TPH), EPA 8015 Modified A

| Parameter | Aqueous* | | Solid* | |
|---|---|---|---|---|
| | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) |
| TPH as Gas | 22+ | 53-971+ | 8+ | 80-96+ |
| TPH as Diesel | 30 | 61-121 | 38 | 56-132 |
| Pentacosane (surrogate) | NA | 52-168 | NA | 54-158 |

Note: NA = not applicable.

*Accuracy and precision based on ESE historical data.
+Accuracy and precision criteria based on method validation data.

Source: ESE.

Table 5-65. Reporting Limit Data for Non-Halogenated Volatile Organic Compounds, EPA 8015 Modified A

| Parameter | Reporting Limit Aqueous* (µg/L) | Reporting Limit Solid† (µg/kg) |
|---|---|---|
| TPH as Gas | 400 | 8,000 |
| TPH as Diesel | 400 | 8,000 |

*Base on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standrd is chosen to be within 5 to 10 times the background noise of the instrument.
†Base on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standrd is chosen to be within 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-64. Precision and Accuracy Data for Nitrocelloluse by Autoanalyzer, UF03 and LF03*

| Parameter | Aqueous+ Precision (RPD) | Aqueous+ Accuracy (% Recovery) | Solid- Precision (RPD) | Solid- Accuracy (% Recovery) |
|---|---|---|---|---|
| Nitrocelllulose | 39 | 39-117 | 29 | 6-64 |

*UF03 and LF03 are ESE's USATHAMA-approved methods.
+Accuracy and precision criteria based on method validation data.

Source: ESE.

Table 5-65. Reporting Limit Data for Nitrocellulose, UF03 and LF03

| Parameter | Reporting Limit Aqueous* (µg/L) | Reporting Limit Solid† (µg/kg) |
|---|---|---|
| Nitrocellulose | 553 | 10,500 |

*Base on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standrd is chosen to be within 5 to 10 times the bachground noise of the instrument.
†Base on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standrd is chosen to be within 5 to 10 times the bachground noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

# 6.0 SAMPLE HANDLING PROCEDURES

## 6.1 INTRODUCTION

The validity of analytical data is dependent on the integrity of the field procedures employed in obtaining a sample. Environmental sampling has many variables which can affect analytical results. The properties of most contaminated materials warrant the analysis of a small aliquot of the bulk of the material. Proper sampling techniques must be employed to obtain a representative sample of the bulk material. For a sample to be representative, it must be collected and handled so as to keep its original physical form and chemical composition as much as possible and to protect against any loss or contamination. To achieve this sample's integrity, quality assurance procedures should be closely followed throughout the sampling effort.

Exhaustive and sometimes expensive actions are taken based on the analytical data generated from field sampling programs. Therefore, it is in the best interest of the investigation, as well as the public, to ensure the quality of the data by ensuring the quality of the samples being delivered to the analyst.

This section of the LCQAP details sample handling requirements in the laboratory.

## 6.2 SAMPLE CONTAINERS CLEANING PROCEDURES

### 6.2.1 CLEANING PROCEDURES

ESE uses commercially cleaned sample containers whenever practical. Only Type 300 series precleaned sample containers provided with certificate of cleanliness will be used. The certificates will be kept on file. Table 6-1 summarizes the application of these cleaning procedures. Cleaned sample containers are stored in a secured

Table 6-1. Sample Container Cleaning Procedures Within the Laboratory

| Analysis/Parameter | Container Type | Matrix | Fraction Code | Cleaning Protocol* |
|---|---|---|---|---|
| Organic extractables include GC, HPLC, GC/MS, and Total Phenols Analyses | Glass jar with Teflon®-lined cap | Water | MS, EC, HB, UP, NP, LC, W, Z | A |
| | Glass jar with Teflon®-lined cap | Soil/Sediment | SS | A |
| | Aluminum foil and plastic bags | Tissue* | TS | NA |
| Organic purgeables including GC and GC/MS Analyses, TOX, Aldicarb | Glass septum vial with Teflon®-lined cup | Water | V, VP, ED, AL, XP | B |
| | Wide-mouth glass jar with Teflon®-lined cap | Soil | SV | B |
| | Aluminum foil and plastic bags | Tissue* | TS | NA |
| Metals | Linear polyethylene cubitainer with polyethylene cap | Water | N | C |
| | Glass jar with Teflon®-lined cap (or new plastic) | Soil/Sediment | SS | A |
| | Plastic bags | Tissue* | TS | NA |
| Inorganics include total cyanide, alkalinity, acidity, residues, BOD, color, MBAS, COD, TOC, chloride, turbidity, sulfate, bromide, sulfide, fluoride, nutrients, and radionuclides | Linear polyethylene cubitainer with polyethylene cap | Water | C, B, S, H, R | D* |
| | Glass jar with Teflon®-lined cap (or new plastic) | Soil | SS | A |
| | Aluminum foil and plastic bags | Tissue* | TS | NA |
| Oil and grease (O&G), odor | Glass jar with Teflon®-lined cap | Water | O,OD | A |
| Oil and grease (O&G) | Glass jar with Teflon®-lined cap | Soil/ Sediment | SS | A |
| Toxicity tests | Linear polyethylene cubitainer with polyethylene cap | Effluent | None | D |

Table 6-1. Sample Container Cleaning Procedures Within the Laboratory (Continued, Page 2 of 2)

Note:
BOD = biochemical oxygen demand.
COD = chemical oxygen demand.
GC/MS = gas chromatography/mass spectrometry.
GC/HPLC = gas chromatography/high performance liquid chromatography.
Glass = amber for all organic water analyses.
MBAS = methylene blue active substance.
NA = not applicable.
TOC = total organic carbons.
TOX = total organic halide.

*Tissue samples for these parameters are first wrapped in aluminum foil and then put in plastic bags.

| *Cleaning Protocol A | B | C | D | Specifications |
|---|---|---|---|---|
| X | X | X | | Wash with hot tap water using laboratory-grade, interference free, nonphosphate detergent. |
| X | X | X | | Rinse 3 times with tap water. |
| X | | X | | Rinse with 1:1 nitric acid (reagent-grade nitric acid diluted with ASTM Type 1 deionized water). |
| X | X | X | | Rinse 3 times with ASTM Type 1 deionized water. |
| X | | | | Rinse with pesticide-grade methylene chloride using 20 mL per 64-oz bottle, 10 mL per 32- or 16-oz bottle, or 5 mL per 8- or 4-oz bottle. Methylene chloride is used as organics rinse. |
| X | X | | | Oven dry, using a forced-air oven, at 105° to 125°C for 1 hour. |
| | | X | | Invert and air-dry in contaminant-free environment. |
| X | X | X | | The containers are sealed with caps containing Teflon® liners or Teflon®-backed septa that had been cleaned the same way as the containers, packed in cartons, and stored until needed. |
| | | | X | No cleaning required; use new cubitainers (only). |

Note: Cleaning protocols A, B, and C are applied by commercial supplier.
Cleaning protocol D is applied by ESE.
Source: ESE.

storage building away from the analytical laboratory until needed. Activities and records associated with contaminant-free containers are maintained.

All sample containers are stored in a secured storage building located away from the laboratory. When containers are needed, they are moved to the sample kit preparation area that is also located away from the laboratory and packed for shipment to the project site. Upon receipt of precleaned sample containers, the purchase order form is dated with date of receipt by the laboratory purchasing personnel and the purchase order form is filed. Documentation associated with the sample containers such as lot numbers and certification statements for 300 series containers are maintained and filed in their department by the kit preparation personnel. These containers are labeled individually with lot numbers, hence, it is not necessary to maintain records of lot numbers used for a particular project.

### 6.2.2 TYPES OF WATER

DI water is defined as ESE water that has been treated by passing it through a standard resin column and an activated carbon unit. The water contains no detectable (i.e., ESE's routine detection limits) heavy metals or inorganic compounds of analytical interest and is relatively free of organic compounds. The water is acceptable for use in the initial rinsing of laboratory glassware and field equipment. Ultrapure water, used for equipment and field blanks, is defined as ESE DI water that has been additionally treated through a Milli-Q® treatment system and contains no organic compounds of analytical interest above ESE's routine detection limits. Organic-free water, used for trip blanks, is prepared by purging American Society for Testing and Materials (ASTM) Type 2 water at 60°C for 24 hours with Grade 6 helium.

Documentation is maintained to demonstrate reliability and "purity" of analyte free water source(s).

DI water other than ESE-treated water may be used if it is of documented equivalent quality. Use of commercially DI or distilled water is discouraged because it often contains phthalate esters.

## 6.3 SAMPLING CONTAINERS, VOLUMES, HOLDING TIMES AND PRESERVATION

### 6.3.1 CONTAINERS AND SAMPLE HOLDING TIMES

Table 6-2 identifies the proper containers, preservation techniques, and maximum holding times established by EPA (40 CFR Part 136). The maximum holding times in Table 6-1 apply to water and soils as noted. Samples that exceed the regulatory holding times will be flagged by the laboratory coordinator in the final deliverable.

### 6.3.2 SAMPLE PRESERVATION

Sample preservation is generally performed in the field. However, sample containers for volatile analysis (water only) are sent to the field with preservatives added to the containers. Sample preservation requirements are listed in Table 6-2.

Grades of the preservatives used are specified as a footnote in Table 6-2. Fresh preservatives are obtained from laboratory stocks prior to each sampling event.

## 6.4 SAMPLE SHIPPING FROM THE FIELD TO THE LABORATORY

A typical environmental sample consists of some type of soil or water matrix; however, other types of samples such as tissues or dust wipes are also collected. Whatever the field sample type, the field crew should package each sample container to ensure its integrity inside the shipping container. This packaging may include packing materials such as Bubble Wrap® or styrofoam fillers.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times

| Measurement | Container[1] | Preservation | Maximum Holding Time[2] (Waters and Soils) |
|---|---|---|---|
| Metals | | | |
| Chromium VI | P, G | Cool, 4°C | 24 hours[3] |
| Mercury | P, G | $HNO_3$ to pH < 2 | 28 days |
| Metals, except chromium VI and mercury (filtered and unfiltered) | P, G | $HNO_3$ to pH < 2 | 6 months |
| Inorganic Tests | | | |
| Acidity | P, G | Cool, 4°C | 14 days[3] |
| Alkalinity | P, G | Cool, 4°C | 14 days[3] |
| Ammonia | P, G | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days[3] |
| BOD | P, G | Cool, 4°C | 48 hours[3] |
| Bromide | P, G | None required | 28 days[3] |
| BOD, carbonaceous | P, G | Cool, 4° | 48 hours[3] |
| COD | P, G | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days[3] |
| Chloride | P, G | None required | 28 days[3] |
| Chlorine, total | P, G | None required | Analyze immediately[3,7] |
| Color | P, G | Cool, 4°C | 48 hours[3] |
| Cyanide, total and amenable to chlorination | P, G | Cool, 4°C, NaOH to pH > 12, 0.6 g ascorbic acid[8] | 14 days[3,8] |
| Fluoride | P | None required | 28 days[3] |
| Hardness | P, G | $HNO_3$ to pH < 2, $H_2SO_4$ to pH < 2 | 6 months[3] |
| Hydrogen ion (pH) | P, G | Cool, 4°C | Analyze immediately[3] |
| Kjeldahl and organic nitrogen | P,G | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days[3] |
| Nitrate | P, G | Cool, 4°C | 48 hours[3] |
| Nitrate (drinking water) | | | |
| Chlorinated | P, G | Cool, 4°C | 28 days |
| Unchlorinated[12] | P, G | $H_2SO_4$ to pH < 2 | 14 days |
| Nitrate-nitrite | P, G | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days[3] |
| Nitrite | P, G | Cool, 4°C | 48 hours[3] |
| Oil and grease | G | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days[3] |
| Organic carbon | P, G | Cool, 4°C, HCl or $H_2SO_4$ to pH < 2 | 28 days[3] |

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 2 of 5)

| Measurement | Container[1] | Preservation | Maximum Holding Time[2] (Waters and Soils) |
|---|---|---|---|
| Inorganics (cont) | | | |
| Orthophosphate | P, G | Filter immediately, cool, 4°C | 48 hours[3] |
| Oxygen, dissolved (DO) | | | |
| Probe | G Bottle and top | None required | Analyze immediately[3] |
| Winkler | G Bottle and top | Fix onsite and store in dark | 8 hours[3] Phenols |
| | G only | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days[3] |
| Phosphorus (elemental) | | G | Cool, 4°C48 hours[3] |
| Phosphorus, total | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days[3] |
| MBAS | P, G | Cool, 4°C | 48 hours[3] |
| Bromates (ion chromatography) | P, G | Cool, 4°C | 30 days[3] |
| Corrosivity (calculated) | P, G | Cool, 4°C | 7 days[9] |
| Odor | G | Cool, 4°C | 6 hours[3] |
| Unionized Ammonia (calculated) | P, G | Cool, 4°C | 8 hours[3] |
| | | $Na_2S_2O_3$ | 28 days[10] |
| Residue, total | P, G | Cool, 4°C | 7 days[3] |
| Residue, filterable | P, G | Cool, 4°C | 7 days[3] |
| Residue, nonfilterable (TSS) | P, G | Cool, 4°C | 7 days[3] |
| Residue, settleable | P, G | Cool, 4°C | 48 hours[3] |
| Residue, volatile | P, G | Cool, 4°C | 7 days[3] |
| Silica | P | Cool, 4°C | 28 days[3] |
| Specific conductance | P, G | Cool, 4°C | 28 days[3] |
| Sulfate | P, G | Cool, 4°C | 28 days[3] |
| Sulfide | P, G | Cool, 4°C, add zinc acetate plus NaOH to pH>9 | 7 days[3] |
| Sulfite | P, G | None required | Analyze immediately[3] |
| Surfactants | P, G | Cool, 4°C | 48 hours[3] |
| Temperature | P, G | None required | Analyze immediately[3] |
| Turbidity | P, G | Cool, 4°C | 48 hours[3] |
| Organic Tests | | | |
| Purgeable halocarbons | G, Teflon®-lined septum | Cool, 4°C, 0.008% $Na_2S_2O_3$[5,6] store in dark | 14 days |

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 3 of 5)

| Measurement | Container[1] | Preservation | Maximum Holding Time[2] (Waters and Soils) |
|---|---|---|---|
| Organics (cont) | | | |
| Purgeable aromatic hydrocarbons | G, Teflon®-lined septum | Cool, 4°C, 0.008% $Na_2S_2O_3$[5,6] HCl to pH2 | 14 days |
| Acrolein and acrylonitrile | G, Teflon®-lined septum | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] Adjust pH to 4-5 | 14 days |
| Phenols | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| Benzidines | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark | 7/40 days for waters[4] |
| Phthalate esters | G, Teflon®-lined cap | Cool, 4°C, store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| Nitrosamines | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| PCBs, pesticides, herbicides | G, Teflon®-lined cap | Cool, 4°C, 0.008% $NA_2S_2O3$[5] store in dark | 7/40 days for water[4] 14/40 days for soils[4] |
| Nitroaromatics and isophorone | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark | 7/40 days for water[4] 14/40 days for soils[4] |
| Polynuclear aromatic hydrocarbons | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| Haloethers | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[6] store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| Volatile organics | G, Teflon®-lined septum | Cool, 4°C, 0.008% $Na_2SO_3$[6] HCL to pH 2 | 14 days |
| EDB, DBCP | G, Teflon®-lined septum | Cool, 4°C, 0.008% $Na_2S_2O_3$[6] | 28 days |
| Chlorinated hydrocarbons | G, Teflon®-lined cap | Cool, 4°C, store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| TCDD | G, Teflon®-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$[6] store in dark | 7/40 days for waters[4] 14/40 days for soils[4] |
| Total organic halogens pH <2 (TOX) | 28 days[3] cap | G, Teflon®-lined store in dark | Cool, 4°C, $H_2SO_4$ to |

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 4 of 5)

| Measurement | Container[1] | Preservation | Maximum Holding Time[2] (Waters and Soils) |
|---|---|---|---|
| Organics (cont) | | | |
| Acid and base/neutral | G, Teflon®-lined | | Cool, 4°C, 0.008% |
| $Na_2S_2O_3$[6] | 7/40 days for waters[4] | | |
| extractables | cap | store in dark | 14/40 days for soils[4] |
| Nitroaromatics and | G, Teflon®-lined | | Cool, 4°C7/40 days for |
| waters[4] | | | |
| Nitramines | cap | | 14/40 days for soils[4] |
| Radiological Tests | | | |
| Alpha, beta, Sr-90, Ra-226, Ra-228, Uranium, photon emitters | P, G | HCL, $HNO_3$ to pH<2 | 6 months |
| Cesium-134, Iodine-131, Titrium | P, G | None | 6 months |
| Tissues | | | |
| Organics, inorganics and radiological tests | Aluminum foil and plastic bag | Freeze, -20°C | 12 months or below |
| Metals tests | Plastic bag | Freeze, -20°C or below | 12 months |

Note: BOD = biochemical oxygen demand. $Na_2SO_3$ = sodium sulfite (ACS grade).
COD = chemical oxygen demand. $Na_2S_2O_3$ = sodium thiosulfate (ACS grade).
G = glass. P = polyethylene.
HCl = hydrochloric acid (metals grade). PCB = polychlorinated biphenyl.
$HNO_3$ = nitric acid (metals grade). NaOH = sodium hydroxide (ACS grade).
$H_2SO_4$ = sulfuric acid (metals grade). °C = degrees Celsius.
NS = none specified by EPA.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 5 of 5)

[1] For nonvolatile organics, containers for soil and sediment samples are glass with Teflon®-lined caps and for volatiles, containers are glass with Teflon®-lined septum.
Soil sample containers for inorganics are glass jars with Teflon®-lined caps, polyethylene (P), or glass (G).

[2] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the laboratory has data on file to show that the specific types of samples under study are stable for the longer time.

[3] Holding times provided are for waters. EPA does not have holding times for these parameters in soil. These water holding times will be used as goals for those methods where a soil analysis is applicable.

[4] 7/40 = 7 days until extraction; 40 days from extraction until analysis. 14/40 = 14 days until extraction; 40 days from extraction until analysis.

[5] Sample preservation should be performed immediately upon sample collection. The only preservation for soil samples is cooling at 4°C. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed (maximum allowable time is 20 hours). $Na_2S_2O_3$ is used only in the presence of residual chlorine.

[6] If residual chlorine is present, sodium thiosulfate is added to the sample vial first, the vial is then filled to almost full volume with sample, acid is added, and finally the vial is filled. Note: It is not recommended to mix the two preservatives (and sample) together in an intermediate vessel.

[7] These parameters are best analyzed in the field. In consideration of shipping limitations, when these analyses are requested of our laboratory for confirmation purposes, ESE's policy is to analyze these constituents within 24 hours of receipt.

[8] The following test should be performed for cyanide samples:
(a) Oxidizing agents--Test the sample using KI-starch paper. If present, add a few crystals of ascorbic acid and test until negative. Add an additional 0.6 gram of ascorbic acid for each liter of sample to remove the chlorine.
(b) Sulfides--When sulfide is present as indicated by a positive test with lead acetate paper, the maximum holding time is 24 hours. Remove the sulfides by (1) filtration of sample if visible particulates are present, (2) precipitation with cadmium nitrate until a negative spot test is obtain, (3) filtration of the precipitate, and (4) addition of NaOH to pH >12 if sulfides are not removed with the previous procedure.

[9] Temperature and pH must be measured onsite at the time of sample collection. Seven days is the maximum time for laboratory analysis of total alkalinity, calcium ion, and total solids.

[10] The results of the measurements of pH, temperature, salinity (if applicable), and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the un-ionized state. Temperature, pH, and salinity must be measured onsite at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within 8 hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with $H_2SO_4$ to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

[11] Chlorinated means that the source water has a detectable amount of residual chlorine, as will be indicated by the field test.

[12] Nonchlorinated means that the source water contains no detectable amount of residual chlorine (i.e., below the detection limit).

Source: ESE.

Sample containers are typically shipped by bonded courier to the laboratory. Samples are shipped by overnight delivery as soon as possible after collection (usually daily), with receiving signature required. Sample receipt and check-in at the laboratory is performed by the sample custodian, as described in Section 7.3.

Samples are usually organized by sample location in each shipping container with all of the fractions collected from a given station grouped together. A possible exception to this procedure would include the collection of large quantities of samples for VOC analyses.

If the samples require chilling/freezing, the sample containers will be isolated from the chilling/freezing materials using appropriate, waterproof materials such as plastic garbage bags. Typically, wet ice is used to chill the samples; reusable blue ice-type chilling products will not be used due to possible chemical interferences. If a sample must be kept frozen in a solid state, dry ice is used.

The chain-of-custody logsheet for the samples in each shipping container is sealed in a plastic Ziploc® bag and taped to the inside of the container. ESE's policy requires sealing all sample shipping containers with evidence tape prior to shipping.

## 6.5 REAGENT AND STANDARD STORAGE

Storage requirements of reagents and standards used are presented in Table 6-3.

Table 6-3. Reagent Storage

| Reagent | Method of Storage |
|---|---|
| Solvents | Stored in original containers in a vented storage room, or stored in double-walled flammable liquid storage cabinets. Stockroom personnel check the storage cabinets daily and tranfer solvents from the storage room to the storage cabinets as needed. Note: Methanol used for volatile organic analyses are stored in the GC-Volatiles and GC/MS-Volatiles analysis areas. |
| Inorganic acids | Stored in original containers in the ESE stockroom. Once issued from the stockroom to a department, the acids are kept in safety carriers and stored along with the carriers in the department's cabinet designated for acids only. |
| Organic acids | Stored in original containers in the ESE stockroom. Once issued from the stockroom to a deparment, the acids are kept in safety carriers and stored along with the carriers in the department's cabinet designated for acids only. Note: Organic acids are stored in separate cabinets from the inorganic acids. |
| Caustics | Stored in original containers in the ESE stockroom. Once issued from the stockroom to a department, the caustic reagents are kept in safety carriers and stored along with carriers in the department's cabinet designated for caustics only. Note: Caustic reagents are stored in separate cabinets from the acids. |
| Other reagents | Stored in the main chemical or standards storage room, or stored in the designated cabinets in each department. Liquids in quantities of one gallon or more must be kept in safety carriers. Standards that require storage at 4°C or at 0°C are stored in each department's refrigerators or freezers (respectively) designated for standards only. |

Source: ESE.

# 7.0 SAMPLE CUSTODY

## 7.1 SAMPLE CUSTODY OBJECTIVES

The primary objective of sample custody is to create an accurate written verified record that can be used to trace the possession and handling of the samples from the moment of collection until receipt by the laboratory. Adequate sample custody in the laboratory will be achieved by means of approved laboratory documentation.

### 7.1.1 DEFINITION OF LEGAL CHAIN OF CUSTODY

A sample for this project is defined to be in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked or otherwise sealed so that tampering will be evident; or
4. It is kept in a secure area, restricted to authorized personnel only.

### 7.1.2 LEGAL CUSTODY PROCEDURES

1. Formal chain of custody starts when the precleaned sample containers are dispatched to the field. The sample kit preparation personnel initiate custody of the sample containers by completing the first line under the "Relinquish By" of the chain-of-custody logsheet (Figure 7-5). Receipt of the sample containers are acknowledged by the field personnel by signing and dating the first line under the "Received By" of the logsheet.
2. The formal chain of custody is signed by the Laboratory Coordinator in the laboratory. At the field, the Field Team Leader or his designate, is responsible to ensure that the chain-of-custody form is maintained.
3. Copies of the chain-of-custody form and/or field sheets are maintained with project records.
4. Errors in all documents are corrected by drawing one line though the error, then signing, and dating the corrections.
5. All documentation/logs are signed/initialed by appropriate personnel.

Due to evidentiary nature of the samples collected, possession must be traceable from the time the precleaned containers leave the laboratory to the field. Field chain of custody actually begins at the laboratory. Sample kits, which refer to the coolers, sample containers, preservatives, and trip blanks, are requested from the kit preparation staff using the Sample Kit Request Form (Figure 7-1). This form is completed by the Laboratory Coordinator and accompanied by the field group logsheets, labels, and any other relevant information. Shipping labels and/or the ESE Shipping Request Form (87201.A) (Figure 7-2) are provided in accordance with current corporate policy on sample kit handling.

The preservatives are packed in fiberboard boxes filled with vermiculites (inert materials compatible with both acids and alkalis) and labeled showing type of preservatives used.

The sample containers, boxed preservatives, trip blanks, if needed, chain-of-custody field logsheet, and a copy of the sample fraction codes are packed in coolers, sealed and shipped to the field by bonded carrier (i.e., UPS or Federal Express). All kit request forms are signed and dated upon completion by kit preparation staff. The number of coolers shipped to the field is documented in the shipping receipts that are kept in a central file located in the Gainesville, Florida, ESE Accounting office. Coolers that are picked up by the field personnel are logged out from the sample kit preparation staff using the Kit Pick-up Log in Figure 7-3. An ESE cooler tracking report indicating the personnel who prepared the kits, cooler number(s), project name and number, contents of each cooler, and the time and date the cooler was released is generated. This report is attached to the kit request form, a copy is packed in the cooler along with the other documentation, and the original filed by the kit preparation staff for future reference. An example of the cooler tracking report is shown in Figure 7-4.

FROM: _____ Date Submitted: _____

PROJECT NAME: _____ Time Submitted: _____

FIELD GROUP(s) / SAMPLE #(s): _____

TRIP BLANKS? Y / N : TYPE: __ GCV __ GCMS # OF: VP _____ V _____ ED _____ Other: _____

QC LABELS REQUIRED? Y / N : IF SO, SAMPLE #'s TO BE MARKED: _____

IF VP FRACTIONS ARE INCLUDED, SHOULD THEY BE PREPRESERVED? Y / N (IF NO, REMEMBER HCL)

TYPE AND # OF REQUIRED PRESERVATIVE BOTTLES (25 mL PER BOTTLE):

_____Sulfuric _____Nitric (18%) _____Hydrochloric _____Zinc Acetate _____Sodium Hydroxide

_____Sodium Thiosulfate _____Sodium Sulfite _____Cadmium Nitrate _____Lead Acetate strips

OTHER SUPPLIES: _Y_ Logsheets _Y_ Trash Bags (for ice) ___Gloves ___Evidence Tape ___Pipettes ___pH Strips

(# of) _____Gals of Barnstead water ___Coolers of Blue Ice Other: _____

FOR PICKUP BY _____ ON __/__/__ OR

SHIP KITS ON: __/__/__ OR MAY SHIP BETWEEN __/__/__ AND __/__/__

TO: _____
Name

_____
Company

_____
Street Address for Delivery (NO P.O. Boxes)

_____
City State Zip

Telephone #(____) _____

Project # _____

SERVICE REQUIRED

Saturday Delivery Required?_____

___ Florida Overnight

___ Fed-X: ___ Next AM ___ Next PM ___ 2-Day/Std

___ UPS: ___ Next Day ___ 2-Day ___ 3-Day

___ Ground (1-2 Day in Fla.)

___ Bus: type service? _____

___ Other: _____

Kits Completed By_____ on __/__/__

_____ # of Coolers

_____ # of Separate Cartons

Transferred to _____ Mailroom _____ Facilities

By _____

On __/__/__

To Arrive on __/__/__ ____ AM ____ PM

Notes: _____

| | | | |
|---|---|---|---|
| 1)_____ | 11)_____ | 21)_____ | 31)_____ |
| 2)_____ | 12)_____ | 22)_____ | 32)_____ |
| 3)_____ | 13)_____ | 23)_____ | 33)_____ |
| 4)_____ | 14)_____ | 24)_____ | 34)_____ |
| 5)_____ | 15)_____ | 25)_____ | 35)_____ |
| 6)_____ | 16)_____ | 26)_____ | 36)_____ |
| 7)_____ | 17)_____ | 27)_____ | 37)_____ |
| 8)_____ | 18)_____ | 28)_____ | 38)_____ |
| 9)_____ | 19)_____ | 29)_____ | 39)_____ |
| 10)_____ | 20)_____ | 30)_____ | 40)_____ |

**Figure 7-1**
**SAMPLE KIT PREP & SHIPPING REQUEST FORM**

SOURCE: ESE

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

# ESE SHIPPING REQUEST

DATE:_____

FROM:_____ EXT:_____

PROJECT/PROPOSAL/CHARGE NO.:_____

TO:_____
Name

_____
Company

_____
Street Address for UPS, Federal Express-(NO P.O. Boxes)

_____
City State Zip

( )_____
Telephone

DELIVERY TIME-PACKAGE MUST REACH DESTINATION BY:

_____ _____
Date Time

SATURDAY SERVICE REQUIRED ☐

DECLARED VALUE $_____

WEIGHT (for more than one package, see other side)_____

TYPE OF SERVICE REQUIRED:

☐ 1st CLASS MAIL
☐ UPS GROUND
☐ UPS 2-DAY
☐ UPS NEXT DAY
☐ FEDERAL EXPRESS 2-DAY
☐ FEDERAL EXPRESS NEXT DAY
☐ DHL
☐ OTHER_____

FORM 87201.A

**Figure 7-2**
**ESE SHIPPING REQUEST FORM**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

## KIT PICK-UP LOG

**This log must be filled out whenever kits are not shipped by UPS or FED-X.**

| Project Name | Field Group | # of Coolers | Relinquished By/Date | Received By/Date |
|---|---|---|---|---|
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**Figure 7-3**
**KIT PICK-UP LOG**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

TB 8232 BL

ESE SAMPLE TRACKING-- ICE CHEST CHECK OUT (A100)
PRINTED ON 17:02:17 03 DEC 1992 PAGE 1

| LMP | COOLER | TO | TIME | COMMENT |
|---|---|---|---|---|
| E03289 | COOL0287 | 3904076-0214-3200 | 11:25 | SITE ID 1,- 11 GLOVES; LOGSHEETS; PIPETTES; TRASH BAGS; 1 GALLON BARNSTEAD WATER |
| E01421 | COOL0471 | 3924031G-0202-3200 | 16:31 | SITE ID 1 - 5 ; LOGSHEETS; TRASH BAGS; |
| E03291 | COOL0553 | 3924031G-0202-3200 | 11:25 | SITE ID 1 - 19 TRASH BAGS; GLOVES; LOGSHEETS; |
| E03180 | COOL0167 | 3924062-0800-3200 | 13:29 | 2 BOX(ES); 500 ML; 1 BOX(ES); 60 ML; BUBBLE WRAP |
| E03180 | COOL0499 | 3924062-0800-3200 | 16:27 | 2 BOX(ES); 500 ML; 1 BOX(ES); 60 ML; BUBBLE WRAP; |
| E03180 | COOL0217 | 3924062-0800-3200 | 16:29 | 1 BOX(ES)500 ML; 3 BOXES 60 ML;BUBBLE WRAP |
| E03180 | COOL0593 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0660 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0655 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0668 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0664 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0653 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0629 | 3924062-0800-3200 | 16:30 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0550 | 3924062-0800-3200 | 16:31 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0472 | 3924062-0800-3200 | 16:31 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E03180 | COOL0267 | 3924062-0800-3200 | 16:31 | 1 BOX(ES); 1 LITER; BUBBLE WRAP; |
| E01421 | COOL0344 | 3924079G-0201-3200 | 16:48 | 6 SODIUM HYDROXIDE; TRASH BAGS; GLOVES; LOGSHEETS; PH STRIPS; PIPETTES; EVIDENCE TAPE;SITE ID13 ; |
| E01421 | COOL0481 | 3924079G-0201-3200 | 16:48 | SITE ID 14 ; LOGSHEETS; TRASH BAGS; |
| E01421 | COOL0151 | 3924079G-0201-3200 | 16:49 | SITE ID 15 ; TRASH BAGS; LOGSHEETS; LOGSHEETS; |
| E01421 | COOL0155 | 3924079G-0201-3200 | 16:51 | SITE 16 ; TRASH BAGS; LOGSHEETS;L0001 |
| E01421 | COOL0117 | 3924079G-0201-3200 | 16:51 | SITE ID 17 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0613 | 3924079G-0201-3200 | 16:52 | SITE ID 18; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0174 | 3924079G-0201-3200 | 16:52 | SITE ID 19 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0669 | 3924079G-0201-3200 | 16:53 | SITE ID 20 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0434 | 3924079G-0201-3200 | 16:54 | SITE ID 21 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0452 | 3924079G-0201-3200 | 16:54 | SITE ID 22 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0670 | 3924079G-0201-3200 | 16:55 | SITE ID 23 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0671 | 3924079G-0201-3200 | 16:55 | SITE ID 24 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0168 | 3924079G-0201-3200 | 16:56 | SITE ID 25 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0672 | 3924079G-0201-3200 | 16:57 | SITE ID 26 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0096 | 3924079G-0201-3200 | 16:57 | SITE ID 27 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0674 | 3924079G-0201-3200 | 16:58 | SITE ID 28 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0149 | 3924079G-0201-3200 | 16:58 | SITE ID 29 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0678 | 3924079G-0201-3200 | 16:59 | SITE ID 30 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0456 | 3924079G-0201-3200 | 16:59 | SITE ID 31 ; TRASH BAGS; LOGSHEETS; |
| E01421 | COOL0480 | 3924079G-0201-3200 | 16:59 | SITE ID 32 ; TRASH BAGS; LOGSHEETS; |

| | |
|---|---|
| E03289 | : VYLRIE MCWILLIAMS |
| E01421 | : ALFREDA MOORE |
| E03291 | : DONNA CREWS |
| E03180 | : DELORES DARLING |
| 3904076-0214-3200 | : - |
| 3924031G-0202-3200 | : 3924031G-0202-3200- , MARK SKROBACZ, ESE INC, 250-A EXCHANGE PLACE, HERDON, VA. VA. |
| 3924062-0800-3200 | : 3924062-0800-3200- ATTN: UMATILLA D & M, DAMES & MOORE (KEVIN PERRETTE ), UMATILLA ARMY DEPOT, BLDG 11, HERMI |
| 3924079G-0201-3200 | : 3924079G-0201-3200- ATTN: RARITAN, KEITH WARD, HOLIDAY INN, 125 RARITAN CENTER PARKWAY, EDISON NJ |

**Figure 7-4**
**EXAMPLE OF COOLER TRACKING REPORT**

SOURCE: ESE

Environmental Science & Engineering, Inc. 12-23-92 *** FIELD LOGSHEET *** FIELD GROUP: EXAMPLE
PROJECT NUMBER 3924000V 0000 PROJECT NAME: COMPANY XXX LAB COORD. PORTIA PISIGAN

| ESE # | SITE/STA | HAZ? | FRACTIONS(CIRCLE) EC EC VP VP VP | DATE | TIME | PARAMETER LIST W610LC |
|---|---|---|---|---|---|---|
| *1 | MW-1 | | EC EC VP VP VP | | | W610LC |
| *2 | MW-2 | | EC EC VP VP VP | | | W610LC |
| *3 | MW-3 | | EC EC VP VP VP | | | W610LC |
| *4 | MW-4 | | EC EC VP VP VP | | | W610LC |
| *5 | MW-5 | | EC EC VP VP VP | | | W610LC |
| *6 | MW-6 | | EC EC VP VP VP | | | W610LC |
| *7 | MW-7 | | EC EC VP VP VP | | | W610LC |
| *8 | MW-8 | | EC EC VP VP VP | | | W610LC |
| *9 | MW-9 | | EC EC VP VP VP | | | W610LC |
| *10 | MW-10 | | EC EC VP VP VP | | | W610LC |

NOTE -CHANGE OR ENTER SITE ID AS NECESSARY; UP TO 9 ALPHANUMERIC CHARACTERS MAY BE USED
-CIRCLE FRACTIONS COLLECTED. ENTER DATE,TIME,FIELD DATA (IF REQUIRED), HAZARD CODE AND NOTES
-HAZARD CODES: I-IGNITABLE C-CORROSIVE R-REACTIVE T-TOXIC WASTE H-OTHER ACUTE HAZARD: IDENTIFY SPECIFICS IF KNOWN
-PLEASE RETURN COMPLETED LOGSHEETS WITH SAMPLES TO Environmental Science & Engineering, Inc.

| RELINQUISHED BY: (NAME/ORGANIZATION/DATE/TIME) | VIA: | REC'D BY (NAME/ORGANIZATION/DATE/TIME) |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |

SAMPLER: Shipped on Ice? Yes/No; I anticipate shipping ____(#) more samples on ___/___
SAMPLE CUSTODIAN: Custody Seals Used? Yes/No; If Yes, Seals Intact? Yes/No Interior Temp? ___Deg C
Preservatives Audited? Yes/No Any Problems? Yes/No; If Yes, describe:

**Figure 7-5**
**CHAIN-OF-CUSTODY FIELD LOGSHEET**

SOURCE: ESE.

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

### 7.1.3 DOCUMENTATION

The records for laboratory sample custody include:

1. Laboratory Forms:

   Sample Kit Request Form (Figure 7-1),
   ESE Shipping Request Form (Figure 7-2),
   Kit Pick-Up Log (Figure 7-3),
   Example of Cooler Tracking Report (Figure 7-4),
   Sample Label (Figure 7-6),
   Standardized Sample Preservation Codes (Figure 7-7),
   Sample Chest Custody Form (Figure 7-8),
   Cold Room Sample Arrival Logbook (Figure 7-9),
   Sample Check In/Out Log (Figure 7-10),
   VOA GC Sample Thru-Log (Figure 7-11),
   VOA GC/MS Sample Thru-Log (Figure 7-12),
   Radiochemistry Sample Storage and Custody Logsheet (Figure 7-13),
   Conductivity Meter Calibration Form (Section 9.0), and
   pH Meter Calibration Form (Section 9.0).

2. Sample Extraction Log (Organic Laboratory/Extraction Logsheet, Figure 7-14).

Errors in all documents are corrected by following the procedure in Section 7.1.2.

## 7.2 LABORATORY CUSTODY

Sample chests (packages/coolers) are transported to the laboratory. The deliverer will sign, date, and indicate the time of delivery, the number of packages, the Laboratory Coordinator or addressee, and any comments including visible or suspected physical condition of the packages into the Sample Chest Custody Logbook (Figure 7-8). The chests are then recorded as having been received by the laboratory in the Sample Chest Custody Logbook by the Sample Custodian.

PRJ# 3924000V 0000 MW-10
COMPANY XXX
EXAMPLE*10-VP
SAMPLER DATE TIME
COND PH

Figure 7-6
SAMPLE LABEL

SOURCE: ESE.

ENVIRONMENTAL SCIENCE
& ENGINEERING, INC.

## ESE KEY TO FRACTION CODES 05/94

| | CODE | PRESERVATIVE | CONTAINER | ANALYSIS TYPE | HOLDING TIMES |
|---|---|---|---|---|---|
| AIR: | AA | 4° C | Various | Various | Various |
| | AO | Exclude Light | Sorbent | Organic | 14 Days |
| | AV | Exclude Light | Charcoal | Volatiles | 14 Days |
| | AT | 4° C | Tedlar | Volatiles | 72 Hrs |
| | FL | Exclude Light | Cassette | Various | Various |
| | HV | | Hi-Vol Filter | TSP/PM10/Metals | |
| SOILS: | SS | 4° C | G, 250 mL | All excl. Vol. | 7-28 Days |
| | SV | 4° C | G, 120 mL | Volatiles | 7-14 Days |
| WATER: | AL | 4° C# | G, 2x40 mL* | Carbamates, EPA531 | 14 Days |
| | B | 4° C; NaOH, pH>12 | P, 1-4 L** | Cyanides | 14 Days |
| | C | 4° C | P, 1-4 L | Various Inorganics | 1-28 Days |
| | CF | 4° C | P, 1-4 L | Filtered Inorganics | 1-28 Days |
| | CL | 4° C | P, 1 L, opaque | Chlorophyll | 14 Days |
| | EC | 4° C, # | G, 1 L | Chlorinated Pests | 7 Days |
| | ED | 4° C, # | G, 2x40 mL* | EDB, DBCP | 14 Days |
| | F | – | P, 1-4 L | Collection prior to Field Filtering | |
| | FI | 4° C, # | G, 40-125 mL | GC/FI Organic | 7 Days |
| | FM | Formaldehyde | P/G, 500 mL | "Quats" | 28 Days |
| | FP | 4° C, # | G, 3x40 mL* | GC/FP Organic | 14 Days |
| | H | Zn Acetate;NaOH,pH>10 | P, 1 L | Sulfides | 7 Days |
| | HB | 4° C, # | G, 1 L | Chlorinated Herbs | 7 Days |
| | IC | 4° C | G, 250mL-1L | IMPA | 40 Days |
| | LC | 4° C | G, 1 L | HPLC Organics | 7 Days |
| | M | 4° C, #, Exclude Light | P, 250 mL | Bacteriologic | 6 Hours |
| | MS | 4° C, # | G, 1 L | GCMS - SVOCs | 7 Days |
| | N | $HNO_3$, pH<2 | P, 1 L | Metals (Total) | 180 Days |
| | NF | $HNO_3$, pH<2 | P, 1 L | Metals (Dissolved) | 180 Days |
| | NC | 4° C | G, 1 L | Nitrocellulose | 7 Days |
| | NG | 4° C | G, 1 L | Nitroglycerin/PETN | 7 Days |
| | NP | 4° C | G, 1 L | GC/NP Organic | 7 Days |
| | NQ | 4° C | G, 1 L | Nitroguanidine | 7 Days |
| | O | 4° C; $H_2SO_4$, pH<2 | G, 1 L | Oil & Grease, TRPH | 28 Days |
| | OC | 4° C; $H_2SO_4$, pH<2 | G, 1 L | TOC (USAEC) | 28 Days |
| | OD | 4° C | G, 1 L | Odor | 2 Days |
| | R | $HNO_3$, pH<2 | P, 1-4 L | Radionuclides | 180 Days |
| | S | 4° C; $H_2SO_4$,pH<2 | P, 1 L | Nutrients; TOC | 28 Days |
| | SF | 4° C; $H_2SO_4$,pH<2 | P, 1 L | Filt'd Nutrients;TOC | 28 Days |
| | V | 4° C, # | G, 4x40 mL | VOCs, ex. Aromatics | 14 Days |
| | VP | 4° C; HCL, pH<2, # | G, 4x40 mL* | VOCs, w/ Aromatics | 14 Days |
| | W | 4° C, # | G, 1 L | Var. Organics | 7 Days |
| | X | 4° C | G, 250 mL | Various | Various |
| | XP | 4° C; $H_2SO_4$, pH<2, @ | G, 2x250 mL* | TOX | 28 Days |
| | Z | 4° C; $H_2SO_4$, pH<2 | G, 1 L | Total Phenols | 28 Days |
| OTHER: | OL | None | G, 10-100 mL | Organic - Oil | 14 Days |
| | TS | Frozen, <-20° C | Various | Frozen Tissue | Various |

FOOTNOTES: # - Add Sodium Thiosulfate ($Na_2S_2O_3$) If Residual Chlorine Present (0.25g/L).
@ - Add Sodium Sulfite ($Na_2SO_3$) if residual chlorine present (0.1M, 1 mL/L).
* - These fractions are prepreserved unless noted otherwise.
** - Test for presence of sulfide and follow EPA procedures (below) as necessary.

### INSTRUCTIONS FOR SAMPLING AND SHIPPING

- Plastic (P) containers may be rinsed with sample; Do not rinse Glass (G).
- Fill completely, especially for volatiles (fill these slowly; achieve positive meniscus; cap; invert; check for air bubbles; top off if needed).
- Preserve with reagents provided as instructed above (unless pre-preserved).
- Special cyanide preservations: When the presence of sulfide is indicated by a positive spot test with lead acetate paper, preservation consists of: 1) precipitation with cadmium nitrate until a negative test is obtained; 2) filtration of the precipitate; and 3) addition of NaOH to pH > 12.
- Fill-out logsheet/chain-of-custody. Indicate: Sample Number (*) and fractions collected; dates/times of collection & shipment; appropriate field notes. Be sure to sign and date the bottom of each page where indicated.
- Ship with bagged ice in ice-chest by express carrier to lab coordinator's attention.

Source: ESE, 1994

**Figure 7-7**
**STANDARDIZED SAMPLE PRESERVATION CODES**

SOURCE: ESE.

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

SAMPLE CHEST CUSTODY FORM

**DO NOT REMOVE FROM CHECK-IN AREA**

| Relinquished By: | | | | |
|---|---|---|---|---|
| Time | Date | Signature | No. of Chests | Coordinator |
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| Received By: | | | | | | |
|---|---|---|---|---|---|---|
| Date | Time | Project Name | Signature | Comments * | Rads>Background | |
| | | | | | Chest? | Sample? |
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* No comment means that the container was relinquished/received in good condition, properly sealed and that there was no evidence of tampering.

**Figure 7-8**
**SAMPLE CHEST CUSTODY FORM**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

COLD ROOM SAMPLE ARRIVAL LOGBOOK

"DO NOT REMOVE FROM COLDROOM"

| Date | Time | Project Name or Field Group | No. Containers or Sample Nos. | Coordinator or Fractions | Signature | Comments |
|---|---|---|---|---|---|---|
| | | | | | | |
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ESE SAMPLE CHECK IN/OUT LOG

| FIELD GROUP | SAMPLE SEQUENCE # | FRACTION | INITIALS | DEPT. # | DATE OUT | DATE IN |
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Figure 7-10
SAMPLE CHECK IN/OUT LOG

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

COMP QAPP 12/92 BL

## ESE VOA GC SAMPLE THRU - LOG WEEK ENDING_____

| Project Coordinator | Samples & Description | Arrival Date | Checked in by | Screen Date | Analysis Date | Complaints | Comments VOA Screen Requested |
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**Figure 7-11**
**VOA GC SAMPLE THRU LOG**

SOURCE: ESE.

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

## ESE VOA GC/MS SAMPLE THRU - LOG

| Sample & Description | Sample Date | Arrival Date | Screen Date | Date Placed in Analyst Fridge | Checked in By | Trip Blank | Comments |
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OCMSVOA.LOG
11/30/93

**Figure 7-12**
**VOA GC/MS SAMPLE THRU LOG**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

## Radiochemistry Sample Receipt Log

| PROJECT COORDINATOR | PROJECT NAME | FIELD GROUP | SAMPLE SEQUENCE | ARRIVAL DATE | DELIVERED BY: | RECEIVED BY: | NOTES |
|---|---|---|---|---|---|---|---|
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RADC:IIEM/SAMRCT/APR93

**Figure 7-13**
**RADIOCHEMISTRY SAMPLE STORAGE AND CUSTODY LOG SHEET**

SOURCE: ESE.

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

660

**ESE.** ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

## ORGANIC LABORATORY/EXTRACTION LOG SHEET

PROJECT: _____ EXTRACTION METHOD: _____
PROJECT NO.: _____ CASE NO.: _____ SAMPLE FRACTION: _____
EXTRACTOR(S): _____ CLEAN-UP: _____ CHECKED BY: _____
DATE: _____ TIME: _____ BALANCE USED: _____

| SAMPLE NO. | VOLUME-ml MASS-gr. | INITIAL/ FINAL pH | TYPE/DATE OF CLEAN-UP | FINAL VOL. SOLVENT | SURRO-GATE ADDED Y/N | COMMENTS |
|---|---|---|---|---|---|---|
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| CHAIN-OF-CUSTODY RECORD | | | | | | |

FORM 81282

**Figure 7-14**
**ORGANIC EXTRACTION LOG SHEET**

SOURCE: ESE.

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The samples are checked in by the Sample Custodian for proper preservation (e.g., pH, temperature), integrity (e.g., leaking, broken bottles), and proper and complete sample documentation and ID. Sample chests or coolers that are not within the 4 $\pm$ 2 degrees Celsius (°C) requirement are reported immediately to the Laboratory Coordinator to determine if resampling will be required. All samples contained in the shipment are compared to the logsheet(s) to ensure that all samples designated on the logsheet have been received. Any changes in station ID from the originally established station ID are noted. The Sample Custodian will note any special remarks concerning the shipment. The Sample Custodian reviews the integrity of all sample fraction containers, checks the accuracy and clarity of all documentation received, and scans all the samples for radioactivity level. The Sample Custodian audits all fractions requiring field preservation to ensure that they have been properly preserved. The Sample Custodian will preserve unpreserved fractions or add additional preservative, if needed, on receipt. Deficiencies in sample preservation, additional preservative added, and all other inadequacies are recorded on the logsheet and reported to the Laboratory Coordinator.

All insufficiencies and/or discrepancies are recorded on the logsheet and immediately reported to the appropriate Laboratory Coordinator. The Laboratory Coordinator will inform the Project Manager and field team. The Project Manager, upon consultation with the Project QA Coordinator, may decide if resampling is required.

After all samples and documentation have been reviewed and appropriately annotated, the Sample Custodian signs the logsheet and submits it to Information Services for processing. Any marks or notes made on the chain-of-custody document by the Sample Custodian should be clearly distinguished from original field notations.

Shipping receipts are stapled on chain-of-custody logsheets and stored in the project file.

Samples are placed in appropriate storage areas in the laboratory depending on storage requirements. The Department Managers or their designee are notified that the samples

have arrived through the distribution of arrival notices. The majority of the samples are stored in the main coldroom with the temperature maintained at 4 ± 2°C. The Sample Custodian will log the samples delivered into the coldroom in the Cold Room Sample Arrival Logbook (Figure 7-9). The coldroom is kept locked when not in use. The water samples for metals analysis (fractions N and NF) are stored in a separate air-conditioned storage room located near the metals sample preparation area. This room is also kept locked if not being used by the analyst. The samples in these storage areas are assigned to labeled shelves by field group. A sample location list is posted at the door of each storage room. Access to samples is limited to authorized personnel, and a Sample Check In/Out Log is maintained (Figure 7-10). ESE has started to institute the bar code system to track samples that were taken in and out of the main coldroom.

The samples for volatile organics are delivered directly to the gas chromatography (GC)-Volatiles or gas chromatography/mass spectrometry (GC/MS) Department by the Sample Custodian and are stored in the department's refrigerators designated for sample storage only. The samples delivered to these departments by the Sample Custodian are logged in the department's Sample Thru-Log (Figures 7-11 and 7-12). Sample fractions for volatiles are stored separate from standards or semivolatile fractions in order to minimize cross-contamination. Samples remain in storage until it is unnecessary to retain them, at which time their disposition (in accordance with Section 8.0) is noted.

The samples for radiochemistry analyses are delivered directly to the Radiochemistry Department and logged into the Sample Storage and Custody Logsheet (Figure 7-13). Samples checked out and checked in from this storage room are also documented in the Sample Storage and Custody Logsheet.

When it is necessary to use another laboratory for sample analysis, the Laboratory Coordinator is responsible for arrangements with the second laboratory. The samples will only be subcontracted to a NEESA, HAZWRAP, FDER, state and federal government agency, or client-approved laboratory (as appropriate). Specific NEESA/HAZWRAP

approval is required prior to subcontracting. Documentation to transfer to another laboratory must include: collection data and time, field ID, laboratory ID, date of sample preparation, and requested analyses.

The samples should be chilled prior to and during shipment. A logsheet indicating samples and fractions sent must accompany the samples to the subcontractor. The subcontractor should sign and date the logsheet upon receipt of the samples. A copy of the signed logsheet will be returned to ESE and placed in the project file.

## 7.3 LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)

CLASS™ is an automated, inhouse-developed LIMS that integrates information from sample collection, laboratory analyses, and QC requirements; and calculates, checks, stores, and reports data in a variety of formats. CLASS™ resides on a Novell Arcnet (using Novell SFT, version 2.11) IBM-PC-compatible network with 1,600 megabytes of storage and is connected to more than 80 personal computers (PCs) throughout the Gainesville chemistry laboratories and offices. CLASS™ is managed by the Laboratory Information Services Department within the Gainesville laboratory. All data from analyses performed by the laboratory are managed and stored using CLASS™.

The database is stored, processed, and retrieved using the database manager Advanced Revelation© (copyright Revelation Technologies). The file structure and indexing provided by Advanced Revelation© allow easy retrieval, grouping, and formatting of data. Incorporated into the system is the ability to combine field data, analytical results, and QC data and produce specially formatted project-specific reports, statistical analyses, plots, and electronic files.

CLASS™ manages the flow of samples and data through the laboratory. Prior to sampling, the Laboratory Coordinator provides information on the number of samples, site IDs, parameters to be analyzed, and estimated collection dates. This information is

entered into CLASS™ and used to produce sample labels and chain-of-custody logsheets (Figure 7-5). A unique ESE number is assigned to each sample, and labels with that number and the site ID are placed on each container for that sample. At each site, samples are collected and placed in the appropriate prelabeled containers. Sampling information is recorded on the field logsheet. Samples accompanied by the field logsheet are sent to the laboratory where they are checked and processed by the Sample Custodian. Samples are stored in the coldroom at 4 $\pm$ 2°C. The logsheet is submitted to the Laboratory Information Services Department, where the samples, along with the date of collection and site ID, are logged into CLASS™. Logsheets are placed in the project file and maintained by Information Services.

ESE uses a combination of EPA Storage and Retrieval (STORET) numbers and company-assigned Method Codes to designate parameters required for analysis. Each STORET-method combination has its own laboratory QC requirements specific to that analytical method stored in CLASS™. A list of all required parameters is logged into the computer with each sample. This list is identified on the field logsheet for each sample.

The sampling information is entered into the computer to activate the parameter list for the samples collected and received by the laboratory. A report (Available Numbers) of samples available for each analysis indicates the number of days left before the holding time is exceeded for each method for each sample. This report is distributed daily to each analytical department, and the information also can be accessed readily from CLASS™ by the Laboratory Coordinator or any analyst in the laboratory.

CLASS™ uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a sample batch, the analyst will designate a specified group of samples in the computer and the sample-parameter status will be updated to "IL" ("In Laboratory"). The analytical batch is assigned a unique batch control number, which is stored with all final data, to facilitate data review, QC reporting, and retrieval of original documentation.

The production of each laboratory batch usually requires several distinct activities. Instrument calibrations are entered first and includes several QC checks by CLASS™. The linear (or quadratic or logarithmic) regression equation and correlation coefficient are calculated from the calibration curve data, and the correlation coefficient is tested to determine whether it is within an acceptable range specific to the analysis. Method blank and control spike information are then entered, and results are calculated and checked against control limits for that method. Sample responses are entered into the batch, and final concentrations are calculated for each sample. Responses are checked to ensure that they are bracketed by the standard curve. The batch printout includes a QC summary showing the automated QC checks, such as holding times, the presence of spikes, and acceptable spike recoveries. Any discrepancies are flagged by the computer for the analyst.

The batch printout also documents that the analyst has checked data entries and provided all required documentation for the analysis. The batch printout is completed, signed, and dated by the analyst, and reviewed and signed by the Department Manager or a designated reviewer.

Analysts use the PC to reserve samples for analysis, calculate final concentrations, and interactively check calibration curves and QC results. By allowing the analyst to enter data directly and check QC and sample results, the analyst is made aware of QC problems. When the analyst has entered the curve and QC and sample data, the batch printout (including checklists), worksheets, copies of notebook pages and any other pertinent documentation (e.g., chromatograms), are placed in a file folder with the assigned batch number and submitted to Information Services. Information Services personnel process the batch in the computer to verify QC and to update the sample records and final calculated concentrations.

Each employee is assigned an individual access code for entry into CLASS™. Laboratory personnel and Laboratory Coordinators are not permitted to update sample records; this is done exclusively by Information Services. All personnel with an access code may retrieve information from the system.

Once a batch has been finalized by Information Services, the batch is locked, and data cannot be changed by the analyst. If a data change is necessary, a Batch Update Request Form (Figure 7-15) must be completed. This form requires the reason for the change,the approval of the Laboratory Department Manager, and the approval of the Laboratory Coordinator. The form is then submitted to Information Services, where the original batch folder is retrieved. Information Services personnel make the change, the form is stapled to the new batch printout, and the updated batch is reviewed and approved by the Laboratory Department Manager and Laboratory Coordinator.

The batch folders, with all supporting documentation, are filed chronologically by department in a secured Information Services storage room; file cabinets with project files are stored similarly. These may be signed out for review by the analysts, Laboratory Coordinators, or QA personnel. A program in CLASS™ is used to track folders that have been checked out. Batch folders and project files are kept a minimum of 5 years.

Laboratory personnel and Laboratory Coordinators use the computer to monitor the flow of data through the system. Data are accessed and reported by sampling event, project, or any subset of samples and parameters. A log of electronic transfers is kept by Information Services. CLASS™ enables the Laboratory Coordinator to:

1. Produce a variety of summary reports of analytical data;
2. Calculate parameters from analytical data (e.g., cation/anion balance);
3. Calculate statistics such as mean, maximum, minimum, and standard deviation;
4. Calculate atmospheric concentrations;

BATCH UPDATE

Batch # _____ Analysis _____ Dept.# _____

Request to (circle one): DEFINALIZE or CHANGE

Initiated by: _____ In Order to:

APPROVED/ACKNOWLEDGED BY:

Dept. Manager _____ Date _____

Lab Coordinator(s) _____ _____

_____ _____

_____ _____

Changes Processed by: _____ _____

UPDATED Batch Reviewed & Approved by: _____ _____

Batch Refinalized by: _____ _____

**Figure 7-15**
**BATCH UPDATE REQUEST FORM**

SOURCE: ESE.

5. Produce a data file to be read into the Statistical Analysis System (SAS) for in-depth statistical analysis;
6. Summarize QC in various formats; and
7. Produce a project-specific export-data file.

Data are stored in the CLASS™ database and can be exported electronically into Lotus and DBASE files. Many client-requested formats have been developed in CLASS™ for electronic data transfer. When a client requests an electronic data transfer, a regularhardcopy data report is usually sent in addition to the electronic file. Copies of both electronic and hard copies are maintained in project files.

Information Services has a staff of computer programmers to maintain and modify CLASS™. Requests for new programs or changes are kept in both electronic and hardcopy files; the names of the person making the request and the programmer are included. Every change made to a program is documented electronically at the end of the program with the date, employee number of the programmer, and a brief description of the change. A summary of these changes is maintained in CLASS™ listing the programs, changes, requestors, and programmers. All program revisions are documented in a revisions file and can be reviewed anytime. Completed requests are tested by the programmer staff and then verified by the requestor.

The Laboratory QC Coordinator validates a portion of the data quarterly by recalculations from the raw values and verification that the computer is performing calculations correctly.

The database is backed up daily except Saturday using optical disks or equivalent high-density storage media. These disks are stored in the Information Services air-conditioned locked storage room located in a separate building. Archived electronic data are stored in special files accessible by Information Services personnel. Laboratory Coordinators can access the status of all data (including archived samples in CLASS™) and may request that sample data be restored for more thorough review.

# 8.0 ANALYTICAL PROCEDURES

## 8.1 STANDARD PROCEDURES

Standard analytical procedures to be used for any project for chemical analysis of water and soil are referenced in Section 5.0. Laboratory Department Managers will ensure that only these standard analytical methods are employed by the staff. Standard analytical methods manuals are required for all departments, and development of the documents are ultimately the responsibility of the department managers. The methods cited in these documents will be the methods normally used. Any deviation from the standard method must be documented in the analyst notebook and approved by the department manager.

For parameters not listed, nonstandard methods may be specified by the client or developed by the laboratory. Nonstandard methods must be validated as described in Section 8.3.

## 8.2 NONSTANDARD METHODS VALIDATION

If other than standard analytical methods become necessary due to a change in work scope, it is necessary to validate the analytical method. The responsible department manager must establish thorough method validation so that the method selected measures the reported parameter with the necessary precision, accuracy, and detection limit and without severe interference by other constituents in the sample. Major modifications of standard methods such as extraction, preparation, and cleanup procedures and/or the application of a standard method to new analytes or matrices will require method validation. Nonstandard methods will be submitted to the appropriate agency (i.e. NEESA, HAZWRAP, FDEP, USAEC, etc) for review and approval prior to use on samples for analyses.

The following subsections constitute the minimum requirements for initial establishment of the accuracy, precision, and detection limit of nonstandard methods.

For each parameter of interest, seven replicate spike samples will be prepared from laboratory blank water (for water methods) or an uncontaminated "standard" appropriate matrix (for soil or tissue methods) at one appropriate analyte concentration. Spiked samples will be analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water will be analyzed. The spiking concentration should be selected such that the final extract or aliquot can be analyzed with less than tenfold dilution in the midrange of the calibration curve.

The detection limit of each parameter of interest will be determined according to the protocols described in 40 CFR Part 136 Appendix B.

Accuracy (Recovery)--The minimum requirements for initial establishment of accuracy for nonstandard methods are as follows:

1. Calculate the found concentration for each spiked sample as follows:
   R = measured concentration = measured concentration in spiked sample minus the measured concentration in unspiked (blank) sample.
2. Calculate the percent recovery for each spiked sample as follows:

$$P = \frac{R}{S} \times 100\%$$

   where: R = measured concentration for each spiked sample, and
   S = target concentration for each spiked sample.

3. Calculate the average percent recovery and relative standard deviation of the percent recovery for the spiked "standard" samples as follows:

$$\overline{P} = \frac{P_1 + P_2 + P_3}{3} = \begin{matrix}(standard\ samples)\\ average\ percent\ recovery\end{matrix}$$

where: $S_r$ = standard deviation of P

GLASSWARE WASHING REQUEST FORM

TO BE DONE

☐ Normal Wash 1) Hot soapy tap water wash
2) Tap water rinse
3) DI rinse

☐ Rinse with DI only!

☐ Other _____ (be specific)

_____

_____

_____

SOLVENT RINSE

☐ Acetone

☐ Other _____

_____

_____

NEEDED BY: _____
Date and Time

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

REQUESTED BY: _____

THE FOLLOWING HAS BEEN COMPLETED

☐ Normal Wash

☐ Rinse with DI only!

☐ Other _____

_____

_____

_____

SOLVENT RINSE

☐ Acetone

☐ Other _____

_____

_____

COMPLETED BY _____

DATE _____

Figure 8-1
GLASSWARE WASHING
REQUEST FORM

SOURCE: ESE.

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& ENGINEERING, INC.

Table 8-1. Glassware Cleaning Procedures

| Analysis/Parameter | Cleaning Protocol* |
|---|---|
| Extractable Organics | 1,2,3,4,5,6 |
| Purgeable Organics (Volatiles) | 1,2,3,4,7,13 |
| HPLC Analyses | 1,2,3,4,5,10 |
| EDB, DBCP, THMS | 1,2,3,4,5,8,13 |
| Trace Metals | 1,2,3,4,12 |
| Nutrients | 1,2,3,4,11 |
| Minerals | 1,2,3,4 |
| Residues | 1,2,3,4,14 |
| Cyanide, Oil and Grease, Phenols | 1,2,3,4 |
| Petroleum Hydrocarbons | 1,2,3,4,5,9 |
| COD, BOD | 1,2,3,4 |
| Radiochemistry | 1,2,3,4 |

Note: DBCP = 1,2-Dibromo-3-chloropropane
EDB = 1,2-Dibromoethane
HCL = Hydrochloric acid
HNO3 = Nitric acid
HPLC = High Pressure Liquid Chromatography
THMS = Trihalomethanes

*Cleaning Procedures

1. Remove all labels using sponge or acetone.
2. Wash with hot soapy water (use Liquinox soap only) using brushes to scrub inside of glasswares, stopcocks, and other small pieces if possible.
3. Rinse three times with hot tap water.
4. Rinse three times with deionized water.
5. Rinse thoroughly with pesticide grade acetone.
6. Rinse with pesticide grade Methylene Chloride.
7. Rinse with pesticide grade methanol.
8. Rinse with pesticide grade hexane.
9. Rinse with appropriate extraction solvent prior to use .
10. Rinse with pesticide grade acetonitrile and pesticide grade methanol prior to use, if needed.
11. Acid rinse with 1:1 HCL, using only metals grade HCL.
12. Acid rinse with dilute HNO3 and then with deionized water prior to use.
13. Bake at 80°C for 3-4 hours.*
14. Bake at 180°C for 3-4 hours.*

*Class A volumetric glassware should not be baked.

Source: ESE.

## 8.4 LABORATORY METHOD MODIFICATIONS

Laboratory method modifications are done either to improve the method efficiency, apply a water method with the addition of appropriate sample preparation/digestion method to soils or add new compounds to an approved method. ESE has several method modifications involving the addition of new compounds to a specific EPA method(s) or applying a water method with the addition of appropriate sample digestion/extraction method to soils. These compounds are listed in Table 8-2 and their QA targets are found in Section 5.0. Method validation have been performed and the method validation packages were submitted to FDEP for approval. These method validation studies were referenced in the appendices of this LCQAP. The method validation packages include precision and accuracy data, method detection limit studies, copy of the method used, and raw data. Method validation were performed on the following compounds listing the method and matrix used:

Table 8-2. Laboratory Method Modification Compounds

| Compound/Parameter | EPA Method | Matrix |
|---|---|---|
| Cyanide | EPA 9010 | Soil |
| Total Organic Carbon | EPA 9060 | Soil |
| Hexavalent Chromium | EPA 3060/7196 | Soil |
| Total Petroleum Hydrocarbons (TRPH) | EPA 3550, 418.1 | Soil |
| TOX | EPA 9020 | Soil |
| Gross Alpha | EPA 3050, 9310 | Soil |
| Gross Beta | EPA 3050, 9310 | Soil |
| Radium 226 | EPA 3050, 9320 | Soil |
| Radium 226, alpha emitters | EPA 3050, 9315 | Soil |
| Radium 228 | EPA 3050, 9320 | Soil |
| Freon 113 | EPA 8010 | Water |
| MTBE | EPA 8010 | Water |
| Isodrin | EPA 8080 | Water & Soil |
| Kepone | EPA 8080 | Water & Soil |
| Metolachlor | EPA 8080 | Water & Soil |
| Kelthane (Dicofol) | EPA 8080 | Water & Soil |
| Alachlor | EPA 8140 | Soil |
| Metribuzin | EPA 8140 | Soil |
| EPTC | EPA 8140 | Water & Soil |
| Butylate | EPA 8140 | Water & Soil |
| Pebulate | EPA 8140 | Water & Soil |
| Vernolate | EPA 8140 | Water & Soil |
| Atrazine | EPA 8140 | Water & Soil |
| Terbufos | EPA 8140 | Water & Soil |
| Haxazinone | EPA 8140 | Water & Soil |
| Famphur | EPA 8140 | Water & Soil |
| O,O,O-Triethyl Phosphorothioate | EPA 8140 | Water & Soil |
| Sulfotepp | EPA 8140 | Water & Soil |
| Thionazin | EPA 8140 | Water & Soil |
| Methylethyl ketone (MEK) | EPA 8240 | Water & Soil |
| Methylisobutyl ketone (MIBK) | EPA 8240 | Water & Soil |
| Acrolein | EPA 8240 | Water & Soil |
| Acrylonitrile | EPA 8240 | Water & Soil |

Table 8-2. Laboratory Method Modification Compounds
(Continued, Page 2 of 2)

| Compound/Parameter | EPA Method | Matrix |
|---|---|---|
| Carbon disulfide | EPA 8240 | Water & Soil |
| Acetophenone | EPA 8270 | Water & Soil |
| 4-Aminobyphenyl | EPA 8270 | Water & Soil |
| 4-Chloroaniline | EPA 8270 | Water & Soil |
| 2,6-Dichlorophenol | EPA 8270 | Water & Soil |
| p-(Dimethylamino)azobenzene | EPA 8270 | Water & Soil |
| 7,12-Dimethylbenz(a)anthracene | EPA 8270 | Water & Soil |
| Ethyl methanesulfonate | EPA 8270 | Water & Soil |
| o-Cresol | EPA 8270 | Water & Soil |
| p-Cresol | EPA 8270 | Water & Soil |
| 1-Napthylamine | EPA 8270 | Water & Soil |
| 2-Nitroaniline | EPA 8270 | Water & Soil |
| 3-Nitroaniline | EPA 8270 | Water & Soil |
| 4-Nitroaniline | EPA 8270 | Water & Soil |
| N-Nitroso-di-n-butylamine | EPA 8270 | Water & Soil |
| N-Nitrosomethylethylamine | EPA 8270 | Water & Soil |
| N-Nitrosopiperidine | EPA 8270 | Water & Soil |
| Pentachlorobenzene | EPA 8270 | Water & Soil |
| Pentachloronitrobenzene | EPA 8270 | Water & Soil |
| Phenacetin | EPA 8270 | Water & Soil |
| 2-Picoline | EPA 8270 | Water & Soil |
| Pronamide | EPA 8270 | Water & soil |
| 1,2,4,5-Tetrachlorobenzene | EPA 8270 | Water & Soil |
| 2,3,4,6-Tetrachlorophenol | EPA 8270 | Water & Soil |
| 2,4,5-Trichlorophenol | EPA 8270 | Water & Soil |
| 1,3,5-Trichlorobenzene | EPA 8270 | Water & Soil |

## 8.5 REAGENT STORAGE

The procedures for storing reagents in the laboratory are presented in (Section 6.0). All reagents are marked with date received and date opened.

## 8.6 LABORATORY WASTE DISPOSAL

It is important that all waste materials generated in the laboratory be disposed of promptly and properly. The following subsections describe the procedures for handling laboratory wastes.

### 8.6.1 LIQUID WASTES

1. In general, no chemical wastes may be disposed of in the sinks.
2. Only certain dilute acid wastes can be disposed of in the sinks.
3. Disposal of Standards and Solutions--As standards and solutions are made, the solvent, constituents, date, and initials must be put on the container. This information must be on the container before it is offered for disposal. Standards containing any amount of organic solvents must not be poured down the sink. Aqueous standards of organic or inorganic (metals, etc.) compounds must either be disposed of in the appropriate waste drum, or picked up by the waste technician.
4. Disposal of Solvent Wastes--All waste solvents should be disposed of in the red waste-solvent containers located throughout the different departments in the laboratory. Solvents should be segregated according to the designated chemical types and placed only in the appropriate waste-solvent container. The waste containers will be emptied on a regular basis. If the containers become full before then, the hazardous materials technician should be called so the containers can be emptied.

Solvents will be segregated as follows:

| Freon Waste | Chlorinated Solvents | Flammable Solvents | | HPLC Solvents |
|---|---|---|---|---|
| Freon-112 | Methylene chloride | Hexane | Benzene | Methanol |
| | Chloroform | Acetone | Toluene | Acetonitrile |
| | | Pentane | Xylene | Water |
| | Carbon tetrachloride | Ethyl ether | Petroleum ether | Tetrahydro-furan |
| | | Isopro-panol | Cyclohexane | Isopropanol |

*Note: Isopropanol may be disposed of in either the flammable or HPLC container.

Specially marked waste cans are available in the water quality laboratory for waste Freon-112. Freon should never be disposed of with other chlorinated solvents.

Glass jugs will not be accepted for solvent waste. Solvents must be segregated as shown and put into the red waste containers. The hazardous waste technician should be called if the solvent cans should fill during the day, and they will be emptied as soon as possible.

5. Disposal of Extracted Water Samples--Water samples which have been solvent extracted should be disposed in the extracted water waste disposal drum located in each department.

### 8.6.2 SOLID WASTES

1. Solvent saturated solids, such as sodium sulfate saturated with methylene chloride, should be disposed of in the red solid waste cans.

2. Soil samples which have been extracted with a solvent should be disposed of in the red solid waste cans in the laboratory. These containers have a closed lid to prevent solvent fumes from entering the laboratory air. Full containers will be collected by the hazardous waste technician on a regular basis. The contents of this container will be allowed to air dry and disposed of in the trash by the hazardous waste technician.
3. Disposal of Old or Contaminated Chemicals--Commercial chemicals that are out of date or contaminated should be left in their original containers. The label should be secured and the date and initials should be marked on the container. The hazardous materials technician should then be called to pick up the material.
4. Disposal of 1-milliliter (mL) and 5-mL Autosample Vials Containing Extracts--These are to be collected in the vial collection containers in each laboratory. The containers will be emptied on a regular basis by the hazardous waste technician. If the container should become full, call the technician to have it emptied.
5. Disposal of Additional Hazardous Material--The contents will be clearly marked on the container or on an accompanying analysis report. Again, clearly date and initial the container. Contact the hazardous materials technician for pickup and disposal.
6. Disposal of Unmarked Containers, or Unknowns--These are brought to the attention of the department manager. Unknowns should not be allowed to accumulate or be misplaced. Unknowns cannot be taken for disposal until they are identified.

### 8.6.3 SAMPLE WASTES

The following procedures are employed in the disposal of excess samples that have completed all necessary testing:

1. Samples stored in the coldroom will be handled as follows:
   a. Samples will be disposed of six weeks after the sampling date unless a longer storage time is authorized by the Laboratory Coordinator.
   b. Prior to disposal, a sample throw-out report will be generated by the coldroom technician and taken to the Laboratory Coordinator/Project Manager for disposal authorization on each project.

c. The Laboratory Coordinator must use the guidelines for sample disposal to determine if the sample is hazardous.

d. Nonhazardous soil samples will be disposed of by bulking them into a drum for offsite disposal. All labels on containers must be removed prior to disposal.

e. Nonhazardous water samples will be disposed by the coldroom technician in the following manner:

   1) Water samples will be bulked into the nonhazardous water waste tanks for offsite disposal.

   2) Empty sample containers should be disposed of by breaking them in the dumpster or recycle containers. Appropriate personal protective equipment (safety glasses, gloves, etc.) must be worn when breaking empty sample containers. All labels must be removed prior to disposal.

f. If samples are deemed hazardous, the Laboratory Coordinator will generate an analysis report for the involved samples. The Laboratory Coordinator's signature on this report will be the authorization for disposal of these samples. The specific compounds for which the sample is deemed hazardous should be marked. Any additional information (i.e., known contamination which was not tested for) should also be marked on the analysis report.

g. The coldroom technician will turn the samples and the analysis reports over to the hazardous waste technician.

h. The hazardous waste technician will store the samples with their report in the hazardous waste storage building until the next hazardous waste pickup.

i. During the storage time, the hazardous waste technician will combine all compatible samples to achieve the smallest overall volume.

2. Samples not stored in the coldroom will be handled as follows:

   a. The same criteria for disposal into the waste treatment system or dumpster apply to all samples.

   b. The responsible Department Manager will give a list of samples to be disposed to the appropriate Laboratory Coordinator.

c. The Laboratory Coordinator uses the guidelines for sample disposal and his knowledge of the sample to determine if the sample should be classified as hazardous.

d. The Laboratory Coordinator will generate analysis reports for those samples deemed to be hazardous. The Laboratory Coordinator will mark the specific compounds for which the sample is hazardous on the report. The Laboratory Coordinator's signature will authorize disposal. The Laboratory Coordinator will give the throw-out list and analysis reports back to the Department Manager.

e. For those samples deemed hazardous, the Department Manager will turn the samples and a signed analysis report over to the coldroom technician for disposal.

# 9.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures establish the relationship between a calibration standard(s) and the measurement of that standard by an instrument or analytical procedure. At a minimum, calibration is required: (1) when an analytical method is first set up, (2) prior to the analysis of any lot or batch of samples, (3) when the instrument detector has been subject to major maintenance, or (4) when the instrument fails the calibration QC checks.

All analytical instruments are calibrated with each use. A series of standard solutions is prepared from stock standards. These standards are either purchased from various vendors in premixed solutions or prepared directly from the stock compound. The preparation of all standard solutions is documented in a standard preparation logbook. All stock standards are dated when received, opened, and prepared (laboratory). These standards are stored in designated areas and checked for expiration dates. Specific calibration requirements for major classes of analytical procedures are described in the following sections.

## 9.1 STANDARD RECEIPT AND TRACEABILITY

Before any standard is purchased from a supplier, traceability and safety must be considered. This includes a consideration of the standards purity. The purity of the analyte of interest must be known at least to the accuracy requirements for its measurement. The manufacturer ensures this through certification and traceability statements. All laboratory standards must be traceable to a NIST (or EPA equivalent) source. Other chemicals must have a purity specification mentioned on their labels. The safety requirements are checked with the material safety data sheets (MSDS) which are supplied by the manufacturer.

Upon receipt, the standard is cross referenced to its purchase order to confirm that what was received is what was ordered. The chemical is hand delivered (special carrying case if required) to the department manager or analyst. The standard receipt date and initials

are noted on each standard. All standards are stored in designated areas for each department (Table 8-2).

## 9.2 STANDARD SOURCES AND PREPARATION

All standards used in the laboratory must be traceable to a reference source to meet the accuracy requirements as outlined in Section 5.0. The concentrations of the working solutions will depend on the calibration range of each analyte of interest. A standard is a solution of an analyte of interest with verifiable accuracy which is used to evaluate that constituent in a sample.

All new standard preparations are recorded in the appropriate standard preparation logbooks. The information recorded are the standard prepared, the source and concentration of the standard, the standard lot number, date prepared, and initials of the preparer.

The protocols for standard sources and preparation are in Table 9-1. All standards should not exceed the storage (use) life for both the stock and working solutions. Each working solution and stock solution should be labeled with date prepared, initials, concentration used, and expiration date.

Secondary dilutions made from stock standards are also recorded in the standard preparation logbook. The lot number of the stock standard used and the notebook number and/or page number will also be indicated in the logbook for traceability. Table 9-1 lists the frequency of standard preparation and storage of standards by instrument group.

## 9.3 LABORATORY INSTRUMENTS

Calibration criteria will be required for analytical operations. All the laboratory instrumentation is listed in Table 9-2. Each of these instruments will be calibrated in a manner consistent with EPA calibration protocols and/or ESE SOPs. Calibration will be documented in a parameter notebook or the analyst's notebook.

Table 9-1. Standard Sources and Preparation

| Instrument Group | Standard Source(s) | How Received | Source Storage | Preparation from Source | Lab Stock Storage | Prep Frequency |
|---|---|---|---|---|---|---|
| ICAP/MS<br>GFAA<br>CVAA<br>FLAA | Various | 1,000 ppm soln. | RT | Intermediate and/or<br>Working Stock | RT<br>RT | >5ppm Monthly<br><5ppm Daily |
| Autoanalyzer<br>($NO_3$, $PO_4$, o-Phos.,<br>CN-, TKN, $NH_4$+$NH_3$,<br>Phenol) | Various | Neat and/or<br>Solution | RT | Primary<br>Working | RT<br>RT | Monthly<br>Semiannually (CN-)<br>Daily (all others) |
| | | | | Intermediate<br>Working | RT<br>RT | Daily<br>Daily |
| IR | Various | Neat (oil) | RT | Combined primary<br>Intermediate<br>Working | RT<br>RT<br>RT | Quarterly<br>Monthly<br>Monthly |
| Visible<br>Spectrophotometer<br>UV/VIS (MBAS, $CR^{+6}$,<br>$S^{=}$, Silica) | Various | Neat | RT | Intermediate<br>Working | Refrigerator | Semiannually and<br>Monthly<br>Daily |
| TOC Analyzer (TOC, TIC) | Baxter | Neat | RT | Intermediate | RT | Monthly |
| COD Reactors | Baxter | Neat | RT | Intermediate | Refrigerator | Semiannually |
| pH Meter | Baxter | Various Buffer<br>Solutions | RT | Primary | RT | Monthly |
| Specific Ion Meter<br>(Fluoride) | Baxter | Neat | RT | Intermediate | RT | Semiannually |
| Amperiometric Titrator<br>(Acidity) | Baxter | Neat | RT | Intermediate | Refrigerator | Monthly |
| Turbimeter | Baxter | Formazin<br>Solution | RT | Working | Not stored | Daily |
| Conductivity Bridge<br>(Specific Conductance) | Baxter | Neat | RT | Primary<br>Working | Refrigerator | Monthly<br>Daily |

Table 9-1. Standard Sources and Preparation (Continued, Page 2 of 2)

| Instrument Group | Standard Source(s) | How Received | Source Storage | Preparation from Source | Lab Stock Storage | Prep Frequency |
|---|---|---|---|---|---|---|
| GC (non-VOA) | Various | Neat | Freezer | Primary | Freezer or Refrigerator | Annually |
| | | | | Intermediate | Refrigerator | Annually or Semiannually |
| | | | | Working | Refrigerator | Semiannually |
| GC (VOA) | Various (Mainly Aldrich) | Neat | Freezer | Mixed Primary | Freezer | Bimonthly |
| | | | | Working | Freezer | Bimonthly |
| | | Mixed Soln. (Early Gasses) | Freezer | Working | Freezer | Weekly |
| LC | Various | Neat | Freezer | Primary | Freezer | Annually |
| | | | | Intermediate | Freezer | Semiannually |
| | | | | Working | Freezer | Weekly |
| GC/MS (non-VOA) | Various (Mainly Suppleco) | Mixed Soln. | Freezer | Working | Freezer | Annually/Semiannually |
| GC/MS (VOA) | Various (Mainly Suppleco) | Mixed Soln. | Freezer | Working | Freezer | Semiannually |
| Radiochemistry (Counters) | Various | Soln. | RT lead-lined | Working | RT Unlined but monitored | >50% decrease in activity |

Note: RT = Room temperature.
IR = Infrared.

Source: ESE.

Table 9-2. List of Laboratory Instruments

| Analysis Type | Number | Instrument |
|---|---|---|
| Gas Chromatography/ Mass Spectrometry: | | |
| Semivolatiles | 2 | HP 5988 GC/MS/DS capillary direct with HP-7671 Autosampler[1] and HP 5987 GC/MS/DS capillary direct with HP-7671 autosampler[1]; both instruments share one HP RTE-6/VM HP 1000 computer system for data acquisition and reduction and have one HP 7959 304MB hard drive and one HP 7914 132MB hard drive for a total storage capacity of 463MB. |
| | 2 | HP 5970B GC/MS/DS capillary direct with HP 7673 Autosampler; both instruments share an HP RTE-A, HP 1000 computer system for data acquisition and reduction; and have two HP-7959 304MB hard drives for a total data storage capacity of 608MB. |
| | 1 | Finnigan INCOS50 GC/MS/DS capillary direct with HP 7673 Autosampler and HP 5890 gas chromatograph; uses a Data General DG-10 computer with 70MB hard drive; and has an IBM-PC/AT for second terminal. |
| Volatiles | 2 | HP 5995 GC/MS/DS[1] using packed column with jet separator interface and HP 5987 GC/MS/DS[1] using packed column with jet separator interface; attached to a Tekmar 2000 liquid sampler (LCS) and Tekmar 2016 sixteen position autosampler (ALS); both instruments share one HP RTE-6/VM, HP 1000 computer system for data acquisition and reduction with HP7920 50MB and HP 7914 132MB hard drives for a total data storage capacity of 314MB. |
| | 1 | HP 5970B GC/MS/DS with megabore column (DB-624, 30M x 0.53 mm ID with jet separator interface; attached to Tekmar 2000 LSC and Tekmar 2016 sixteen position ALS; uses a HP RTE-A, HP 1000 computer system for data acquisition and reduction with two HP 7959 304 MD hard drives for a total data storage capacity of 608MB. |
| | 1 | HP 5989 Engine GC/MS/DS with capillary direct megabore column, DB-624, 75M x 0.53 mm ID; attached to Tekmar 2000 LSC and Tekmar 2016 sixteen position ALS; uses a HP -425T Apollo workstation, HP-UX UNIX computer system for data acquisition and reduction utilizing the Target 2 and the Envisions software developed by THRU-PUT for HP; the compute system has to HP-6000 660MB hard drives for a total data storage capacity of 1.3GB. |

Table 9-2. List of Laboratory Instruments (Continued, Page 2 of 5)

| Analysis Type | Number | Instrument |
|---|---|---|
| | 1 | Finnigan INCOS500 XL/E GC/MS/DS with megabore column. DB-624, 75M x 0.53 mm ID with jet separator interface and Varian 3400 gas chromatograph; attached to Tekmar 2000 LCS and Tekmar 2016 sixteen position ALS; uses Data General Eclipse MV/1000 computer with 179MB hard drive and a Compaq Desk-Pro 386/20E for second terminal. |
| Gas Chromatography | 11 | HP 5890 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. Three of the GCs are attached to a HP 3350 Laboratory Data System (LDS) and 7 to a PE Nelson Data Acquisition System. |
| | 2 | HP 5890 GC configured for automatic sampling and equipped with dual Nitrogen-Phosphorus Detector. The GC is attached to a PE Nelson Data Acquisition System. |
| | 2 | HP 5890 GC configured for automatic sampling and equipped with Flame Ionization and Flame Photometric Detectors. The GC is attached to a PE Nelson Data Acquisition System. |
| | 2 | HP 5890 GC configured for automatic sampling and equipped with Photoionization, Electron Capture, and Flame Ionization Detectors. The GC is attached to a PE Nelson data Acquisition System. |
| | 1 | HP 5880 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. |
| | 2 | HP 5730 GC configured for automatic sampling and equipped with Electron Capture Detectors. The GCs are attached to a PE Nelson data Acquisition System. |
| | 1 | Shimadzu GC-14A configured for automatic sampling and equipped with dual Nitrogen-Phosphorus and Flame Ionization Detector. The GCs are attached to a PE Nelson Data Acquisition System. |
| | 2 | Varian 3400 GCs configured for automatic sampling. One GC is equipped with just a Flame-Photometric Detector and the other GC with dual Flame-Photometric and Flame Ionization Detectors. |

Table 9-2. List of Laboratory Instruments (Continued, Page 3 of 5)

| Analysis Type | Number | Instrument |
|---|---|---|
| | 4 | Tracor 540 GCs equipped with series mounted Photoionization and Electrolytic Conductivity Detectors. Two of the GCs are attached to a Tekmar Purge and Trap LCS 2000 sample concentrator and 16 position Tekmar ALS 2016 automatic sampler. And two of the other GCs are attached to a Tracor Purge and Trap LCS-2 sample concentrator and 10 position Tekmar ALS automatic sampler. The GCs are attached to a PE Nelson Data Acquisition System. |
| HPLC* | 1 | Schimadzu SCL-6A/SIL-6A (Controller/Injector) Gradient HPLC System with LC-6A pumps (2), ABI/Kratos 520 PCRS (post-column reactor), and Beckman 110B pumps(2); equipped with SPD-551 UV Detector and RF-551 Fluorescence Detector; and attached to a PE Nelson Data Acquisition System. |
| | 1 | Shimadzu SCL-6B/SIL-6B (Controller/Injector) Isocratic HPLC System with Beckman 110B pump; equipped with ABI/Kratos 757 UV Detector; and attached to a PE Nelson Data Acquisition System. |
| | 1 | Shimadzu SCL-6B/SIL-6B (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with Kratos 757 UV Detector. |
| | 1 | Shimadzu SCL-6A/SIL-6A (Controller/Injector) Isocratic HPLC System with LC-6A pump; equipped with SPD-6AV UV/Visible Detector; and attached to a PE Nelson Data Acquisition System. |
| | 1 | Shimadzu SCL-6B/SIL-6B (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with SPD-6A UV Detector; and attached to a PE Nelson Data Acquisition System. |
| | 1 | Hewlett Packard 1090 with ternary gradient solvent delivery, equipped with Diode Array Detector (DAD) and 2 UV/Visible channel outputs. |
| | 1 | Shimadzu SCL-6A/SIL-6A (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with SPD-6AV UV/Visible Detector, RF-535 Fluorescence Detector and Haake DI contant temperature circulating batch; and attached to a PE Nelson Data Acquisition System. |

Table 9-2. List of Laboratory Instruments (Continued, Page 4 of 5)

| Analysis Type | Number | Instrument |
|---|---|---|
| | 1 | Shimadzu SCL-10A/SIL-10A (Controller/Injector) Gradient HPLC system with LC-10A pumps (2); equipped with SPD-10A UV Detector; and attached to a PE Nelson Data Acquisition System. |
| Metals | 1 | Perkin-Elmer Elan 5000 Inductively-Coupled Plasma/Mass Spectrometer System |
| | 1 | Jarrell-Ash 61E Inductively Coupled Plasma Emission Simultaneous Air Spectrometer System |
| | 1 | Jarrel-Ash 1100 Inductively Coupled Plasma Emission Simultaneous Air Spectrometer System. |
| | 1 | Perkin-Elmer Inductively Coupled Plasma Emission Sequential Spectrometer System. |
| | 2 | Perkin-Elmer Model 5100 Atomic Absoption Spectrophotometer System equipped with Flame and Graphite Furnace Model HGA 600 with Zeeman Backgound Correction. |
| | 1 | Perkin-Elmer 5100 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace Model HGA 600 and Zeeman Background Correction. |
| | 1 | Perkin-Elmer Model 4100 ZL Atomic Absoption Spectrophotometer System equipped with Graphite Furnace and Zeeman background correction. |
| | 1 | Perkin-Elmer 3030 Atomic Absoption Spectrophotometer System equipped with Graphite Furnace and Zeeman Background Correction. |
| | 2 | Perkin-Elmer Models MHS 20 and 50B Cold Vapor Mercury Analyzer. |
| | 1 | Buck Scientific 400 Cold Vapor Mercury Analyzer. |
| | 1 | Perkin-Elmer Model 3100 Atomic Absoption Spectrophotometer equipped with Flame, Hydride Generator, and cold Vapor attachment and $D_2$ background correction. |
| | 1 | Merlin Hg-1000 Atomic Absorption/Fluorescence Mercury Analyzer System |
| | 1 | Perkin-Elmer FIAS-200 automatic flow injection Mercury/Hydride System with amalgamation attachment. |

Table 9-2. List of Laboratory Instruments (Continued, Page 5 of 5)

| Analysis Type | Number | Instrument |
|---|---|---|
| Inorganics | 1 | Dorhman DC-190 TOC Analyzer. |
| | 6 | Dionex 4000I (4) and 2000I (2) Ion Chromatographs with autosampler. |
| | 6 | Technicon II (5) and TRACCS 800 (1) Autoanalyzer. |
| | 3 | HACH 16500 COD Reactors. |
| | 6 | Corning 125 (3) pH meters. |
| | 2 | Orion 801 and 701 Specific Ion Meters with 6-position electrode switch. |
| | 1 | Perkin Elmer 1420 Scanning Infrared Spectrophotometer. |
| | 1 | Perkin-Elmer 552 UV-Visible Spectrophotometer. |
| | 1 | Baush & Lomb Spectronic 20 Visible Spectrophometer. |
| | 1 | Wallace-Tiernan Amperiometric Titrator. |
| | 3 | Hach 2100A Turbidimeter |
| | 1 | Hach Ratio Turbidimeter |
| | 8 | Metler H80 (1), Metler AE160 (5), and Metler PC2000 (2) Analytical Balances |
| | 1 | Ohaus Analytical Balance |
| | 2 | Sartorius Analytical Balance |
| Radiochemistry | 2 | EG&G Berthold LB770-2 10 Channel Simultaneous Low Background Gas Flow Proportional Counter with PC Interface-2. |
| | 1 | EG&G Ortec Multichannel Analysis (MCA) System linked with one 20% High Purity Germanuim Model GEM20180 Detector, one 35% High Purity Germanium Model GEM 35185 Detector, two EG&G Ortec Octete (16-Detector) PC Model 1000 Detectors Alpha Spectroscopy System, and a Bicron Model P-14-W Sodium Iodide Detector. |
| | 1 | Beckman LS1801 Liquid Scintillation Detector. |
| | 12 | Ludlum Model 312 Alpha Scintillation Detectors with Model 1000 Scalers. |
| | 1 | R.J. Harvey OX-600 Biological Oxidizer System. |

The other HPLC equipments we have that can be attached to any of the five HPLC systems, if needed are: RF-535 Fluorescence Detector; Raytest Ramona 5-LS Radiochemistry Detector; Beckman 110A pump; Gilson FC-80K fraction collector; Shimadzu FCV-2AH column switching valve; Waters WISP 712 autosampler; Altex 210 manual injector; and Wescan Conductivity Detector.

Specific calibration requirements for major classes of analytical procedures are described in Sections 9.3.1 through 9.3.12. If the calibration requirements of the specified analytical method are more stringent than the procedures described in this LCQAP, the method procedures will be followed.

## 9.3.1 GAS CHROMATOGRAPH/HIGH PRESSURE LIQUID CHROMATOGRAPH (GC-NONVOLATILES/HPLC) CALIBRATION

Standard Curve Calibration--Initial calibration standard solutions will be prepared by sequential dilution of a single stock standard solution to cover the analytical working range of the method. These may be either composite standards of more than one analyte or single-analyte solutions. The concentrations will be adjusted to take into account the instrumental and method detection limit. A minimum of three initial calibration standard concentrations or the number of standards specified by the method covering the working range and a blank will be prepared and analyzed. The initial calibration standards and the blank will be analyzed in every analytical run. For EPA 608 and 8080 methods only, the required calibration standards and a blank are analyzed every analytical run for single-component pesticides. However, for multi-component pesticides (Methods 608 and 8080 only one calibration standard of each pesticides will be analyzed. This standard will be used qualitatively to determine if there are hits in the samples. If there are hits in the samples, initial calibration standards of the appropriate multi-component pesticides and samples are reanalyzed. At least one calibration standard at the middle or high range of the curve will be analyzed every 20 samples and repeated at the end of the run. A QC check standard is analyzed every time new calibration standards are prepared and analyzed to verify acceptability of the new calibration standards.

The initial calibration curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. The concentrations of the standards may be expressed in units of mass injected or in terms of the concentration of the standard solution, if the injection volume is constant for standards and samples. QC evaluation criteria for initial calibration, recalibration, and continuing calibrations are as follows:

1. The initial calibration curve and the subsequent recalibrations possess a minimum of three points and a blank or possess the number of calibration standards specified by the method,
2. The correlation coefficient of the curve is 0.995 or greater,
3. Continuing calibration standards are within 15 percent of the same initial calibration standard for GC (25 percent for NP detector) and within 10 percent of the same initial calibration standard for HPLC,
4. The QC check standard must be within the acceptance range provided by the vendor or within 25 percent of the standard's true if a standard from a different source or lot number is used, and
5. The calibration curve brackets the response for all samples.

Corrective actions taken if these calibration QC criteria are not met are listed in Section 13.0.

The concentration (or amount) of the injected sample will be obtained by entering the response for the sample into the initial calibration curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

For Los Alamos project, calibration requirements for organochlorine pesticides and PCBs specified in CLP SOW 12/90 will be followed.

### 9.3.2 GAS CHROMATOGRAPH (GC-VOLATILES) CALIBRATION

Standard Curve Calibration--Calibration standard solutions will be prepared as needed by sequential dilution of a single stock standard solution (prepared every 2 months) to cover the analytical working range of the method. These may be either composite standards of more than one analyte or single-analyte solutions. The concentrations will be adjusted to take into account the instrumental and method detection limit. A minimum of three calibration standard concentrations, or the number of standards specified by the method

covering the working range and a blank, will be prepared and analyzed. The calibration standards and the blank will be analyzed in every analytical run. At least one calibration standard at the middle to high range of the curve will be analyzed every 20 samples and repeated at the end of the run to ensure constant instrument response. A QC check standard is analyzed every time new calibration standards are prepared to verify acceptability of the new calibration standards. Calibration is as described in Section 9.3.1.

### 9.3.3 GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) TUNING AND CALIBRATION

GC/MS Tuning--Daily instrument tuning will be practiced to ensure the instrument is calibrated and in proper working condition. The GC/MS will be tuned daily with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and bromofluorobenzene (BFB) for volatiles analysis. The mass intensity specifications for BFB and DFTPP are contained in Table 9-3. ESE performs mass calibration in conjunction with the daily instrument tuning.

Table 9-3. Mass Intensity Specifications for DFTPP and BFB

| Key Ions | Ion Abundance Criterion |
|---|---|
| For DFTPP* | |
| 51 | 30 to 60 percent of mass 198 |
| 68 | Less than 2 percent of mass 69 |
| 70 | Less than 2 percent of mass 69 |
| 127 | 40 to 60 percent of mass 198 |
| 197 | Less than 1 percent of mass 198 |
| 198 | Base peak, 100-percent relative abundance |
| 199 | 5 to 9 percent of mass 198 |
| 275 | 10 to 30 percent of mass 198 |
| 365 | Greater than 1 percent of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | Greater than 40 percent of mass 198 |
| 443 | 17 to 23 percent of mass 442 |
| For BFB* | |
| 50 | 15 to 40 percent of mass 95 |
| 75 | 30 to 60 percent of mass 95 |
| 95 | Base peak, 100-percent relative abundance |
| 96 | 5 to 9 percent of mass 95 |
| 173 | Less than 2 percent of mass 174 |
| 174 | Greater than 50 percent of mass 95 |
| 175 | 5 to 9 percent of mass 174 |
| 176 | Greater than 95 percent but less than 101 percent of mass 174 |
| 177 | 5 to 9 percent of mass 176 |

*Reference: Test Methods for Evaluating Solid Waste, EPA-SW-846, 3rd Edition, November 1986.

Source: ESE.

GC/MS Calibration--Relative response factors for the individual compounds will be determined as follows:

$$RF = \frac{A_C \, Q_{IS}}{A_{IS} \, Q_C}$$

where: A = integrated area taken from the extracted ion current profile,
Q = quantity of material,
C = compound, and
IS = internal standard.

Initial calibration, using a minimum of five levels of the compound, will be used to determine the instrument linearity. The average response factor (RF) will be calculated for each compound. The response factors for the System Performance Check Compounds (SPCC) must be $\geq$0.30 (0.25 for bromoform) for EPA 8240 and $\geq$0.05 for EPA 8270. The percent relative standard deviation (% RSD) will be calculated for each calibration check compound (CCC). The percent RSD of the CCCs in the initial calibration must be $\leq$30 percent ($\leq$35 percent for EPA Method 625 only).

A 1-point calibration using a midlevel standard from the initial calibration will be used daily for all subsequent analysis. The RFs of the SPCC for EPA 8240 and 8270 in this continuing calibration standard must meet the minimum response factors specified for the initial calibration previously mentioned. The RFs of the calibration check compounds in this daily calibration standard should be $\leq$25 percent ($\leq$20 percent for EPA Method 625 only and $\leq$ 30 percent for EPA Method 8270) difference from the average RFs in the initial calibration. All other analytes should have a percent difference of 30 percent. Corrective actions taken if the QC criteria for calibrations are not met are listed in Section 13.0.

The minimum required internal standards (IS) are chlorobenzene-d5, 1,2-dichloroethane-d4, and 1,4-dichlorobenzene-d4. for volatiles and 1,4-dichlorobenzene-d4, napthalene-d8, acenapthene-d10, phenanthrene-d10, chrysene-d12, and peylene-d12 for semivolatiles. A retention time and response check will be performed on every internal standard for samples that will be analyzed. The retention time (RT) of the IS in the sample must be $\leq \pm$ 30 seconds from the previous daily calibration. The response area of the IS in the sample should not be > factor of 2 (-50% to +100%0 from the previous calibration.

### 9.3.4 GENERAL INORGANIC AND ORGANIC PARAMETERS CALIBRATION

Standard Curve Calibration--This section applies to those inorganic and organic analyses procedures [ion chromatography, colorimetric, spectrophotometric, potentiometric, infrared (IR) and ultraviolet (UV) absorption, turbidimetric] that use a standard curve for calibration [except total organic carbon (TOC) and chemical oxygen demand (COD)]. Working standard solutions will be prepared by sequential dilution of a single-stock standard to bracket the analytical working range of the method. Working standard solutions may be either composite standards of more than one analyte or single-analyte solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of every analytical run, and at least one midlevel standard, which is the continuing calibration verification (CCV) standard, will be reanalyzed at minimum intervals of every 20 samples and at the end of the run to check for constant instrument response.

The preparation of calibration standards is verified by the analysis of the ICV solution. The initial calibration verification (ICV) is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically, an EPA or NIST reference is used as the ICV and is prepared according to the supplier's instructions.

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1. The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;
3. The response for the CCV analyzed at minimum intervals of every 20 samples (every 10 samples for cyanide for Los Alamos project) during the run and at the end of the run is within 20 percent of true value (15 percent of true value for cyanide for Los Alamos project);
4. The ICV is within 10 percent of the element's true value; and
5. The calibration curve brackets the response for all samples.

Corrective action procedures taken if these QC evaluation criteria are not met are provided in Section 13.0. The sample concentration will be obtained by entering the response for the sample into the working curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

### 9.3.5 TRACE METALS ANALYSIS CALIBRATION

Atomic Absorption Spectroscopy (AAS) Standard Curve Calibration--Working standard solutions will be prepared to include the analytical working range of the method; these solutions may be either composite standards of more than one metal or single-metal solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of every analytical run, and at least one midlevel standard will be analyzed at minimum intervals of every 20 samples (every 10 samples for cyanide for Los Alamos project) during the run and at the end of the run to check for constant instrument response.

The calibration is verified by the analysis of the ICV solution. The ICV is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically an EPA or NIST reference is used as the ICV and is prepared according to the supplier's instructions. For Los Alamos project, the ICV for cynanide analysis should be distilled with the batch of samples analyzed.

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1. The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;
3. The response for the midlevel standard analyzed at minimum intervals of every 20 samples (every 10 samples for Los Alamos project) during the run and at the end of the run is within 20 percent of true value (10 percent of true value for GFAA for Los Alamos project);
4. The ICV is within 10 percent (20 percent for CVAA for Los Alamos project) of the element's true value; and
5. The calibration curve brackets the response for all samples.

Refer to Section 13.0 for the corrective action procedures taken if these QC evaluation criteria for calibration are not met.

The concentration of the sample is obtained by entering the response for the sample into the working curve equation and determining the sample concentration after all appropriate digestate and sample dilution factors have been applied.

Inductively Coupled Argon Plasma (ICAP) Single Point Calibration--This procedure uses a single standard concentration for each element to obtain an instrument response (emission counts) and is analyzed in every analytical run. A second single point, emission counts obtained when aspirating a blank solution (undigested, acidified DI

water), is used in conjunction with the standard to calibrate the instrument in concentration units.

The calibration is verified by the analysis of an ICV solution, which is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. The elemental concentrations of the calibration verification solution must be within the calibration range of the instrument and at concentrations other than those used for instrument calibration.

A multi-element interference check solution (ICS) and a method blank (acidified DI water that is carried through the digestion process) are analyzed each day prior to analyzing the samples. For Los Alamos project, the ICS will be analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS is used to verify the correction of spectroscopic interference caused by emissions adjacent to analyte emission lines.

The CCV solution is analyzed at minimum intervals of every 20 samples (10 samples for Los Alamos project) during the run and at the end of the run to document constant instrument response. This solution contains one-half the concentration of each element present in the calibration standards. This solution may be prepared by dilution of an aliquot of the calibration standard or prepared as a separate solution in a manner analogous to the calibration standard preparation procedure.

QC evaluation criteria for the instrument calibration standard are as follows:

1. A calibration standard and a calibration blank are used.
2. All the values for the ICV are within 10 percent of each element's true value.
3. Values for the ICS are 20 percent of each element's true value.
4. The measured concentrations of the elements in the CCV solution, for which calibration was performed, are within 10 percent of their respective true values.

Corrective action procedures if these QC evaluation criteria are not met are provided in Section 13.0.

Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) Single Point Calibration -- This procedure uses a single standard concentration for each element to obtain an instrument response (intensity counts) and is analyzed in every analytical run. A second single point, intensity counts obtained when aspirating a blank solution (undigested, acidified DI water), is used in conjunction with the standard to calibrate the instrument in concentration units.

The calibration is verified by the analysis of an ICV solution, which is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. The elemental concentrations of the calibration verification solution must be within the linear range of the instrument and at concentrations other than those used for instrument calibration.

A multi-element interference check solution (ICS) and a method blank (acidified DI water that is carried through the digestion process) are analyzed each day prior to analyzing the samples. For Los Alamos project, the ICS will be analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS is used to verify the adequate application of elemental interference equations.

The CCV solution is analyzed at minimum intervals of every 10 samples during the run and at the end of the run to document constant instrument response. This solution contains one-half the concentration of each element present in the calibration standards. This solution may be prepared by dilution of an aliquot of the calibration standard or prepared as a separate solution in a manner analogous to the calibration standard preparation procedure.

QC evaluation criteria for the instrument calibration standard are as follows:

1. A calibration standard and a calibration blank are used.
2. All the values for the ICV are within 10 percent of each element's true value.
3. Values for the ICS are adequately corrected for in the interference equations.
4. The measured concentrations of the elements in the CCV solution, for which calibration was performed, are within 10 percent of their respective true values.

Corrective action procedures if these QC evaluation criteria are not met are provided in Section 13.0.

### 9.3.6 GRAVIMETRIC METHODS CALIBRATION

Two general types of analytical balances are used at ESE: (1) the more sensitive microanalytical balance, and (2) the top-loading balance. The calibration of the microanalytical balances is verified daily by weighing the following Class S and NIST-certified weights [in grams (g)]:

| Weight (g) | Tolerance Limits |
|---|---|
| 0.2 | ± 0.0005 |
| 1.0 | ± 0.0005 |
| 3.0 | ± 0.0005 |
| 5.0 | ± 0.0005 |

The calibration of the top loading balances are verified daily by weighing the following Class S and NIST-certified weights:

| Weight (g) | Tolerance Limits |
|---|---|
| 5 | ± 0.02 |
| 20 | ± 0.05 |
| 50 | ± 0.05 |

The results are recorded in the instrument logbook. If these criteria are not met, the weight may be reweighed. If the criteria are not met for the second weighing, the balance is taken out of service and repaired. The Class S weights are sent to the

manufacturer yearly for calibration and recertification. Two sets of Class S weights are available in-house.

Qualified service personnel calibrate the analytical balances semiannually. The semiannual calibration is documented by a tag on the instrument. A set of NIST-certified weights is used to check the calibration daily. Results are recorded in the instrument notebook.

### 9.3.7 TITRIMETRIC METHODS CALIBRATION

In all cases, standard reference materials are used to calibrate the titrant and back titrant. Preparation of these materials is described in Standard Methods or other methods manuals. Known solutions of the parameter to be analyzed will be prepared and analyzed to verify titrant standardization and the analyst's ability to discern the endpoint.

### 9.3.8 TOC CALIBRATION

The Dohrman TOC analyzer is calibrated with a standard reference material using a single-point calibration. The standard is analyzed before beginning every analytical run. The linearity of the calibration is verified with a low-level and high-level standard to bracket the sample concentration. The linearity checks must be within 5 percent. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples and at the end of the run, and the response must be within $\pm$15 percent of true value.

### 9.3.9 COD CALIBRATION

A reference material will be used to verify the 0- and 500-mg/L reading to the standard curve developed by Hach Chemical Company for COD on a spectrophotometer using the prepared sample vials. The 500-milligrams-per-liter (mg/L) standard must be within 5 percent.

### 9.3.10 BOD CALIBRATION

The oxygen probe is calibrated daily according to the manufacturer's air calibration procedure. The temperature of the incubator used for the BOD analysis will be read and recorded twice daily when in use.

### 9.3.11 TOTAL ORGANIC HALIDES (TOX) CALIBRATION

The TOX analyzer is calibrated with a standard reference material using a 3-point calibration. The linearity of the calibration is verified with a low-level and high-level standard to bracket the sample concentration. The linearity checks must be within 5 percent. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples, and the response must be within $\pm$15 percent of true value.

### 9.3.12 RADIOCHEMISTRY CALIBRATION

In compliance with the State of Florida DHRS Radioactive Materials licensing regulations, control charts for efficiencies and backgrounds are kept for all instruments used in radiochemical counting. All standards used in calibrations and QC spiking are either from NIST or EPA. Count rates are calculated using computer software.

Alpha/Beta Proportional Counter--The 10-chamber, low-background alpha/beta proportional counting system is calibrated for counting efficiencies on a quarterly basis with Am-241 and Cs-137 standards. The alpha/beta self-absorption calibration curve for each counting chamber is determined biannually. Performance check standards are counted prior to each analytical run, and count rates must be within historical control limits. Background is also checked prior to each analytical run, and the count rate must be within historical control limits. Historical limits for the performance check standards and backgrounds are generated quarterly.

Liquid Scintillation Counter--The Beckman Liquid Scintillation Counting System is calibrated prior to each run of samples with a set of check sources and applicable standards (tritium, C-14, Pb-210, and Ra-226) provided by the manufacturer. The H

Number Quench Efficiency Correction Curve is derived from the standards and applied to the data to account for counting efficiencies.

Lucas Cell Readers for Ra-226 Counting--Each Lucas Cell and the 12 matching cell readers are calibrated with known Ra-226 standard annually. The performance check standard is analyzed prior to each instrument use, and count rates must be within historical control limits generated quarterly. A background check is also performed prior to each instrument use, and the reading must be $\leq$ 1 cpm.

Gamma Spectroscopy--Both the Na(l) and Ge(Li) detector systems are calibrated before each analysis with known source standards, depending on the matrix to be analyzed. Performance standards are counted prior to each instrument use, and count rates must be within historical limits generated quarterly. Background is also checked prior to each instrument use.

Alpha Spectrometers-- Alpha spectrometers are calibrated biannually for efficiency with known electroplated standard sources. Detectors are checked for performance daily by counting standard sources, and count rates must be within historical control limits generated quarterly. Background is checked weekly, and the count rate must be $\leq$ 0.02 cpm in any channel.

### 9.3.12.1 TRACER AND CARRIER RECOVERY ACCEPTANCE CRITERIA

All alpha activity measurements by alpha spectrometry require the use of another isotope of the same element as a tracer. A known activity of the tracer is added to the sample at the beginning of the analysis and subsequently measured by alpha spectrometry. Recovery of this tracer should be at least 40%. Samples with tracer recoveries less than 40% require reanalysis.

Analysis of the Radium-228, Strontium-89, Strontium-90, and Lead-210 require the addition of other elements as chemical carriers. The following is a list of the common chemical carriers used for these isotopes and their required minimum percent recoveries:

Radium-228

| | |
|---|---|
| Yttrium Carrier | 40% Recovery |
| Barium Carrier | 40% Recovery |

Strontium-89/90

| | |
|---|---|
| Yttrium Carrier | 40% Recovery |
| Barium Carrier | 40% Recovery |

Lead-210

| | |
|---|---|
| Lead Carrier | 40% Recovery |
| Bismuth Carrier | 40% Recovery |

Samples with carrier recoveries less than the above minimum recoveries require reanalysis.

### 9.3.13 pH CALIBRATION

Calibrate the pH meter with three buffer solutions at pH 4, 7, and 10 prior to use. Set the pH meter temperature selector to ambient temperature. Place the probe on the pH 7 buffer and adjust the calibration switch until it reads 7.00. Repeat the procedure with the pH 4 buffer solution. Read the pH of the pH 10 buffer solution. It should be 10 $\pm$ 0.05; if not, check the pH probe and internal solution and repeat the calibration procedure.

### 9.3.14 SPECIFIC CONDUCTIVITY CALIBRATION

Calibrate the instrument with 0.01 M and 0.10 M KCL solutions. The conductivity reading of the 0.01 M KCL must be 1,413 umhos $\pm$ 15% and the 0.10 M KCL 12,900 $\pm$ 15%. If the calibration standards are outside the acceptance criteria, prepare new standards and recalibrate the instrument.

### 9.3.15 PENSKY-MARTENS CLOSE-CUP TESTER CALIBRATION

Determine the ignitability of the p-xylene standard prior to use of the Pensky-Martens Close-Cup Tester. The standard should ignite at 27.2 $\pm$ 1°C. If not, check the condition and operation of the apparatus, especially the tightness of the lid, the action of the shutter, and the position of the test flame. After adjustment, repeat the test with the p-xylene standard. Read and record the barometric pressure at the time of analysis.

### 9.3.16 DISSOLVED OXYGEN CALIBRATION

The dissolved oxygen probe should be calibrated daily or prior to use in saturated air by moving the calibration knob such that the reading is at the appropriate saturation value indicated on the instrument. Read and record the temperature at the time of reading.

## 9.4 STANDARDIZATION OF TITRATION SOLUTIONS

All titrants used in the laboratory are standardized against a primary standard. This ensures that the normality of the standard being used is at the correct level. Table 9-6 lists the solutions that require standardization, the standards used, and the frequency of standardization.

Table 9-4. Standardization of Titrating Solutions

| Solutions Req. | Primary Standard Source | Frequency of Standardization |
|---|---|---|
| Chloride:<br>Silver nitrate | Sodium chloride | Every run |
| Alkalinity:<br>Sulfuric acid | Sodium carbonate | Every run |
| Sulfite:<br>Potassium iodide-iodate | Sulfamic acid | Every run |
| Hardness:<br>EDTA | Calcium carbonate | Every run |

Source: ESE.

# 10.0 PREVENTIVE MAINTENANCE

To minimize the occurrence of instrument failure and other system malfunctions, a preventive maintenance program for laboratory instruments is implemented. Routine maintenance is performed as needed depending on how often the instrument is used. There are some parts of the instrument that will wear out faster and therefore will require replacement more frequently than the others. These wearable or expendable parts are kept in supply and evaluated during analysis. The major instrumentation in the laboratory is covered by the manufacturer's service contracts or agreements.

## 10.1 DOCUMENTATION

All maintenance performed on the instruments are documented in each instrument's maintenance logbook which is kept with the instrument. The date, initials of the analyst performing the maintenance, and the type of maintenance performed are recorded in this maintenance logbook. Receipts from the routine maintenance performed by the manufacturer's representative are kept in folders and filed in the department's file cabinets. Preventive maintenance for each major piece of laboratory equipment is listed in Table 10-1.

## 10.2 CONTINGENCY PLAN

In the event of instrument failure, every effort will be made to analyze samples within holding times by alternate means. If the redundancy in equivalent instrumentation is insufficient to handle the affected samples, efforts will be made to secure the same or equivalent analyses by a NEESA, HAZWRAP, USACE or FDEP-approved laboratory, when required. The Project Manager will be advised of any required changes in methodology or location; the Project Manager should then notify the state or government agency or the client.

Table 10-1. Preventive Maintenance

| Instrument | Activity | Frequency |
|---|---|---|
| Gas Chromatographs | Change septums | Weekly or as needed |
| | Check carrier gas | Daily |
| | Change carrier gas | As needed (when pressure falls below 100 psi) |
| | Cut off edge of a capillary column | As needed |
| | Replace oxygen traps used in the gas lines | Annually or as needed |
| | Clean ECD | Annually or as needed |
| | Replenish Electrolytic Conductivity Detector | Monthly or as needed |
| | Clean detectors | Annually or as needed |
| | Check system for gas leaks | At each column change |
| High Performance Liquid Chromatographs | Replace piston seals | Quarterly |
| | Replace or rebuild the the check valves | As needed (when performance of the instrument decreases) |
| | Clean detector flow cell | As needed |
| | Check pumps | As needed |
| | Replace guard column frits | As needed (when the HPLC system pressure increases) |
| | Clean detectors | Annually or as needed |
| Gas Chromatograph/Mass Spectrometer | Clean source and system | As needed |
| | Cut off ends of capillary columns | As needed |
| | Change columns | As needed |
| | Change injection point lines | Monthly or as needed |
| | Routine maintenance performed by the manufacturer | Annually |
| Atomic Absorption Spectrophotometers (Furnace and Cold Vapor) | Clean furnace windows | Daily |
| | Check plumbing connections | Daily |
| | Change graphite tubes | Daily or as needed |
| | Clean sample cells | Daily |
| | Check gases | Daily |
| | Check optics | Annually (on contract) |
| | Change graphite contact rings | As needed |
| Inductively Coupled Plasma (ICAP) | Routine maintenance performed by the manufacturer | Annually (on contract) |
| | Clean the torch and nebulizer | Every six months or as needed |
| | Check tubing | Daily |

Table 10-1. Preventive Maintenance (Continued, Page 2 of 4)

| Instrument | Activity | Frequency |
|---|---|---|
| Inductively Coupled Plasma/ Mass Spectrometer | Routine maintenance performed by the manufacturer | Annually (on contract) |
| | Clean the torch and nebulizer | Every six months or as needed |
| | Check tubing | Daily |
| | Change oil in rotary vane | Quarterly or as needed |
| | Check interface cones | Daily |
| | Check gas pressure, gas flows, and vacuum pressure | Daily |
| Autoanalyzers | Clean tubing | As needed |
| | Check tubings | Daily |
| | Check optics | Daily |
| | Clean optics | As needed |
| | Replace the lamp | As needed (when darkening is evident) |
| Colorimeter/ Turbidimeters | Check optics | Daily |
| | Check light source | As needed |
| Spectrophotometer | Routine maintenance performed by the manufacturer | Quarterly (on contract) |
| TOX Analyzer | Clean electrodes | Daily |
| | Replace all solutions | Daily |
| | Clean absorber module and the furnace unit | Every six months or as needed |
| | Clean sampler boat | Monthly |
| | Check gases and tubing | Daily |
| TOC Analyzer | Check gases and tubing | Daily |
| | Change pump tubes | Prior to each use |
| | Flush digestion tubes | After each use |
| Ionanalyzers/Conductivity | Check probe | Daily |
| | Change probe solution | As needed |
| Ion Chromatograph | Routine maintenance performed by the manufacturer | Every six months (on contract) |
| | Check system for leaks | Daily prior to each run |
| | Check line pressure | Daily prior to each use |
| | Clean conductivity cells | Every six months |
| | Clean injection loops | Every six months |
| | Change columns | As needed |
| | Replace tubings in the sample path | Every six months |

Table 10-1. Preventive Maintenance (Continued, Page 3 of 4)

| Instrument | Activity | Frequency |
|---|---|---|
| Turbidimeter | Clean the instrument | Prior to each use |
| DO Meter and Probe | Check to make sure that the mechanical zero is set properly | Prior to each use |
| | Check DO probe membrane | Prior to each use |
| DO Meter and Probe (cont) | Replace membrane | As needed (when tears, wrinkles, or bubbles are observed) |
| | Replace probe | As needed |
| Analytical Balances | Clean the balance | Daily |
| | Check alignment and balance | Daily |
| | Routine maintenance and calibration performed by the manufacturer | Semiannually |
| Radiochemistry: | | |
| Alpha/Beta Proportional Counter | Check gas flow | Daily |
| | Check counting chambers | Monthly or as needed |
| Liquid Scintillation Counter | Check counting system | Prior to each use |
| Alpha Spectrometer | Clean detectors | As needed |
| | Clean sample chambers | As needed |
| | Check vacuum | Daily |
| | Check voltage | Daily |
| Radon Flask Counters | Clean the face of the photomultiplier tube | Daily |
| | Check microswitch | Daily |
| Gamma Spectroscopy | Refill liquid nitrogen in the dewar for the Ge(Li) detector | Weekly |
| | Check all cabling to the gamma detectors | Monthly |
| Biological Oxidizer | Clean ladel | Prior to each use |
| | Clean sample boats | Prior to each use |
| Ovens: TS, TSS, TDS | Check temperature | Prior to use |
| | Calibrate thermometer | Annually |

Table 10-1. Preventive Maintenance (Continued, Page 4 of 4)

| Instrument | Activity | Frequency |
|---|---|---|
| Refrigerators/Freezers | Check temperature | Daily |
| | Calibrate thermometer | Annually |
| BOD Incubator | Check temperature | Prior to use |
| | Calibrate thermometer | Annually |

Note: TDS = total dissolved solids. TS = total solids. TSS = total suspended solids.

# 11.0 QC CHECKS, ROUTINES TO ASSESS PRECISION AND ACCURACY, AND CALCULATION OF METHOD DETECTION LIMITS

## 11.1 INTERNAL QC CHECKS

Analytical QC procedures are those steps taken by the laboratory in day-to-day activities to achieve the desired accuracy, precision, reliability, and comparability of analytical data. Each Laboratory Department Manager and coordinator is responsible for performing the analysis in accordance with the defined quality control practices outlined in this LCQAP.

For all analyses performed by ESE, the QC checks described in this section are mandatory unless alternate procedures are given in the specific project QA Plan or otherwise agreed upon by the Laboratory Coordinator and ESE's client. Table 11-1 summarizes minimum QC sample requirements. If method QC requirements are more stringent than those listed in Table 11-1, the method requirements will be followed. Sections 3.0 and 5.0 contain QC evaluation criteria for laboratory methods and calibrations. Section 11.4 describes precision and accuracy calculations used to control samples. Laboratory Department Managers are responsible for reviewing QC criteria for each method performed by their department. Permanent changes to the acceptance criteria must be approved by the Laboratory Department and Division Managers and will be incorporated into this document in accordance with Section 3.4, Document Control. Project-specific revisions may be documented in the specific project QA Plan (DER Form 17-160.900 for FDEP projects only).

Table 11-1. Minimum QC Sample Requirements[+]

| Analysis | Standard Matrix (QC Check Standard) Blank | Standard Matrix (QC Check Standard) Spike* | Sample Matrix** Spike | Sample Matrix** Replicate Spike | Sample Replicate | Surrogate Spike | Filter Blank (as required) |
|---|---|---|---|---|---|---|---|
| INORGANIC | | | | | | | |
| [a]All analyses except b,c,d | 5% | 5% | 5% | 5%++++ | -- | -- | 5% |
| [b]pH, residues, specific conductivity, turbidity, dissolved oxygen | 5% | -- | -- | -- | 5% | -- | -- |
| [c]Radiochemistry only | 5% | 5% | 5% | 5%++++ | 10%+++ | -- | 5% |
| [d]TCLP | 5% | 5% | ++ | -- | -- | -- | 5% |
| ORGANIC | | | | | | | |
| [a]All analyses | 5% | 5% | 5%**** | 5%**** | -- | 100%*** | 5% |
| [d]TCLP | 5% | 5% | ++ | -- | -- | 100%*** | 5% |

Note: -- = not applicable for this analysis.

*Standard Matrix Spike (QC Check Standard) is a spike into a blank matrix which is carried through sample preparation, sample digestion or extraction to sample analysis. The blank matrix is a reagent blank for aqueous samples and a standard soil for solid matrix, if available; if standard soil is not available, spiking is done on a reagent blank. This spike is also called a QC Check Standard because the standards used to prepare the spiking solution are from a different source than those used for the calibration standards.

+Actual number rounded up to nearest whole number, i.e., 5% = 1QC for 1-20 samples; 2QC for 21-40, etc.

**Sample Matrix Spike is a spike into a sample matrix which is carried through sample preparation, sample digestion, or extraction to sample analysis.

++5%, or 1 per waste type, whichever is greater.

***Surrogate(s) will only be spiked into all environmental and QC samples if specified by the method.

+++Required for drinking water analyses and Los Alamos project only.

****For Los Alamos project, sample matrix spike and sample matrix spike duplicate for organic analysis are not required, however, they will be analyzed if requested by the client.

++++For inorganic analysi, sample matrix duplicate is not required for Los Alamos project but will be analyzed upon request by the client.

Source: ESE.

For QC purposes, the number of samples extracted and/or prepared for instrumental analysis as one group in one 24-hour period will constitute a batch. The number and type of QC samples specified in Section 11.0 will apply to this sample batch. For example, a group of samples that is extracted on the same day and (if required) undergoes concentration and cleanup procedures on subsequent days would be considered one sample lot for QC purposes.

For analyses where no sample extraction or preparation is required, the number of samples that can be analyzed as one set during a 24-hour period will determine the number of samples per sample lot for QC purposes.

When required by the specific project QA Plan, the Project QA Coordinator may insert into a sample lot either a spiked sample or a duplicate of a previously analyzed sample for QC purposes. The Project QA Coordinator will monitor the results of this sample to ensure that the analysis meets QA criteria for the project.

Blind QC check samples are analyzed by the laboratory semiannually to evaluate the laboratory's overall system. If the blind QC check sample data are not acceptable, results will be reported in the QA report to FDEP.

Spikes will be placed into sample matrices for all analyses except pH, residues, specific conductivity, and turbidity. Samples will be split into duplicates, spiked, and analyzed. The relative-percent difference between the spike and the replicate spike will be used to assess analytical precision. Selection of the sample to be split and spiked may be made by the client or by the laboratory.

Control spikes (standard matrix spikes or QC check standards) will be placed into standard matrices for all analyses except pH, residues, specific conductivity, and turbidity. This spike will be used to control the method and verify the calibration

standards, if an ICV is not analyzed. A sample replicate will be prepared and analyzed for pH, residues, specific conductivity, and turbidity. The relative-percent difference between the sample and the replicate will be used to assess analytical precision.

It is ESE's policy to control sample analyses on those QC criteria that are actually under the control of the technicians and analysts performing the analytical procedure. Therefore, emphasis is placed on calibration, method blanks, and QC check standard (standard matrix spike) results. When these are within criteria, acceptable method performance is documented. Sample matrix spikes will be reported and evaluated for precision and accuracy but not necessarily used for method control. A sample matrix spike that has recoveries outside of criteria limits will be evaluated against other available QC data within a batch to determine if the method is in control and if sample flagging is warranted. Failure of a sample matrix spike to achieve the acceptance criteria when a QC check sample in the same batch has acceptable recoveries often documents only that the method employed is not applicable to that particular matrix, not that the method is out of control.

In the following subsections, those criteria marked with an asterisk (*) will be used to control the sample analysis. Precision and spike recovery checks are discussed in further detail in Section 11.4. In addition to the QC samples specified in the following subsections, field QC blanks must be prepared and analyzed as described in Section 11.1.

### 11.1.1 GC/MS MINIMUM QC

For GC/MS analyses, the following minimum QC checks will apply, except for CLP SOW:

1. All samples spiked with surrogates.
2. At least 5 percent spikes in sample matrix (MS) with selected analytes and surrogates will be analyzed (analyzed only if requested for Los Alamos project).

3. At least 5 percent duplicate spikes in sample matrix (MSD) with selected analytes and surrogates will be analyzed (analyzed only if requested for Los Alamos project).
4. At least 5 percent QC check spikes in blank matrix with selected analytes and surrogates will be analyzed.
5. At least 5 percent method blanks spiked with surrogates will be analyzed.
6. One calibration standard will be run daily. Response factors must be within 25 percent (20 percent for EPA 625) of initial calibration response factors for selected calibration check compounds. Response factors of the SPCC must be $\geq$0.05 for EPA 8270 and $\geq$0.30 for EPA 8240 (0.25 for bromoform)
7. Instrument tuning protocols will be performed and be within criteria prior to analysis.
8. Continuing calibration standard will be analyzed at a frequency of 5 percent.

### 11.1.2 GC AND HPLC MINIMUM QC

For GC-nonvolatiles, GC-volatiles, and HPLC analyses the following minimum requirements will apply, except for CLP SOW:

1. All samples spiked with surrogate, if specified by the method.
2. At least 5 percent spikes in sample matrix (MS) with selected analytes and surrogate(s) (if applicable) will be analyzed (analyzed only if requested for Los Alamos project).
3. At least 5 percent duplicate spikes in sample matrix (MSD) with selected analytes and surrogate(s) (if applicable) will be analyzed (analyzed only if requested for Los Alamos project).
4. At least 5 percent QC check spikes in blank matrix with selected analytes and surrogate (if applicable) will be analyzed.
5. At least 5 percent method blanks spiked with surrogates (if applicable) will be analyzed.
6. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.

7. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
8. Samples will be within concentration range of the standards.
9. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples and at the end of a run, and response of the control analytes must be within 15 percent of initial response for GC (25 percent for NP detector) and 10 percent of initial response for HPLC.
10. Detection limits for each parameter will be determined and checked to ensure they meet limits specified for the field group.

### 11.1.3 TRACE METALS--ATOMIC ABSORPTION, ICAP SPECTROSCOPY, AND ICAP/MASS SPECTROMETRY MINIMUM QC

For each batch of samples analyzed by AAS, ICAP or ICP/MS, the following QC checks will apply, except for CLP SOW:

1. At least 5 percent spikes in sample matrix (MS) with selected elements will be analyzed.
2. At least 5 percent duplicate spikes in sample matrix (MSD) with selected elements will be analyzed (analyzed only if requested for Los Alamos project).
3. At least 5 percent QC check spikes in blank matrix with selected elements will be analyzed.
4. At least 5 percent method blanks will be analyzed.
5. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.
6. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
7. Samples will be within concentration range of the standards (or of the ICAP instrument).

8. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples and at the end of a run, and response of the control elements must be within 20 percent (10 percent for ICAP and ICP/MS) of true value.
9. At least 5 percent filter blanks will be analyzed with all filtered samples.
10. Detection limits for each element will be determined and checked to ensure they meet limits specified for the field group.

### 11.1.4 MISCELLANEOUS METHODS MINIMUM QC

For each batch of samples analyzed by ion chromatographic, colorimetric, spectrophotometric, turbidimetric, IR, UV absorption, radiochemical, and titrimetric methods (except for pH, residues, specific conductivity, turbidity, and DO), the following QC checks will apply:

1. At least 5 percent QC check spikes in standard matrix will be analyzed.
2. At least 5 percent sample matrix spikes (MS) will be analyzed.
3. At least 5 percent duplicate control spikes in sample matrix (MSD) will be analyzed (analyzed only if requested for Los Alamos project).
4. For radiochemistry methods, at least 10 percent sample replicates will be analyzed for drinking water samples and Los Alamos project.
5. At least 5 percent method blanks will be analyzed.
6. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.
7. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
8. For radiochemistry methods, performance check standards will be analyzed and background checks performed at the required frequency.
9. Samples will be within concentration range of the standards.
10. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples (10 samples for cyanide for Los Alamos project) and at the end of

a run, and responses must be within 20 percent (10 percent for cyanide for Los Alamos project) of true value.

11. At least 5 percent filter blanks will be analyzed with all filtered samples.
12. Detection limits for analytes will be determined and checked to ensure they meet limits specified for the field group.

For each batch of samples analyzed for pH, residues, specific conductivity, turbidity, and DO, the following QC checks will apply:

1. At least 5 percent sample replicates will be analyzed.
2. At least 5 percent method blanks will be analyzed.
3. At least 5 percent filter blanks will be analyzed with all filtered samples.
4. Detection limits for analytes will be determined and checked to ensure they meet limits specified for the field group.
5. Continuing calibration standards will be analyzed at a frequency of 5 percent.

## 11.2 ROUTINE METHODS USED TO ASSESS PRECISION AND ACCURACY

### 11.2.1 PRECISION

Precision is a measure of agreement among measurements performed using the same test procedure. Precision will be assessed for applicable parameters by calculating the RPD of two duplicate spike samples as follows:

$$RPD = \frac{|\ R_1 - R_2\ |}{(R_1 + R_2)/2} x\ 100$$

where: $R_1$ and $R_2$ = concentration of Replicate Spikes 1 and 2, respectively.

This calculated RPD value is compared to the criteria specified in this LCQAP. The procedures used to determine the precision targets are listed in Table 11-2.

### 11.2.2 ACCURACY

Accuracy is the degree of agreement between a sample's target value (known concentration) and the actual measured value. Accuracy for this project is measured by calculating the percent recovery (R) of known levels of spike compounds into appropriate sample matrices. Percent recovery is calculated as follows:

$$R = \frac{100x[(SpikeSampleConc.)(Sample+SpikeVol.) - (SampleVol.)(SampleConc.)]}{(SpikeConc.)(SpikeVolume)}$$

The following equation is an example of how this would be calculated:

1 mL of spike with concentration of 100 ppb

10 mL of sample with concentration of 10 ppb

spiked sample concentration of 20 ppb

$$=100x\frac{(20)(11)-(10)(10)}{(1)(10)}=100x\frac{120}{100}=120\,percent$$

Each calculated R value is compared to the accuracy criteria listed in Section 5.0. The accuracy ranges provided in Section 5.0 are based on the mean accuracy measured or expected (based on EPA data) for each parameter plus or minus three standard deviations of the mean. The procedures used to determine the accuracy targets are listed in Table 11-2. If RPD or R values for standard spikes or sample matrix spikes within a batch do not meet acceptance criteria for QC check samples as specified in Section 5.0, results reported for samples in this batch may require flagging and/or re-analysis. The Laboratory QC Manager or designee will be notified and the necessary corrective action implemented.

### 11.2.3 CONTROL CHARTS OF ACCURACY

Control charts will be maintained for standard laboratory spike samples for FDEP, NEESA, and other specified programs. Initial control charts are prepared using historical ESE data, or are derived from published EPA method data if sufficient inhouse data are

unavailable. Control chart limits are updated yearly or more often as needed using historical data.

Control charts are graphical "pictures" that demonstrate statistical control, monitor trends in a measurement process, and diagnose a measurement problem.

The formulas used to establish and maintain control charts for standard laboratory spike QC samples are as follows:

$$USL\text{-}_x = \bar{X} + 3SD$$
$$UWL\text{-}_x = \bar{X} + 2SD$$
$$LWL\text{-}_x = \bar{X} - 2SD$$
$$LCL\text{-}_x = \bar{X} - 3SD$$

where: $X$ = mean of the recoveries of the laboratory spikes,
$SD$ = Standard deviation of the mean,
$UCL$ = Upper control limit,
$UWL$ = Upper warning limit,
$LWL$ = Lower warning limit, and
$LCL$ = Lower control limit.

All recoveries will be plotted on the appropriate matrix-specific control charts.

An out-of-control situation for accuracy control charts may be indicated by the following:

1. Any one point plots outside the control limits.
2. Any eight consecutive points plot on the same side of the mean.
3. Any six consecutive points trend in the same direction.
4. A cyclical pattern is evident.
5. Any three consecutive points plot within the control limits but outside the warning limits.

The occurrence of any of these events will be investigated; corrective actions will be taken as required to return the system to a state of statistical control. All corrective actions will be documented.

Table 11-2. Methods Used to Generate Precision and Accuracy Targets

| Method | Purpose | Concentration Level | Method References |
|---|---|---|---|
| Sample Duplicate | Precision | NA | STMD2216, ASTMD 32974, 110.2, 1110, SM 2330,360.1, 150.1, 9040, 9045, 160.1, 160.1, 160.3, 900.0, 903.1, 904, 905, 908 |
| Standard Matrix Spike (QC Check Standard) | Accuracy | Low Level | 7481, 270.2, 7740, 286.2, 7911, 8240 |
| | | Mid Level | 200.7, 202, 204.2, 206.2, 208.2, 210.2, 213.2, 218.1, 219.2, 220.1, 239.2, 243.2, 245.1, 249.2, 258.1, 272.2, 273.1, 279.2, 283.2, 286.2, 6010, 7020, 7041, 7060, 7091, 7131, 7191, 7201, 7200, 7210, 7421, 7460, 7470, 7471, 7480, 7610, 7770, 7841, 310.0, HACH 8000, 335.3, 9010, 300, 325.3 353.2, 9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1, 370.1, 375.4, 376.2, 9030, 305.1, 405.1, 9060, 330.1, 7196, 340.2, 130.2, 413.1, 9071, 9073, 420.1, 9066, 425.1, 418.1, 9071, 9073, 9020, 900.0, 9310, 9315, 904.0, 905.0, 908.0,501.2, 504, 505, 507, 515.1, 524.2, 531.1, 601, 8010, 602, 8020, 604, 8040, 605, 606, 8060, 608, 617, 8080,610, 8310, 615, 8150, 622, 614, 8140, 624, 8240, 8260, 625, 8270, 632, 612, 8120, 619, 633, 645, 8330, UW32, LW12, 547, 630.1 (Mod) |
| | | High Level | 903.0, 9320 |
| Sample Matrix Spike | Accuracy | Low Level | 7481, 270.2, 7740, 286.2, 7911, 8240 |
| | | Mid Level | 200.7, 202, 204.2, 206.2, 208.2, 210.2, 213.2, 218.1, 219.2, 220.1, 239.2, 243.2, 245.1, 249.2, 258.1, 272.2, 273.1, 279.2, 283.2, 286.2, 6010, 7020, 7041, 7060, 7091, 7131, 7191, 7201,7200, 7210, 7421, 7460, 7470, 7471, 7480, 7610, 7770, 7841, 310.0, HACH 8000, 335.3, 9010, 300, 325.3, 353.2, 9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1, 370.1, 375.4, 376.2, 9030, 305.1, 405.1, 9060, 330.1, 7196, 340.2, |

Table 11-2. Methods Used to Generate Precision and Accuracy Targets (Continued, Page 2 of 2)

| Method | Purpose | Concentration Level | Method References |
|---|---|---|---|
| | | | 130.2, 413.1, 9071, 9073, 420.1, 9066, 425.1, 418.1, 9071, 9073, 9020, 900.0, 9310, 9315, 904.0, 905.0, 908.0,501.2, 504, 505, 507, 515.1, 524.2, 531.1, 601, 8010, 602, 8020, 604, 8040, 605, 606, 8060, 608, 617, 8080,610, 8310, 615, 8150, 622, 614, 8140, 624, 8240, 8260, 625, 8270, 632, 612, 8120, 619, 633, 645, 8330, UW32, LW12, 547, 630.1 (Mod) |
| | | High Level | 903.0, 9320 |
| Sample Matrix Spike Duplicate | Precision | Low Level | 7481, 270.2, 7740, 286.2, 7911, 8240 |
| | | Mid Level | 200.7, 202, 204.2, 206.2, 208.2, 210.2,213.2, 218.1, 219.2, 220.1, 239.2, 243.2,245.1, 249.2, 258.1, 272.2, 273.1, 279.2,283.2, 286.2, 6010, 7020, 7041, 7060, 7091,7131, 7191, 7201, 7200, 7210, 7421, 7460,7470, 7471, 7480, 7610, 7770, 7841, 310.0,HACH 8000, 335.3, 9010, 300, 325.3 353.2,9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1,370.1, 375.4, 376.2, 9030, 305.1, 405.1,9060, 330.1, 7196, 340.2, 130.2, 413.1,9071, 9073, 420.1, 9066, 425.1, 418.1,9071, 9073, 9020, 900.0, 9310, 9315, 904.0,905.0, 908.0,501.2, 504, 505, 507, 515.1,524.2, 531.1, 601, 8010, 602, 8020, 604,8040, 605, 606, 8060, 608, 617, 8080,610,8310, 615, 8150, 622, 614, 8140, 624, 8240,8260, 625, 8270, 632, 612, 8120, 619, 633,645, 8330, UW32, LW12, 547, 630.1 (Mod) |
| | | High Level | 903.0, 9320 |

Note: Low Level = Concentration from the reporting limit to 5 times the detection limit.
Mid Level = The mean level between the reporting limit and the upper end of the linear range.
High Level = Concentration at the upper end of the linear range.

## 11.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

### 11.3.1 METHOD DETECTION LIMITS (MDLS)

The detection limit of the method is the lowest sample concentration which can be reliably recovered and measured in the sample matrix with a low background level. To determine absolute MDL, statistically based procedures are available from EPA methods. The MDL studies will be performed annually.

The detection limit is defined as follows for all measurements:

$$MDL = t_{(n-1,\ 1-\alpha,\ =\ 0.99)} \times S$$

where:

MDL = method detection limit,

S = standard deviation of the replicate analyses, and

$t_{(n-1,\ 1-\alpha,\ =\ 0.99)}$ = Students t-value appropriate to a 99-percent confidence level and a standard deviation estimate with n-1 degrees of freedom.

The reporting limits in Section 5.0 are derived from MDLs.

### 11.3.2 PRACTICAL QUANTITATION LIMIT (PQL)

The PQL is defined as 12 times the standard deviation that is derived from the procedures used to determine MDL.

## 11.4 COMPLETENESS

Completeness is not an FDER requirement but is an ESE-required objective.

Completeness is defined by EPA as "a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions" (EPA, 1980). A completeness of at least 90 percent for each

parameter is the objective for this project. Following completion of the analytical testing, percent completeness will be calculated as follows:

$$\text{Completeness (\%) for parameter y} = \frac{\text{\# of valid y values reported}}{\text{\# of samples collected for analysis of y}} \times 100$$

If completeness is less than 90 percent for any parameter(s), the Project Manager will be notified immediately. The Project Manager is responsible for determining if resampling will be necessary to meet project objectives and will inform the Project QA Officer and Laboratory Coordinator of the decision.

# 12.0 DATA REDUCTION, VALIDATION, AND REPORTING

## 12.1 DATA REDUCTION

Data transfer and reduction are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and, in the case of data reduction, that accepted statistical techniques are used. ESE will use its in-house-developed CLASS™ for data management.

If applicable, example calculations must be included with the analytical method to facilitate review. The entry of input data and calculations should be checked and the signature/initials of the analyst or individual entering the data and reviewer(s) should accompany all data transfers with and without reduction.

For routine analyses performed at the Gainesville Laboratory, sample response data will be entered into CLASS™ by the analyst or other designated individual(s). The computer calculates the following:

1. Linear, quadratic, or logarithmic regression line for standards,
2. Coefficients of variation for replicates,
3. Spiked recoveries,
4. Reference sample concentrations, and
5. Sample concentrations.

Linear or quadratic equations will be used to calculate final data for laboratory analyses requiring a calibration curve:

$$\text{Concentration} = \text{Intercept} + M\,(\text{Response}) + M2\,(\text{Response})^2$$

The equation used to calculate final data is dependent on the linearity of the standard curve and method of analysis.

Purgeable organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_{sa})(Q_{is})}{(RF)(A_{is})(PV)}$$

where: $A_{sa}$ = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample,

$Q_{is}$ = quantity of the internal standard [nanograms (ng)],

RF = response factor (see Section 8.0),

$A_{is}$ = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample, and

PV = purge volume (mL).

Semivolatile organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_{sa})(Q_{is})}{(A_{is})\,(RF)} \times \frac{1}{FE} \times \frac{1}{\text{volume}} \times DF$$

where: $A_{sa}$ = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample;

$A_{is}$ = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample;

$Q_{is}$ = quantity of the internal standard (ng);

RF = response factor (see Section 8.0);

$$FE = \text{fraction extract analyzed} = \frac{\text{Volume injected } (\mu L)}{\text{extract volume } (\mu L);}$$

volume = volume of extracted sample (mL); and

$$DF = \text{dilution factor} = \frac{\text{final extract volume for injection (mL)}}{\text{extract volume prior to dilution (mL).}}$$

The final data for GC/MS semivolatiles and volatiles analyses are calculated by the computer data acquisition system attached to each mass spectrometer.

QC acceptance criteria (Section 5.0) for the relative percent difference of replicate matrix spike recoveries and for the range of acceptable recoveries are electronically stored for each STORET number/method code combination. If the samples in a batch (sample lot) do not pass all the QC checks (Section 11.0), then the results reported in all samples processed in the same sample set may be considered as suspect and the analyses may need to be repeated.

Completed batch folders are stored in a secured central location arranged by departments and numerically by batch number. Strip charts, chromatograms, copies of parameter notebooks, and all other pertinent raw data and other documentation will be stored in the batch folders.

Once the data set is complete for each sampling effort, the Laboratory Coordinator organizes the information in final reports appropriate to project requirements. This Laboratory Coordinator is responsible for final QC review and release of the data.

### 12.1.1 THE DOCUMENTATION RECORDS

#### 12.1.1.1 GC/HPLC

Prior to analysis, the analyst must obtain a file folder and all applicable logsheets and data sheets.

Extraction Logsheet--An extraction logsheet (Figure 7-14), filled out by the analyst performing the sample extraction, will accompany each lot of samples throughout analysis. This sheet will include at least the following data:

1. Project name and number,
2. Extractor's initials,
3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analyte group [e.g., pentachlorophenol (PCP), PAHs, OCPs],
8. Initial volume or wet weight of sample extracted,
9. Initial/final pH (water sample),
10. Extracting solvent,
11. Final volume/solvent,
12. Lot number(s) of solvent(s) used,
13. Date of cleanup (if required),
14. Notes and comments affecting the extraction procedure, and
15. Appearance of each sample.

After extraction is complete, extraction logsheets will be filed in the batch folder and accompany the extracted samples to the instrumental analyst. Each extract vial will be properly labeled and include the following information:

1. Project name,
2. Field group name,

3. Sample number,
4. Extraction concentration factor,
5. Date extracted, and
6. Extractor's initials.

Instrument Logbooks--During analysis, the following information will be recorded in the instrument notebook:

1. A log of the types of analyses run on the instrument, to include:
   a. Column conditions and temperature zones,
   b. Sample numbers or other identification of samples,
   c. Reference to a method describing the analysis,
   d. Analysis date,
   e. Detector used [e.g., flame ionization detector (FID)], and
   f. Detector conditions.
2. Service records, which are kept in a separate maintenance log.

Chromatograms--At the time of analysis, the analyst will include on the chromatogram the following information:

1. Date and time of analysis,
2. Analyst's initials,
3. Instrument used,
4. Field group name,
5. Sample number and other identification for each chromatogram, and
6. Concentration/dilution factor for each sample.

The chromatograms, extraction logsheet, and copies of instrument logbooks will be placed in the batch file folder.

Chromatographic Logsheets--For each analysis, the analyst will record all pertinent information on a standard curve data sheet and chromatographic data logsheet. The standard curve data sheet lists the standards, their concentrations, and the respective responses. The chromatographic data sheet lists the samples in order of injection and the factors needed for calculating the concentrations. A sample calculation using calculated response factors will appear on the back of the chromatographic data sheet if responses are calculated manually.

After the analysis and data reduction are complete, the chromatograms and worksheets will be stored in the batch file folder and the data entered into CLASS™. The folder will be turned in to Laboratory Information Services for processing and storage in the secured central filing location.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the method will be prepared. Procedures used in preparing the standards will be recorded in the standards preparation notebook. The following information must be recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight measured,
6. Volumetric flask volume,
7. Calculated concentration,
8. Solvent name and lot number,
9. Dilutions, and
10. Expiration date.

Immediately after an analytical standard has been prepared, the standard will be transferred to an amber glass vial or bottle and properly labeled. Standards should be refrigerated when not in immediate use.

12.1.1.2 GC/MS

Prior to analysis, the extracting analyst must obtain a batch file folder and all applicable data sheets and logsheets.

Extraction Logsheet--Once a batch has been established, the sample extraction and analysis procedure begins. A GC/MS extraction logsheet (Figure 7-14), filled out by the analyst performing the sample extraction, will accompany the batch throughout analysis. This sheet will include at least the following data:

1. Project name and number,
2. Analyst's initials,
3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analyte group (i.e., acids, base/neutrals),
8. Initial volume or wet weight of sample extracted,
9. Initial/final pH,
10. Extract solvent,
11. Final volume/solvent,
12. Lot number(s) of solvent(s) used,
13. Date of cleanup, and
14. Notes and comments affecting the extraction procedure.

After extraction, extraction logsheets will be filed in the batch file folder and accompany the extracted samples to the instrument analyst. The extract vial will be properly labeled. The label will contain the following information:

1. Project name,
2. Field group,
3. Sample number,
4. Extraction concentration factor and solvent used,
5. Date extracted, and
6. Extractor's initials.

Sample Screening--Sample extracts may be screened by GC employing flame ionization detection (GC/FID) prior to GC/MS analysis to permit dilution of extracts (as required) to concentration levels compatible with the GC/MS instrument and column capabilities.

Spectral Data and GC/MS Computer Quantitation Report--The quantitative sample and standard data generated by the GC/MS data system and all mass spectral information will be labeled according to EPA-CLP 2/88 SOW and placed in the batch file folder. Manual data reduction sheets also will be placed in this folder.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the instrument are prepared. Procedures used in preparing the standards must be recorded in the preparer's laboratory notebook. The following information will be recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight (or volume) measured,
6. Volumetric flask volume,

7. Calculated concentration,
8. Solvent name and lot number, and
9. Dilutions.

The analytical standard will be transferred immediately to a properly labeled glass amber bottle or vial after preparation. Standards should be refrigerated when not in use.

GC/MS Instrument Logbooks--Whenever the GC/MS is used for sample analysis, the following information will be recorded in an instrument logbook:

1. Instrument conditions of the gas chromatograph,
2. Instrument conditions of the mass spectrometer,
3. Analyst's initials,
4. Date of analysis,
5. Sample number,
6. Dilution factor, and
7. Frame reference number (FRN).

Compound Identification--Compound identification will be made in terms of the full-scan mass spectrum obtained in the electron impact mode at 70 electronvolts (eV). Compound identification will require the presence of all significant major ions at the appropriate relative abundance as obtained with an authentic compound or reference spectrum from a reputable literature source. The selection of significant ions is strongly compound dependent, and because of this and other considerations, the identification of compounds will entail considerable professional judgment and experience.

The most convincing evidence for compound identification is comparison of spectrum with that of an authentic compound obtained under identical operation conditions. When this is not possible due to compound availability, computer identification or manual library search will be used.

When no tentative matches are found in the library, identification will be based on application of known fragmentation patterns, empirical correlations, and isotope abundance data. All data reported as a result of library searches will be reported as tentatively identified compounds (TICs).

Compound Quantification--The technique of extracted ion current profiles will be employed for the preliminary qualitative searching and for quantification of individual compounds. Appropriate internal standards will be employed to permit quantification in terms of the relative response to these internal standards. Concentration calculations and data reduction procedures are given in Section 10.1.

Spiking with Internal Standards--All samples will be spiked with quantitation standards just prior to the GC/MS analysis (Section 11.2). Appropriate internal standards will be selected for the remaining categories.

GC/MS Instrumental Detection Limits--The instrumental detection limit refers to the least quantity of material required to provide a total mass spectrum of sufficient quantity to permit compound identification. The mass spectrum must contain all major ions with the appropriate relative abundance within 20 percent of either an authentic compound analyzed under identical conditions or an appropriate reference spectrum from the literature.

Data Management--Output from the gas chromatography/mass spectrometry/ data system (GC/MS/DS) is variable, depending on the project. However, all raw data such as mass chromatograms will be stored on magnetic tape. The final results are transmitted to CLASS™ by project and sample number. Quantification reports present the calculation results. The FRN is obtained from the quantification reports. All magnetic tapes are kept in sequential order with respect to the FRN. By following this sequence, it is possible to obtain all raw data for a particular sample number. The GC/MS computer

generates a data file that is transmitted to CLASS™. Laboratory Information Services personnel process the transmitted data and generate a batch report. The batch is returned to the analyst for review. The batch folder, containing the quantification report, batch report, copies of logsheets, and other pertinent raw data is turned into Laboratory Information Services for processing and storage in the secured central filing location.

**12.1.1.3 Trace Metals**

Prior to analysis, the analyst must obtain a file folder and all applicable logsheets and data sheets.

Digestion or Sample Preparation Logsheet--A digestion or sample preparation logsheet, filled out by the analyst performing the sample digestion or sample preparation, will accompany each lot of samples throughout the analysis. This logsheet will include the following data:

1. Method used (GFAA, CVAA, ICAP, ICP/MS)
2. Analyst's initials,
3. Date sample digested,
4. Initial volume or weight,
5. Final volume,
6. Spiking solution used and date spiking solution prepared,
7. Field Group,
8. Sample numbers, and
9. Notes or comments affecting the digestion procedure.

Strip Charts--At the time of analysis (currently only applicable to mercury by cold vapor), the following information will be recorded on the strip chart:

1. Analyst's name, initials, or employee number;
2. Date of analysis;
3. Instrument/method used;

4. Element of interest;
5. Instrument conditions;
6. Sample matrix; and
7. Comments.

During analysis, the analyst will indicate on the strip chart sample numbers, QC samples, blanks, and standards.

After the data have been reduced and recorded in the instrument notebook, the strip charts are placed in a batch file folder together with the copies of the digestion logsheet, copies of the instrument logbook, and reduction sheets. These data are entered manually or automatically uploaded to CLASS™ to generate a uniquely numbered batch. The analyst reviews the data and validates the correct transcription of data into CLASS™. Then, the batch is signed and submitted to Laboratory Information Services to be stored in the secured central filing system.

For ICAP, ICP/MS and GFAA, the instrument computers produce data files that are evaluated and transmitted to CLASS™. The analyst then generates a batch for review. The batch folder containing the batch report, the data file, copies of logsheets, and all other pertinent raw data are turned in to Laboratory Information Services for processing and storage in the secured central filing location.

Laboratory Notebooks--Each instrument will have its own laboratory notebook. After each analysis, the analyst will record in the notebook the following information:

1. Problems encountered during the digestion/analysis,
2. Comments about the samples and/or analytical procedure,
3. Instrument used,
4. Method used (GFAA, CVAA, ICAP, ICP/MS),
5. Date of analysis,

6. Analyst(s),
7. Element,
8. Sample matrix,
9. Instrument conditions,
10. Field group,
11. Sample numbers,
12. QC data, and
13. Raw data.

Standards--Stock standard solutions are purchased from vendors. These stock solutions are certified by the vendor for purity and concentration.

Standard preparations are recorded in a logbook. The information recorded includes preparer's name, lot number, date of preparation, volumes used, calculated concentrations, and dilutions.

Volumetric dilutions are made from the stock solution to obtain working solutions. Serial dilutions are then made from the working solutions to obtain working standards to be used to generate standard curves. Working standard solutions are stored in volumetric flasks and properly labeled with the following information:

1. Preparer's name or initials,
2. Date of preparation,
3. Element(s),
4. Concentration, and
5. Expiration date (if not prepared daily).

### 12.1.1.4 Inorganics

Raw data for most inorganic analyses is documented through the use of parameter notebooks. The notebooks may vary slightly in format dependent upon the type of analysis, but, at a minimum will contain the following:

1. Analysis date,
2. Parameter,
3. STORET and method code,
4. Standard curve range and responses (where applicable),
5. Analytical batch number,
6. Instrument conditions (where applicable),
7. Method reference,
8. Sample, standard, QC sample and blank identification and responses or concentration as applicable, and
9. Analyst's signature.

Raw data for specialized instrumental analyses are documented in the following sections.

**Inorganic Analysis by Autoanalyzer**

Strip Charts--The following information will be recorded on the strip chart:

1. Analyst's name, initials, or employee number,
2. Date of analysis,
3. Instrument used,
4. Analytical parameter,
5. Analytical batch number,
6. Standard calibration setting,
7. Sample, standard, QC sample, and blank sample identification above appropriate peaks, with dilution factors when applicable.

After the data have been reduced and recorded in the parameter notebook, the strip charts are placed in a batch file folder with copies of the notebook pages and any additional related information. These data are entered manually or are electronically uploaded to CLASS™ to generate a uniquely numbered batch. The batch is reviewed for correctness and signed by the analyst and submitted for peer review. When peer review is complete, the reviewer signs and submits the batch to Laboratory Information Services to be finalized and stored in the secured central filing location.

Laboratory Notebooks--Each analytical parameter has its own laboratory notebook. During analysis, the following information is recorded:

1. Date of analysis,
2. Parameter,
3. STORET and method code,
4. Batch number,
5. Instrument conditions,
6. Calibration standard setting and response,
7. Standard curve range and date of preparation,
8. Sample and QC identification numbers, and
9. Analyst's signature.

**Inorganic Analysis by Ion Chromatography**

Chromatograms--All information on the chromatograms from each analytical run is electronically recorded from the input provided during run set up. This information includes the following:

1. Analyst's initials;
2. Analytes;
3. Analysis date and time;
4. Instrument identification;
5. Integration parameters;

6. Sample, standard, and QC sample identification with concentrations and responses; and
7. Dilution factors when appropriate.

These data are electronically uploaded to CLASS™ and a unique batch number is assigned. The data are reviewed by the analyst for correctness, signed and submitted for peer review. When peer review is complete, the reviewer signs and submits the batch to Laboratory Information Services to be finalized and stored in the secured central filing location.

Laboratory Notebooks-Each instrument has its own laboratory notebook. The following information is recorded in the notebook during the set up of the analytical run:

1. Analysis date,
2. Analyte,
3. STORET and method code,
4. Instrument identification and operating conditions,
5. Calibration standards and preparation dates,
6. Notes and comments as appropriate, and
7. Sample and QC sample identification numbers with dilution factors when applicable.

**12.1.1.5 Radiochemistry**

Instrument Logbooks--Each instrument will have its own laboratory notebook. After each analysis, the analyst will record in the notebook the following information:

1. Type of analysis being performed,
2. Name of person(s) doing the analysis,
3. Sample names and numbers,
4. Notes of GM surveys performed on samples prior to counting,
5. Background information on samples prior to counting,
5. Background information on each detector,

6. Analysis date,
7. Documentation concerning sample analysis (Did samples appear to have significant count rate?),
8. Physical appearance of samples,
9. Flow rate of gases, and
10. Detector conditions.

Service records are maintained in a separate logbook and contain all information pertinent to calibration, cleaning, and repair of instrumentation.

## 12.2 DATA VALIDATION

Unless otherwise specified by the client, the following procedures for review/validation of data are employed.

### 12.2.1 LABORATORY ACTIVITIES

Data review is initiated by the bench analyst upon conversion of raw data into reportable data. The bench analyst reviews preliminary data entries, calculations, holding times and precision, accuracy, and calibration checks. The analyst provides explanation and/or corrective action for any method control parameters which are outside criteria and signs the analytical batch when ready to release the data for further processing and review.

The analyst's supervisor or a designated reviewer also reviews the analytical batch documentation associated with the batch (such as sample preparation/ digestion/extraction logsheets, instrument logsheets, copy of sample preparation, etc.) and any explanations or corrective actions provided by the analyst. If the supervisor or designee is not satisfied with the explanations or corrective actions, an additional explanation or corrective action is provided in the batch. The supervisor or designee signs the analytical batch when satisfied with the data.

The Laboratory Coordinator reviews analytical data batches that have explanations and corrective actions and signs the analytical batch when satisfied with the data. The Laboratory Coordinator also reviews all final data reports for inconsistencies and completeness prior to releasing the reports to the client; qualification or flagging, if needed, of data and/or QC summaries are provided as appropriate.

The Laboratory QA/QC Coordinator performs quarterly audits to check that required QC procedures are being followed. This procedure entails random review of analytical batches to see that the QC designated for the analysis are being consistently performed. A record of this audit is maintained by the Laboratory QA/QC Coordinator. The Laboratory QA/QC Coordinator also initiates and follows up on corrective actions to resolve QC problems.

The minimum QA/QC data that should be included in the data batch are the following:

1. Sample data (matrix, date of extraction, and date of analysis);
2. Parameter, result, and test method identification;
3. Sample-specific detection limits for each parameter; and
4. Results of laboratory control data, method blanks, spikes, and replicates (as required).

## 12.3 DATA REPORTING

Data reporting is accomplished by the Laboratory Coordinator using CLASS™. The data flow scheme for CLASS™ is presented in Figure 12-1. All client data and pertinent field information are entered into CLASS™ directly from the chain of custody sheets. A copy of this information is given to the Laboratory Coordinators for verification to ensure that all pertinent information is available and correct. An example of a Results of Analysis Report is shown in Figure 12-2. CLASS™ sorts all available samples for analyses for each parameter by due date, client ID, field group, etc. Daily reports are generated by

Laboratory Information Services and sent to each analytical department to notify them of samples that are due for analysis.

Each analyst who enters their analytical information directly into CLASS™ as a batch report. The analysts enter standard curves (linear, quadratic, or logarithmic), method blank and control spike data into CLASS™ to create a batch. Sample responses are entered into the batch and the final results are then calculated according to the methods specified in Section 7.0 of the CompQAP and Section 12.1. The analysts check all their data to ensure that all information is available and correct before signing the batch report. The analyst's supervisor or Department Manager then reviews the final batch report and signs it to verify that all data are accurate as reported. The batch is then finalized by Laboratory Information Services. Once a batch is finalized, the analyst or analyst's supervisor cannot change the data. Any requests for corrections are sent to Laboratory Information Services where the changes are made. The Laboratory Coordinator generates and prepares from CLASS™ the final report to the client. The Laboratory Coordinator reviews the final reports for inconsistencies and completeness. Deliverables will comply with NEESA/HAZWRAP or project specific requirements for the DQOL (Data Quality Objective Level) specified for each project. Prior to the release to the client the final report is peer reviewed using the checklist in Figure 12-3.

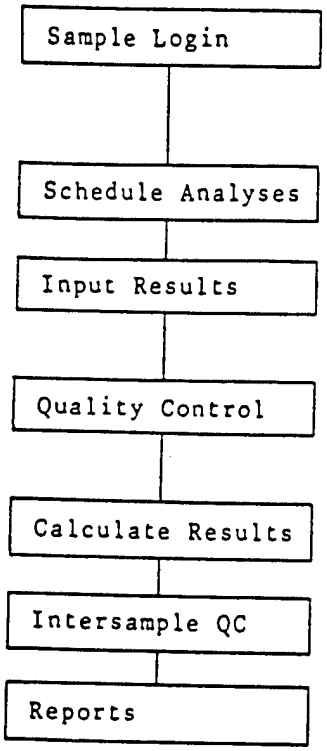
## 12.4 DATA STORAGE

A hard copy of all batch folders, supporting documents, and project files are filed chronologically by department in the secured centralized batch storage located in a separate building. The newer batch folders are also stored chronologically by department in locked file cabinets located in Information Services Department. The batch folders include copies of sample preparation/digestion/extraction logsheets, copies of instrument logsheets and standard preparation logbook pages, laboratory chain of custody, and raw data. The batch folders may be checked out for review by laboratory analysts, Laboratory Coordinators, or laboratory personnel. A program for tracking folder status and custody is available in CLASS™. This program is used to track folders that have

been checked out. In addition, any personnel checking out a batch folder from Laboratory Information Services is required to sign, date, indicate the batch numbers, and department numbers on the Document Control Logbook (Figure 12-4). When the laboratory or QA personnel are finished reviewing the batch folders, they are returned to Laboratory Information Services and the Document Control Logbook is signed and dated. At a minimum, all project files are kept for 5 years.

The original laboratory notebooks and analysts notebooks are used until they are filled, then sequentially numbered and archived by the supervisor within each department.

All data stored in the CLASS™ database are backed up every day except Saturday using electronic optical disks or equivalent high-density storage media. Disks are stored in special files and archived in a separate building in a secured air-conditioned location.




--activate samples
--set permanent station codes
--store collection time and date
--store field data

--list of samples available for each parameter sorted by due date

--reserve analysis
--input calibration, quality control and sample data

--calculate calibration curve, spike recovery, replicate and reference sample quality control

--calculate and store final sample concentrations

--check for data inconsistencies
--perform interparameter calculations

--produce reports of sample data, quality control, and statistical analyses

**Figure 12-1**
**FLOWCHART OF THE CLASS PROGRAM**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

Environmental Science and Engineering DATE 3/01/91 STATUS: FINAL

PROJECT NUMBER: 99999 0000 PROJECT NAME: EXAMPLE PROJECT
FIELD GROUP: XXXXXXX LAB COORDINATOR: ALAN CHEMY

RESULTS OF ANALYSIS

| PARAMETERS / UNITS | STORET METHOD | MW5 XXXXXX 1 | MW6 XXXXXX 2 | MW7 XXXXXX 3 | MW10 XXXXXX 4 |
|---|---|---|---|---|---|
| DATE / TIME | | 04/13/90 11:15 | 04/13/90 13:00 | 04/12/90 15:00 | 04/13/90 14:00 |
| LEAD,TOTAL UG/L | 1051 GFAA | 16.1 | 79.9 | 22.8 | 13.9 |
| 1,2-DIBROMOETHANE (EDB) UG/L | 77651 EC | <0.013 | 0.078 | <0.013 | <0.013 |
| CARBON TETRACHLORIDE UG/L | 32102 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| CHLOROBENZENE UG/L | 34301 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| CHLOROETHANE UG/L | 34311 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| CHLOROFORM UG/L | 32106 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| CHLOROMETHANE UG/L | 34418 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,1-DICHLOROETHANE UG/L | 34496 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,2-DICHLOROETHANE UG/L | 34531 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,1-DICHLOROETHYLENE UG/L | 34501 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| TRANS-1,2-DICHLORO ETHENE UG/L | 34546 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| METHYLENE CHLORIDE UG/L | 34423 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,1,2,2-TETRACHLORO ETHANE UG/L | 34516 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| TETRACHLOROETHENE UG/L | 34475 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,1,1-TRICHL'ETHANE UG/L | 34506 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| 1,1,2-TRICHL'ETHANE UG/L | 34511 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| TRICHLOROETHENE UG/L | 39180 HA | <1.00 | <1.00 | <1.00 | <1.00 |
| BENZENE UG/L | 34030 PI | <1.00 | 25.0 | <1.00 | <1.00 |
| ETHYLBENZENE UG/L | 34371 PI | <1.00 | <1.00 | <1.00 | <1.00 |
| TOLUENE UG/L | 34010 PI | <1.00 | <1.00 | <1.00 | <1.00 |
| XYLENES,TOTAL UG/L | 81551 PI | <1.00 | 150 | <1.00 | <1.00 |
| METHYL-T-BUT'ETHER UG/L | 98676 PI | 2.90 | 5.50 | <1.00 | 5.42 |
| VOA,TOTAL(BTEX,T) UG/L | 97512 PI | <1.00 | 175 | <1.00 | <1.00 |

**Figure 12-2**
**FINAL RESULTS OUTPUT FROM THE DATA PROGRAM**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

# ESE ANALYTICAL SERVICES DELIVERABLE CHECKLIST

Project #_____ Project Name_____

Field Group(s)_____

| | | | | Comment |
|---|---|---|---|---|
| Department's "excepted" batch checklist(s) reviewed? | Y | N | | |
| Corrective actions*/data flagging required? | Y | N | | |
| If yes, performed? | Y | N | | |
| Blank data (equipment, rinseate, field, trip) reviewed? | Y | N | NA | |
| Corrective actions*/data flagging required? | Y | N | | |
| If yes, performed? | Y | N | | |
| Field dupe data reviewed? | Y | N | NA | |
| Corrective actions*/data flagging required? | Y | N | | |
| If yes, performed? | Y | N | | |
| Data set reviewed against historical? | Y | N | NA | |
| Data set reviewed for reasonableness? | Y | N | | |
| If yes, by ____ self, or _____ | | | | |
| In general, were project QC requirements met? | Y | N | | |
| If no, add comments below. | | | | |
| QC deliverable prepared and reviewed? | Y | N | NA | |
| Deliverables in conformance with requirements? | Y | N | | |

* Attach a copy of any corrective action

Comments:

Completed by_____ Reviewed by_____

Date_____ Date_____

Distribution:

WHITE - Information Services YELLOW - Laboratory Coordinator PINK - Div. Administration

**Figure 12-3**
**DELIVERABLE CHECKLIST**

SOURCE: ESE.

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.**

## DOCUMENT CONTROL

PLEASE USE SEPARATE LINE FOR EACH BATCH CHECKED-OUT!

| INITIALS | DATE/TIME OUT | DOCUMENT TYPE (e.g. Batch #, F.G. Files, etc...) | DOCUMENT DEPT # | DATE/TIME RETURN | INITIALS | ✓ |
|---|---|---|---|---|---|---|
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(CONTROL 06/92)

Figure 12-4
DOCUMENT CONTROL LOGSHEET

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

# 13.0 CORRECTIVE ACTION

Corrective action is necessary when any measurement system fails to follow this LCQAP. Items that may need corrective action range from a minor problem of a field team member failing to sign a field form to a major problem of an analyst using an improper analytical method. For this reason, corrective action protocols must be flexible.

## 13.1 ANALYTICAL

In general, items needing corrective action fall into three "correction" categories: short-term, long-term, and QC; each item requires different action.

### 13.1.1 SHORT-TERM CORRECTIVE ACTIONS

These actions consist of minor and major problems that can be corrected immediately. Examples include failure to date or sign a standard form, incorrectly preserving sample, and errors in data entry. Corrective action is initiated by verbally calling attention to the problem followed by written notification.

### 13.1.2 LONG-TERM CORRECTIVE ACTIONS

The actions consist of minor and major problems that require a series of actions to resolve the problem. The actions to be taken are coordinated by the Laboratory QA/QC Manager or his designee, and a QA corrective action and routing form (Figure 13-1) is used to track the action. An example of this type of corrective action is as follows:

> Problem--A laboratory analyst fails to calibrate a pH meter prior to use.
>
> Corrective Action--The problem is identified by the person originating the corrective action, responsibility is assigned to an appropriate person (may be someone other than person failing to calibrate the instrument), re-training of the analysts in the use of the instrument is required, and the instrument is calibrated in prior to the next analysis. The QA/QC Coordinator audits this process to assure that it is completed in an expeditious manner.

# CORRECTIVE ACTION REQUEST AND ROUTING FORM

1. Identification of a problem: CA#_____

Originator: _____ Date: _____

Nature of Problem: _____

2. Determination of Required Action:

Responsibility Assigned to: _____ Due Date: _____

Recommended Action: _____

3. Implementation of Required Action:

Responsibility Assigned to: _____ Due Date: _____

4. Assuring Effectiveness of Action:

Responsibility Assigned to: _____ Due Date: _____

Procedure to Assure Effectiveness: _____

Corrective action status: _____ Acceptable _____ Unacceptable

Signature: _____ Date _____

**Figure 13-1**
**QUALITY ASSURANCE CORRECTIVE ACTION REQUEST AND ROUTING FORM**

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

### 13.1.3 QUALITY CONTROL CORRECTIVE ACTION

Consists of corrective action following a failure to meet QC criteria specified in this LCQAP and the analytical methods. Actions taken consist of two types: those resolved within each analytical department and those resolved outside the department. Examples outlining the differences between these two types of corrective action are as follows:

**WITHIN DEPARTMENT ACTION**

| QC Failure | Department Action |
|---|---|
| Tuning results for GC/MS fail criteria in Methods 624 and 625 | Analyst retunes instrument |
| Standard curve correlation coefficient is less than 0.995 | Analyst investigates problem and reruns curve and samples |
| Sample response falls outside calibration curve | Analyst dilutes sample into range of curve |

**OUTSIDE DEPARTMENT ACTION**

| QC Failure | Department Action |
|---|---|
| Holding times are exceeded | Notify Project Manager, Laboratory Coordinator, and Project QA Coordinator; resampling may be necessary |

The corrective action procedures that will be taken by the Gainesville Laboratory following a failure to meet QC criteria specified in this LCQAP and the analytical methods are summarized in Tables 13-1 through 13-6.

Corrective actions in the laboratory are documented and tracked using the Corrective Action Form (Figure 13-1).

Corrective actions may be initiated for each measurement system (individual disciplines) by subproject managers or other responsible individuals such as the Laboratory QA/QC Manager, Department Manager, or Division Manager. The Laboratory QA/QC Manager,

Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Initial calibration verification standard (ICV) | +/- 10% of true value | Rerun standard, if still out of control, recalibrate instrument. |
| Calibration blank (ICB) out | $\leq$ two times DL (listed in Table 5-3) | Rerun the blank, if still of control, reprocess and reanalyze the blank. |
| Calibration curve correlation coefficient | $\geq$ 0.995 | Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument or document why data are acceptable. |
| Calibration curve | Brackets all sample responses | Dilute and reanalyze within the calibration curve range or document why data are acceptable if reanalysis is not possible. |
| Continuing calibration verification standard (CCV) | +/- 20% of true value | Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV. |
| Method blank (MB) | $\leq$ two times DL (listed in Table 5-3) | Determine the cause of the blank problem, redigest set, if necessary, or document why data are acceptable. |

Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy (Continued, Page 2 of 2)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Standard matrix spike (QC check standard) | See Table 5-2 for percent recovery control limits | Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike | See Table 5-2 for percent recovery control limits | If standard matrix spike analytes are within control limits, qualify the data. If not, determine and correct the problem, redigest and reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike | See Table 5-2 for RPD control limits | If standard matrix spike analytes are within control limits, qualify the data. If not, determine and correct the problem, redigest and reanalyze samples, if necessary, or document why data are acceptable. |

Note: DL = detection limit.
RPD = relative percent difference.

Source: ESE.

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy and Inductively Coupled Plasma/Mass Spectrometry

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Initial calibration verification standard (ICV) | +/- 10% of true value | Rerun standard, if still out of control, recalibrate instrument. |
| Calibration blank (ICB) | $\leq$ two times DL (listed in Table 5-2) | Rerun the blank, if still out of control, reprocess and reanalyze the blank. |
| Interference check standard (ICS) | +/- 20% of true value (for ICAP only) | Rerun standard, if still out of control, recalibrate instrument and reverify calibration. |
| Continuing calibration verification standard (CCV) | +/- 10% of true value | Rerun standard, if still out of control, recalibrate instrument and reanalyze all samples run since last acceptable CCV or document why data are acceptable. |
| Method blank (MB) | $\leq$ two times DL (listed in Table 5-2) | Determine the cause of the blank problem; redigest samples if necessary or document why data are acceptable. |

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy and Inductively Coupled Plasma/Mass Spectrometry (Continued, Page 2 of 2)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Standard matrix spike (QC check standard) | See Table 5-2 for percent recovery control limits | Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike | See Table 5-2 for percent recovery control limits | If standard matrix analytes are within control limits, qualify the data. If not, determine and correct problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike duplicate | See Table 5-2 for RPD control limits | If standard matrix analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze the samples, if necessary, or document why data are acceptable. |

Note: DL = detection limit.
RPD = relative percent difference.

Source: ESE.

Table 13-3. Summary of Corrective Action Procedures for Inorganics, Oil and Grease, Petroleum Hydrocarbons, and TOX

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Calibration curve correlation coefficient | $\geq$ 0.995 | Rerun calibration standards if still out of control prepare new calibration standards and recalibrate the instrument, or document why data are acceptable. |
| Calibration curve | Brackets all sample responses | Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable. |
| Calibration blank | $\leq$ two times the DL (listed in Table 5-3) | out of control, reprocess and reanalyze the blank. |
| Continuing calibration verification standard (CCV) | +/- 20% of true value | Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV or document why data are acceptable. |
| Method blank (MB) | $\leq$ two times the DL (listed in Table 5-3) | Determine the cause of the blank problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Sample replicate (RP)* | See Table 5-2 for RPD | Determine and correct control limits problem, reanalyze sample, if necessary, or document why data are acceptable. |

Table 13-3. Summary of Corrective Action Procedures for Inorganics, Oil and Grease, Petroleum Hydrocarbons, and TOX (Continued, Page 2 of 2)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Standard matrix spike (QC check standard) | See Table 5-2 for percent recovery control limits | Determine and correct problem, reanalyze samples if necessary or document why data are acceptable. |
| Sample matrix spike | See Table 5-2 for percent recovery control limits | If standard matrix analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike duplicate | See Table 5-2 for RPD control limits | If standard matrix analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze the samples, if necessary, or document why the data are acceptable. |

Note: DL = detection limit.
RPD = replicate percent difference.

*Sample replicate is only required for residues, pH, specific conductivity, and turbidity analyses.

Source: ESE.

Table 13-4. Summary of Corrective Action Procedures for Radionuclides

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Background check | See Section 9.4.12 | Check and clean the instrument and repeat the background check. |
| Performance check standard | See Section 9.4.12 | Check the instrument and recount the standard, if still out of control, recalibrate the instrument and recount the standard or document why data are acceptable. |
| Method blank | $\leq$ two times the RL (listed in Table 5-3) | Determine the cause of the blank problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Sample replicate (RP) | See Table 5-2 for RPD control limits | Determine and correct the problem, reanalyze sample, if necessary, or document why data are acceptable. |
| Standard matrix spike (QC check standard) | See Table 5-2 for percent recovery control limits | Determine and correct the problem, reanalyze standard samples, if necessary, or document why data are acceptable. |
| Sample matrix spike | See Table 5-2 for percent recovery control limits | Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable. |

Table 13-4. Summary of Corrective Action Procedures for Radionuclides

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Sample matrix spike duplicate | See Table 5-2 for RPD control limits | Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable. |

Note: RL = reporting limit
RPD = replicate percent difference

Source: ESE.

Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Calibration curve correlation coefficient | $\geq$ 0.995 | Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable. |
| Calibration curve | Brackets all sample responses | Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable. |
| Continuing calibration standard (CCS) | +/- 15% of standard initial response for GC (except for NPD which is +/-25%) and +/- 10% of standard initial response for HPLC | Rerun standard, if still out of control, recalibrate instrument and reanalyze samples when last CCS is acceptable, or document why data are acceptable. |
| Method blank (MB) | < than two times DL for nonvolatile organics (listed in Tables 5-9 to 5-13, 5-16 to 5-35, and 5-40 to 5-61) | Determine and correct cause of the blank problem, reanalyze the samples, if necessary, or document why data are acceptable. |
| Method blank (MB) | No greater than five times DL (listed in Tables 5-5 and 5-7) for methylene chloride, acetone, toluene, and xylene organics. All other analytes must be $\leq$ two times DL (listed in Tables 5-5 and 5-7) | Reanalyze another MB. If second MB exceeds criteria, clean and recalibrate analytical system or document why data are acceptable. |

Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography
(Continued, Page 2 of 3)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Standard matrix spike (SP) | See Tables 5-4 to 5-13, 5-16 to 5-35, and 5-40 to 5-61 for percent recovery control limits | Determine and correct the problem, reanalyze samples if necessary, or document why data are acceptable. |
| Sample matrix spike | See Tables 5-4 to 5-13, 5-16 to 5-35, and 5-40 to 5-61 for percent recovery control limits | If standard matrix spike analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Sample matrix spike duplicate | See Tables 5-4 to 5-13, 5-16 to 5-35, and 5-40 to 5-61 for RPD control limits | If standard matrix analytes are within control limits, qualify the data. If not, determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable. |
| Surrogates* (SUR) | See Tables 5-18, 5-20, and 5-28 for percent recovery control limits | If surrogates in the MB or SP are within control limits, qualify data. If not, reanalyze samples with surrogates outside criteria or document why data are acceptable. |

Table 13-5. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography
(Continued, Page 3 of 3)

Note: DL = detection limit.
GC = gas chromatography.
HPLC = high pressure liquid chromatography.
NPD = nitrogen-phosphorus detector.
RPD = relative percent difference.

*Surrogate/surrogates will only be spiked in samples if specified by the method.

Source: ESE.

Table 13-6. Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| DFTPP or BFB instrument tuning | See Table 8-1 for tuning criteria | Retune instrument until within criteria. |
| Initial calibration standards | Percent RSD of RF of the calibration check compounds (CCC) are $\leq$ 30 percent ($\leq$35 percent for Method 625) | Rerun calibration standards, if still out of criteria, prepare new calibration standards and rerun standards. |
| One-point daily calibration | RFs of CCCs are $\leq$25 percent ($\leq$20 percent for Method 625) from average RFs in the initial calibration | Rerun standard, if still out of control, rerun calibration curve, or document why data are acceptable. |
| Method blank (MB) | < two times the DL (listed in Table 5-39) for semivolatile organics | Evaluate the impact of the presence of any target analytes in the method blank, the presence of low concentrations of phthalate may be acceptable. Reextract and reanalyze samples if presence of target analytes are unacceptable or document why data are acceptable. Background substraction may be applied. |

Table 13-6. Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry (Continued, Page 2 of 3)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Method blank (MB) | No greater than 5 times the DL (listed in Tables 5-15 and 5-37) for methylene chloride, acetone, toluene, and xylene for volatile organics. All other analytes must be ≤ two times DL (listed in Tables 5-15 and 5-37) | Reanalyze another MB. If second MB exceeds criteria, clean and recalibrate the analytical system or document why data are acceptable. |
| Surrogate (SUR) | See Tables 5-14, 5-36, and 5-38 for percent recovery control limits | If surrogates in the MB or SP are within limits, qualify the data. If not, reanalyze samples with surrogates outside criteria or document why data are acceptable. |
| Standard matrix spike (SP) | See Tables 5-14, 5-36, and 5-38 for percent recovery control limits | If surrogates in the MB are within control limits, qualify the data. If surrogates in the MB are not within control limits, determine and correct the problem, reextract and reanalyze the sample, if necessary or document why data are acceptable. |

Table 13-6. Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry (Continued, Page 3 of 3)

| Quality Control | Acceptance Criteria | Corrective Action |
|---|---|---|
| Sample matrix spike | See Tables 5-14, 5-36, and 5-38 for percent recovery control limits | If standard matrix spike compounds are within criteria, qualify the data. If not, check surrogates in the MB or SP, if within criteria, qualify the data. If both QCs are outside criteria, determine and correct the problem, reanalyze the samples or document why the data are acceptable. |
| Sample matrix spike duplicate | See Tables 5-14, 5-36, and 5-38 for RPD control limits | If standard matrix spike compounds are within criteria, qualify the data. If not, check surrogates in the MB or SP, if within criteria, qualify the data. If both QCs are outside criteria, determine and correct the problem, reanalyze the samples if necessary or document why data are acceptable. |

Note: DL = detection limit.
RPD = relative percent difference.

Source: ESE.

Laboratory Department Manager, or Laboratory Director will be responsible for approving the corrective action.

## 13.2 EXTERNAL SOURCES

Corrective action may also be initiated from external sources. This may include performance sample results, split samples, audits (onsite or field by EPA, HRS, FDEP, USATHAMA, HAZWRAP, NEESA, etc.), and data validation/review. Corrective actions recommended by agencies such as EPA, DEP, etc. are prioritized, promptly acted on, and overseen by the Project QA Coordinator or Laboratory QA/QC Manager. Actions taken to resolve the problem will be documented and kept by the Project QA Coordinator or Laboratory QA/QC Manager.

# 14.0 PERFORMANCE AND SYSTEM AUDITS AND PERSONNEL TRAINING

## 14.1 INTRODUCTION

Two types of audit procedures will be used to assess and document performance of laboratory staff: system audits and performance audits. These are performed at frequent intervals by the Laboratory QA/QC Coordinator and QA Coordinator. These audits form one of the bases for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements.

## 14.2 SYSTEM AUDITS

System audits are inspections of training status, records, QC data, calibrations, and conformance to SOPs without the analysis of check samples. System audits are performed quarterly.

The system audit protocol for the laboratory is summarized as follows:
The QA Coordinator and Laboratory QA/QC Manager or designee will perform the laboratory system audit using the checklist in Figures 14-1 through 14-4. The documents to be reviewed are:

a. Parameter and/or laboratory notebooks;
b. Instrument logbooks;
c. Sample log-in, dispensing, and labeling for analysis;
d. QC criteria update for spike recoveries; and
e. Verify that deficiencies in the last audit were corrected.

Coldrooms, Freezers and Sample Storage Areas - Dept. 1213

AUDITOR_____
DATE_____

| No. | Item | Yes | No* | Comment(s) |
|---|---|---|---|---|
| 1 | Is the work area clean and organized? | | | |
| 2 | Are SOPs available for receipt, storage and tracking of samples? | | | |
| 3 | Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected. | | | |
| 4 | Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)? | | | |
| 5 | Are the Sample Tracking forms properly filled out? | | | |
| 6 | Is the Sample Location report updated on a regular basis and placed next to the door of each storage area? | | | |
| 7 | Are all storage areas secured at all times? | | | |
| 8 | Are the temperature logs for the coldrooms and freezers filled out completely and corrections made properly? Are appropriate corrective actions taken for all out-of-control readings? | | | |
| 9 | Is a condensed SOP for check-in/check-out posted next to each storage room door? | | | |
| 10 | Is the Sample Check-In/Check-out log filled out completely? | | | |
| 11 | Is proper documentation available for tracking the disposal of samples? | | | |

Additional Comments:

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-1
AUDIT CHECKLIST FOR SAMPLE STORAGE AREAS

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

Sample Receiving - Dept. 1220

AUDITOR
DATE

| No. | Item | Yes | No* | Comment(s) |
|---|---|---|---|---|
| 1 | Is the work area clean and organized? | | | |
| 2 | Are SOPs available for receipt, log-in and transfer of samples to storage areas? | | | |
| 3 | Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected. | | | |
| 4 | Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)? | | | |
| 5 | Is the Sample Custodian filling out all required information on the chain of custody (COC) form (cooler temp., seals intact? etc.)? | | | |
| 6 | Are the Sample Chest Custody Forms filled out completely? | | | |
| 7 | Is the Sample Custodian completely filling out the Cold Room Sample Arrival logbook? | | | |
| 8 | Is the Sample Custodian auditing 10% of all samples (except VOA samples) to verify that samples are properly preserved? Is documentation available? | | | |
| 9 | Are samples labelled properly? | | | |

Additional Comments:

Hood Maintenance - Dept. 1213

| No. | Item | Yes | No* | Comment(s) |
|---|---|---|---|---|
| 1 | Have fume hoods been calibrated within the last year? Are they labelled as to when last tested? | | | |

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-2
AUDIT CHECKLIST FOR SAMPLE RECEIVING AND GLASSWARE WASHROOM AREAS

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

Sample Preparation Areas
Department_____ AUDITOR_____
DATE_____

| No. | Item | Yes | No* | Comment(s) |
|---|---|---|---|---|
| 1 | Is the work area clean and organized? | | | |
| 2 | Are SOPs available for receipt, storage and tracking of samples? | | | |
| 3 | Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected. | | | |
| 4 | Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)? | | | |
| 5 | Are samples and standards stored separately to avoid contamination? | | | |
| 6 | Are spike solutions, surrogate solutions, (Org. only) and reagents labelled clearly and appropriately (including plastic squeeze bottles)? | | | |
| 7 | Are there expired standards/reagents in the laboratory? Are they clearly labelled as "expired" or "for qualitative use only"? | | | |
| 8 | Is glassware stored so as to avoid contamination? | | | |
| 9 | Do all log books have control numbers? | | | |
| 10 | Are sample preparation logs completely filled out, including preparer and reviewer signatures? | | | |
| 11 | Are automatic pipettes and syringes calibrated each day of use? (Inorganic Division only) Are all water bath thermometers in use calibrated against a NIST thermometer? (Organic Division) Are the calibrations documented in the appropriate logbooks? | | | |
| 12 | Are instrument run logs made properly (e.g., microwave, GPC)? | | | |
| 13 | Are instrument maintenance logs filled out completely and corrections made properly? | | | |
| 14 | Are extracts (sample vials) labelled properly? | | | |
| 15 | Are sample extract/digest chain of custody logs filled out completely and corrections made properly? | | | |
| 16 | Are properly labelled waste containers available? | | | |
| Additional comments: | | | | |

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-3
AUDIT CHECKLIST FOR SAMPLE PREPARATION AREAS

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

Sample Analysis Areas
Department_____ AUDITOR_____ DATE_____

| No. | Item | Yes | No* | Comment(s) |
|---|---|---|---|---|
| 1 | Is the work area clean and organized? | | | |
| 2 | Are SOPs available? | | | |
| 3 | Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected. | | | |
| 4 | Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)? | | | |
| 5 | Are samples and standards stored separately to avoid contamination? | | | |
| 6 | Are spike solutions, surrogate solutions (Org. only), calibration standards and reagents labelled clearly and appropriately (including plastic squeeze bottles)? | | | |
| 7 | Is glassware stored so as to avoid contamination? | | | |
| 8 | Do all log books have control numbers? | | | |
| 9 | Are standard and reagent prep. logbooks filled out completely and corrections made properly? Are lot numbers of neat standards recorded? | | | |
| 10 | Are instrument calibration checks performed prior to analysis? (Mandatory for Radiochemistry, only) | | | |
| 11 | Are instrument run logs filled out completely and corrections made properly? | | | |
| 12 | Are instrument maintenance logs filled out completely and corrections made properly? | | | |
| 13 | Are samples (analysis vials) labelled properly? | | | |
| 14 | Are sample chain-of-custody (COC) logs (VOA samples) or sample extract/digest COC logs filled out completely and corrections made properly? | | | |
| 15 | Are properly labelled waste containers available? | | | |

Additional Comments:

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-4
AUDIT CHECKLIST FOR SAMPLE ANALYSIS AREAS

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

In addition, the Laboratory QA/QC Coordinator or QA Coordinator may monitor analyses randomly to assure adherence to approved analytical methods.

3. Final Reports--The Project QA Coordinator may review all final reports and deliverables before they are sent to the client.

The Gainesville Laboratory is externally audited regularly by the following agencies:

1. State of Florida Department of Health and Rehabilitative Services,
2. State of New Jersey Department of Environmental Protection and Energy,
3. State of California Department of Health,
4. State of Utah Department of Health,
5. U.S. Army Corps of Engineers,
6. American Industrial Hygiene Association, and
7. Army Environmental Center (AEC) (formerly U.S. Army Toxic and Hazardous Materials Agency).

## 14.3 PERFORMANCE AUDITS

The results of interlaboratory studies may be evaluated by the Project QA Coordiantor as part of the performance audits. This evaluation is performed at least quarterly. ESE is participating in the following proficiency programs:

1. National Institute of Occupational Safety and Health (NIOSH) through its Proficiency Analytical Testing (PAT) and Environmental Lead Proficiency Analytical Testing (ELPAT) Programs,
2. EPA Water Pollution and Water Supply proficiency programs,
3. EPA Radiochemistry Intercomparison Study and Blind Performance Samples,
4. U.S. Army Corps of Engineers,
5. U.S. Department of Energy's Environmental Measurements Laboratory Quality Assessment Program, and
6. U.S. Department Of Energy's Hazardous Waste Remedial Actions Program (HAZWRAP).

The following licenses, accreditations, certifications and validations are held by the Gainesville Laboratory:

1. American Industrial Hygiene Association (AIHA),
2. State of Florida Department of Health and Rehabilitative Services for environmental and drinking water analyses,
3. New Jersey Department of Environmental Protection,
4. State of Maryland Department of Health and Mental Hygiene,
5. State of Florida Department of Health and Rehabilitative Services for Radiochemistry,
6. State of California Department of Health Services for hazardous waste testing analyses,
7. State of Tennessee Department of Health and Environment for drinking water and underground storage testing analyses,
8. State of Utah Department of Health,
9. U.S. Army Environmental Center,
10. U.S. Army Corps of Engineers,
11. U.S. Navy, and
12. U.S. Department of Energy's HAZWRAP.

Peer review of all deliverable reports and data will be performed by technically qualified individuals from each major discipline represented in the deliverable. Figure 14-5 is a sample Deliverable Review Sheet.

# ESE | DELIVERABLE REVIEW SHEET

SHORT TITLE: _____ CLIENT: _____

PROPOSAL/PROJECT NUMBER: _____ DATE/TIME TO LEAVE ESE: _____

PROPOSAL/PROJECT MANAGER: _____ CLIENT DUE DATE: _____

AUTHOR(S): _____ DOCUMENT COORDINATOR: _____

_____

| REVIEW CHECKLIST<br>REVIEWER SHOULD PLACE CHECK UNDER APPROPRIATE REVIEW CATEGORY<br>D—DRAFT F—FINAL | | REQUIRED REVIEW | | | | | | | | OPTIONAL REVIEW<br>BY AUTHOR OR OTHER QUALIFIED INDIVIDUAL | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | ① Organization/ Presentation Reviewed | | ① Technical Approach and Conclusions Reviewed | | ② Proposal Costs Reviewed (If Applicable) | | ③ Laboratory Data Checked (If Applicable) | | Tables, Figures, Text Data Checked | | Critical Calculations Checked | | Computer Models/ Programs Verified | | Field Measurements Checked | |
| REVIEWER'S INITIALS | DOCUMENT/SECTION | D | F | D | F | D | F | D | F | D | F | D | F | D | F | D | F |
| | Document | | | | | | | | | | | | | | | | |
| | SECTION(S) | | | | | | | | | | | | | | | | |
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APPROVALS _____ _____ _____ (as appropriate)

AUDITED BY: _____ (QA Manager or designee)

REMARKS: _____

① Required review by at least one reviewer other than author in these categories.
② Required F & A review for proposals.
③ Required check by laboratory coordinator or other individual for laboratory data reports.

ORIGINAL TO PROJECT MANAGER—COPY TO QA

FORM 83345

**Figure 14-5**
DELIVERABLE REVIEW SHEET

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

## 14.4 PERSONNEL TRAINING

The Gainesville Laboratory personnel are trained on health and safety, QA/QC procedures, analytical methods, and the laboratory data management system as specified in the laboratory's SOP on personnel training (SOP-AS3210-004). New personnel are trained prior to performing any actual laboratory work. Laboratory personnel are also required to attend the health and safety and laboratory QA/QC procedures refresher courses that will be offered yearly. The training that each laboratory personnel had attended are documented on the personnel's training records that are maintained by the Laboratory QA/QC Coordinator.

# 15.0 QUALITY ASSURANCE REPORTS

Activities and actions to be reported will include:

1. Results of ongoing performance, systems and analytical method audits, and

2. Data quality review and significant QA/QC problems with proposed corrective action procedures.

The Laboratory QA/QC Manager reports the results of these activities to the Gainesville Laboratory Management. The QA/QC report is done on a quarterly basis or immediately upon discovery of a problem requiring corrective action.

For NEESA and HAZWRAP projects a Quality Assurance progress report summarizing the QA/QC activity associated with the sample collection, receipt, analysis, and data reporting for NEESA or HAZWRAP samples is submitted monthly to these agencies.

STATEMENT OF QUALIFICATIONS
SOLICITATION #05235

TO PERFORM
OFFSITE ANALYTICAL LABORATORY SERVICES
PICATINNY ARSENAL, DOVER, NEW JERSEY

Prepared for:

ICF KAISER ENGINEERS, INC.
1301 Continental Drive, Suite 101
Abingdon, Maryland 21009

Prepared by:

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
14220 West Newberry Road
P.O. Box 1703
Gainesville, Florida 32607

October 14, 1994

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# INTRODUCTION

Environmental Science & Engineering, Inc. (ESE) laboratories offer over 27 years of experience, extensive full service analytical capabilities, national credentials, and a rigorous quality assurance/quality control (QA/QC) program to industrial, engineering, and government clients. ESE is exceptionally qualified in the areas of organics, inorganics, air toxics, radiochemical, and high explosive/munition analyses in a wide variety of environmental and waste matrices including water, soil, air, tissue, and hazardous waste. ESE is one of the few laboratory companies in the nation with the capability to provide all these services to its clients.

Located in Gainesville, Florida; Peoria, Illinois; and Denver, Colorado, ESE's laboratories comprise one of the largest and most well-equipped environmental laboratory networks in the nation. With over $15 million in instrumentation and facilities and a staff of more than 250 scientists, technicians, and support personnel, ESE's laboratories are qualified to handle high volume production analyses, quick turnaround, large-scale multi-year projects, and methods development and validation. Combined with a state-of-the-art laboratory information management system linking all of the laboratories together, ESE's laboratory network is designed to provide responsive and comprehensive analytical services.

ESE's analytical laboratories have an outstanding performance track record on contracts with major Fortune 500 industrial companies and federal, state, and municipal agencies. In the past 10 years alone, ESE's experienced laboratory technical staff have successfully performed major environmental analytical support projects totaling over $80 million for governmental clients including the U.S. Army Environmental Center (AEC), U.S. Department of Energy (DOE), U.S. Army Corps of Engineers (USACE), the U.S. Navy, the U.S. Air Force, and the U.S. Environmental Protection Agency (EPA)-Contract Laboratory Program (CLP). ESE has also provided over $20 million in analytical support services to major industrial clients on contamination assessment, pollution abatement, remedial action, and

environmental survey efforts. The ESE team is thoroughly familiar with the stringent QA/QC and documentation standards required to provide legally defensible data, and has proven capabilities to provide high quality data for large and small projects in a timely and cost effective manner.

ESE's additional key qualifications include:

- Significant capacity of several hundred samples per week in most major analytical method categories;
- Certification and/or approval in 20 states, and by USACE, U.S. Army, U.S. Air Force, DOE, and U.S. Navy;
- Certification by American Industrial Hygiene Association (AIHA) as an industrial hygiene network;
- Successful participation in the EPA-CLP organic and inorganic programs;
- Flexible data management and reporting capabilities including Lotus® 123 and CLP type formats;
- State-of-the-art PC-based laboratory information system in all laboratories with automated QC checks and sample tracking capabilities;
- Sophisticated analytical method development capability;
- Frequently audited QA and sample custody program approved by, and used effectively on various governmental and industrial projects; and
- Proven commitment to client satisfaction and meeting project requirements.

The following proposal sections are structured to mirror the RFP instructions and to assist the evaluator in locating the required information.

## a. CAPABILITIES

SUMMARY OF ESE LABORATORY CAPABILITIES AND SERVICES

ESE's laboratories have substantial analytical capacity for conventional organic, inorganic, and waste characterization methods. Considerable flexibility exists for assigning tasks where current capacity and technical capability best allows the client to be served in a quality and timely manner. This is especially important for large, multi-year projects, and for meeting demanding client turnaround time requirements.

The laboratories offer full analytical support capabilities for:

- Remedial Actions
- Resource Conservation and Recovery Act (RCRA) Investigations and Compliance
- Superfund Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Investigations
- Clean Air Act Compliance
- Stormwater Analyses
- Industrial Hygiene Monitoring
- National Pollutant Discharge Elimination System (NPDES) Permitting
- Drinking Water Testing
- Asbestos and Lead in Paint Assessments

State-of-the-Art instrumentation in ESE's laboratories include:

- Gas Chromatography/Mass Spectrometry (GC/MS) and High Performance Liquid Chromatography (HPLC)
- Ion Chromatography
- Atomic Absorption Spectrometry (AAS) and Inductively Coupled Plasma (ICP)
- ICP/MS
- Classical Water Quality Analyses
- Alpha, Beta, and Gamma Counting

- Optical Microscopy
- Extensive Robotics and Automated Sample Preparation Equipment

Specialized capabilities and services in which ESE's laboratories offer significant experience and capabilities include:

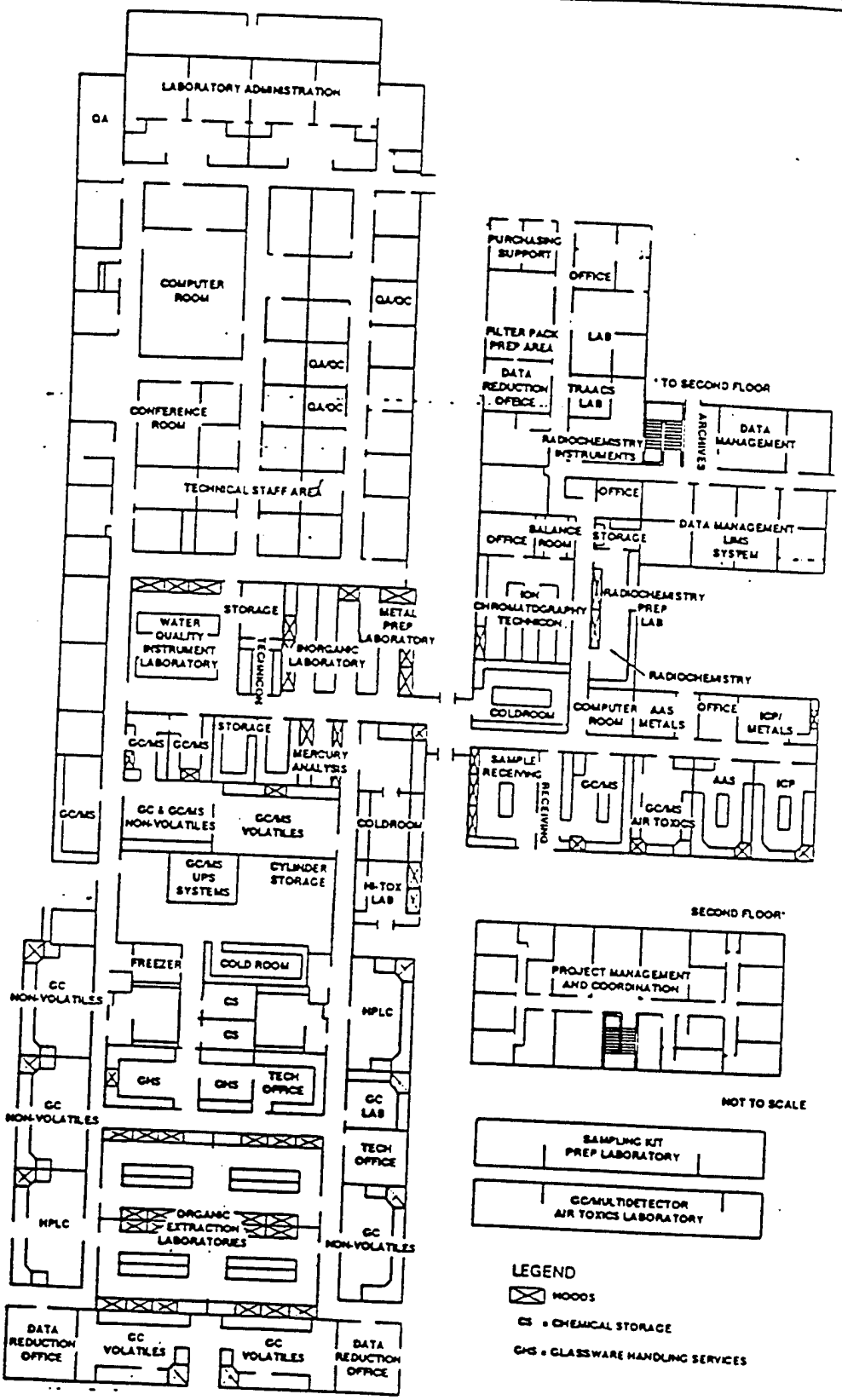
- Explosives and Munition Compounds Analyses
- Air Toxics Capabilities for Inorganics and Organics
- Radiochemistry Analyses
- Agent Degradation Product Analyses

ESE is one of the few private environmental laboratory companies in the nation that has both radiochemical and explosives analytical services.

The laboratory's capacity and expertise for explosives analysis is unsurpassed nationwide. Working with the AEC, ESE has developed numerous methods for explosives and agent precursor/byproduct determinations. Thousands of samples from military installations throughout the nation have been analyzed by ESE over the past decade.

ESE's analytical laboratories currently comprise a total of 66,000 square feet ($ft^2$) of laboratory and office space distributed over three locations: 38,000 $ft^2$ in Gainesville, 15,500 $ft^2$ in Peoria, and 12,500 $ft^2$ in Denver. In order to better facilitate the movement of samples through the laboratories and maintain uncompromised chain-of-custody, each of ESE's laboratories contains separate functional areas dedicated to sample receipt, sample storage, sample preparation, and sample analysis. Floor plans for each of ESE's laboratories are included in the following figures.

Each of the network laboratories have approximately 500 $ft^2$ of space dedicated to sample check-in, accommodating the receipt, identification, and organization of large numbers of samples. Ample fume hoods are available for safe handling of potentially hazardous samples. In these areas, samples are unpacked, logged in to the lab



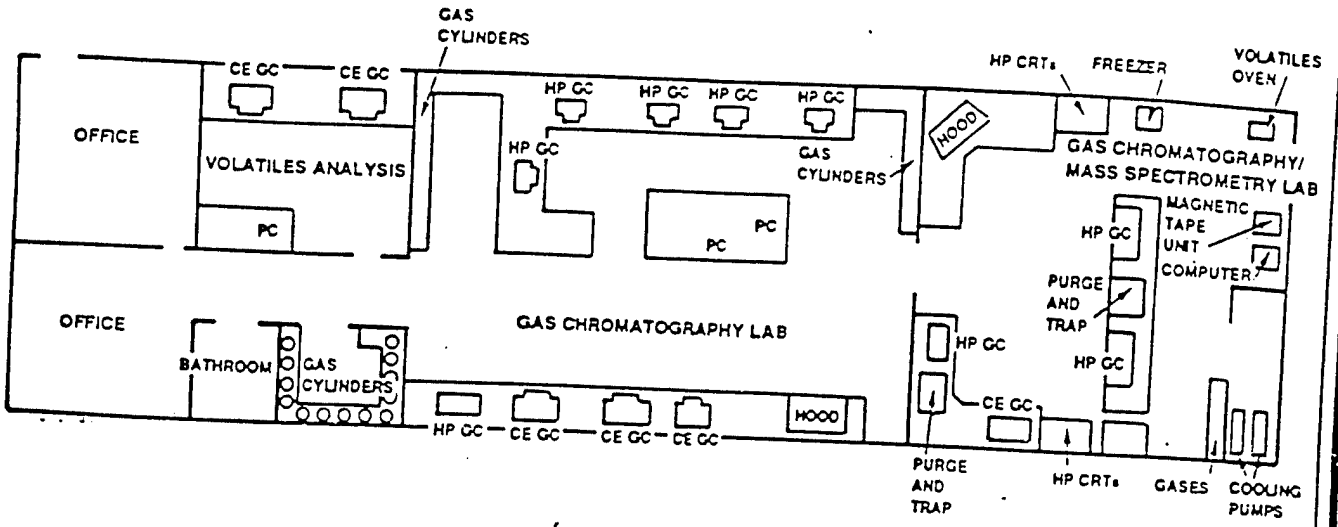

NOT TO SCALE

GAINESVILLE LABORATORY FLOOR PLAN
GAINESVILLE, FLORIDA

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

CHEM C&E 552 BL




LEGEND

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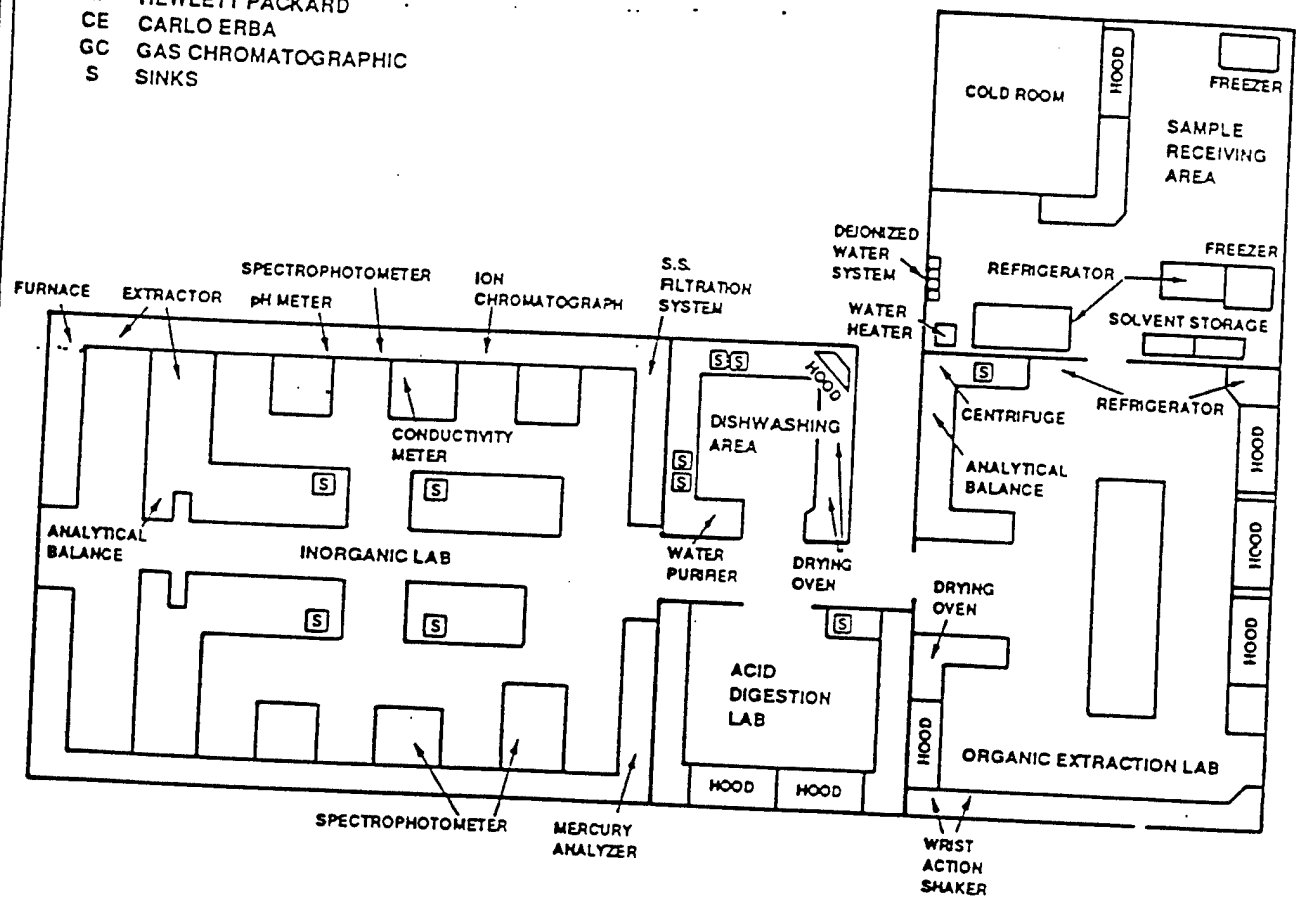
CE CARLO ERBA
GC GAS CHROMATOGRAPHIC
S SINKS




NOT TO SCALE

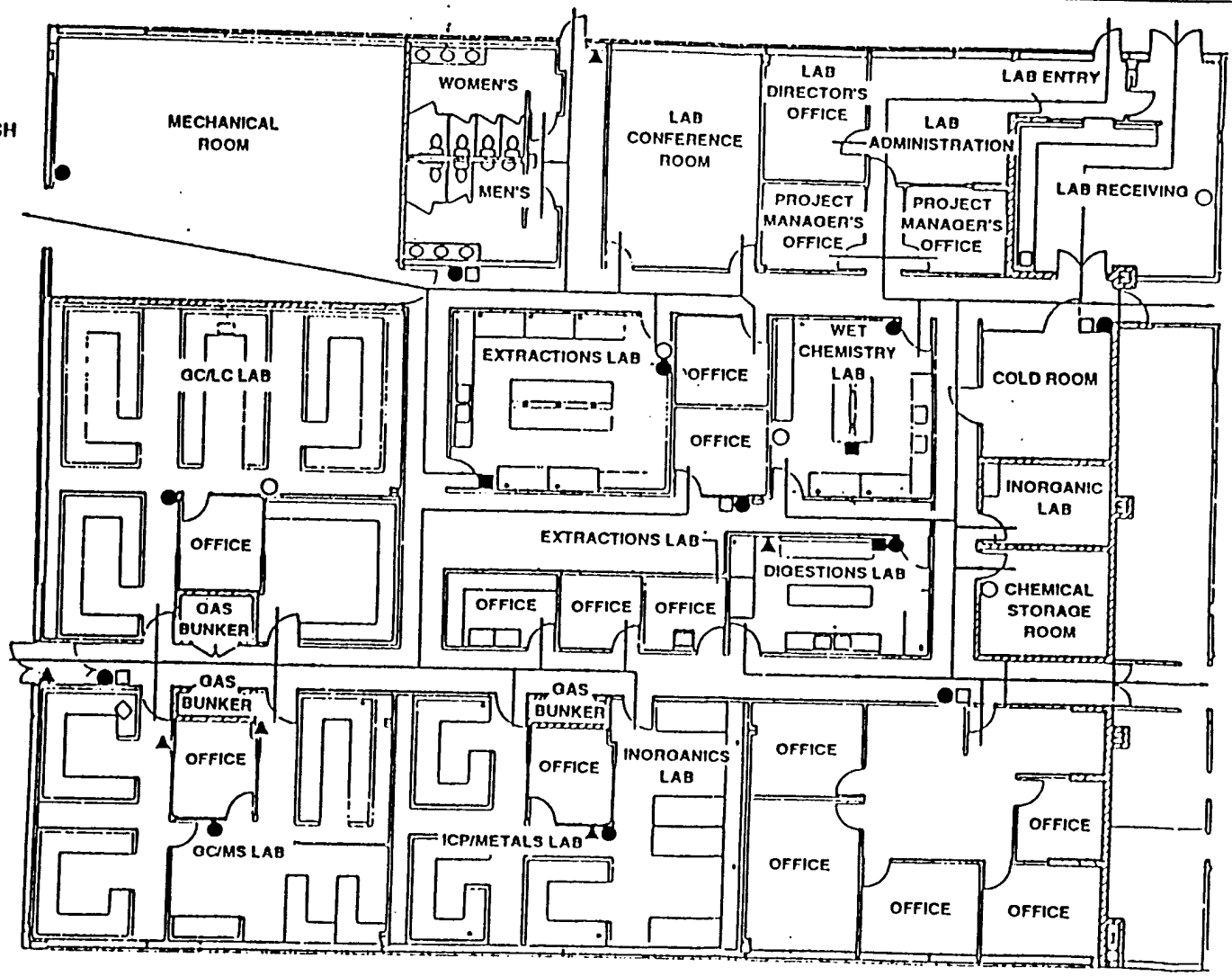
DENVER LABORATORY FLOOR PLAN
DENVER, COLORADO

SOURCE: ESE

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

LEGEND

- ● FIRE EXTINGUISHER
- ○ SPILL KITS
- ■ SAFETY SHOWER/EYE WASH
- □ FIRE BLANKET
- ▲ FIRE PULL BOX




NOT TO SCALE

PEORIA LABORATORY FLOOR PLAN
PEORIA, ILLINOIS

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

tracking system, and screened for radioactivity. Over 11,000 $ft^3$ of walk-in cold rooms are used for storage of environmental samples at 4°C, and approximately 1500 $ft^3$ of walk-in and reach-in freezers are available for storing biota samples at -10°C or below. Throughout the laboratory facilities, numerous smaller refrigerator and freezer units are maintained for storage of standards, spiking solutions, and sample extracts.

The laboratories have dedicated areas for organic extraction, inorganic preparation, metals digestion, GC/MS and HPLC/MS analysis, GC and HPLC analysis, ICP and AA analysis, classical water quality analysis, radiological analysis, toxic chemicals handling, special projects, and additional support areas housing ovens, analytical balances, glassware washing, kit preparation, chemicals storage, waste storage, etc.

The laboratories are supplied with demineralized water for glassware washing and other functions. Demineralized-distilled water is available for reagent preparation, and supplies of organic-free water are maintained at all times for use in trace organic analysis.

Each laboratory facility is equipped throughout with a full range of safety equipment, including fume hoods, eye washes, emergency showers, emergency lights, fire extinguisher, spill cleanup kits, emergency breathing equipment, fire pull boxes, smoke alarms, warning signs, lighted exit signs, safety glasses, and fire blankets.

ESE's laboratories have documented Chemical Hygiene Plans in operation at each facility which provide for training, information, and procedures to maintain analyst safety.

## 1. ANALYTICAL INSTRUMENTATION

ESE's laboratories utilize state-of-the-art analytical instrumentation for multi-matrix chemical analyses. Table a.1-1 provides a list of major analytical instrumentation available at ESE.

Table a.1-1. Major Analytical Instrumentation

| Instrument | Total |
|---|---|
| Organics | |
| GC/MS | 31 |
| LC/MS | 1 |
| GC | 59 |
| HPLC | 18 |
| Metals | |
| ICP/MS | 1 |
| ICP | 6 |
| AA | 15 |
| Mercury Analyzer | 4 |
| Water Quality | |
| TOC | 2 |
| TOX | 2 |
| IC | 8 |
| Autoanalyzer | 8 |
| Spectrophotometer | 8 |
| Radiochemistry | |
| A-B Proportional Counter | 3 |
| Alpha Spectrometer | 32 |
| Alpha Scintillation Counter | 12 |
| Liquid Scintillation Counter | |
| Ge-Li Detector | 2 |
| NaI Detector | 1 |
| Biological Oxidizer | 1 |

Source: ESE.

## ORGANICS

ESE's laboratories have 31 GC/MS, half of which are dedicated to volatile organic analyses and half to semivolatile organic analyses. All GC/MS systems are equipped with automatic samplers and state-of-the-art computer data systems. The mass spectral libraries are the combined Wiley/National Institute for Standards and Technology (NIST) library with 77,000 reference spectra and the EPA/NIST Library with 42,000 reference spectra. The GC/MS facilities also have stand alone GC/FIDs for sample screening. In addition, HPLC/MS is available for specialized analysis of thermally labile, polar/water soluble, and macro-molecular organic species.

Currently there are 59 GCs with flame ionization detectors (FID), nitrogen-phosphorus detectors (NPD), flame photometric detectors (FPD), photoionization detectors (PID), Hall electrolytic conductivity detectors (HECD), electron capture detectors (ECD), and thermal conductivity detectors (TCD) available for project use. In addition, there are 18 HPLCs with ultraviolet (UV), fluorometric, conductivity, electrochemical, and radiochemical detectors. Several units are equipped with post-column derivitization modules and fluorescence spectrometers. A photodiode array spectrometer is also available for simultaneous multi-wavelength monitoring. Autosamplers are used with the GCs and HPLCs for sample handling efficiency, increased sample throughput, and greater precision and accuracy. Automated data acquisition systems are dedicated to each chromatographic unit.

Dedicated GC/MS and GC units are equipped with cryogenic trapping and selective sampling devices for analysis of volatile air toxics collected using SUMMA® canisters, and semi-volatile and volatile organics using sorbent traps.

## INORGANICS

Extensive capability for inorganic metals analysis is provided by 6 ICAPs, both simultaneous and sequential, 15 AA spectrophotometers, and an ICP/MS. The bulk of the metals work is accomplished with the ICAPs. The ICP/MS is available for special projects and R&D work. The AAs are available with flame, graphite furnace,

hydride, and cold vapor capabilities as needed. The ICAPs and AAs are equipped with automated sample introduction and data handling systems.

For water quality and air/industrial hygiene studies, a total of 8 Auto analyzers are available for the routine analysis of sulfur dioxide, nitrite, nitrate, phosphate, sulfate, nitrogen, and silica. Eight ICs are also available to enhance ESE's capabilities to analyze for trace level anionic species, munition and chemical agent degradation products, and for a variety of ionic materials, including organic salts and acids.

RADIOCHEMISTRY

ESE maintains a state-of-the-art radiation counting laboratory and a wet laboratory reserved specifically for radiochemical analysis. In-house instrumentation includes: a Gamma-Ray Spectrometer consisting of a 3-inch by 3-inch sodium-iodide scintillation detector housed in a 4-inch steel shield and coupled to a 1024-channel pulse height analyzer; two High-Performance Germanium-Lithium Spectrometers coupled to 2048-channel pulse height analyzers; three Berthold Low Background Gas-Flow Alpha/Beta Proportional Counting Systems with cosmic ray guard detectors surrounded by 4-inch shield of lead bricks; a Radon Gas Counting System consisting of a Ludlum Radon Flash Photomultiplier Tube coupled to an amplifier scaler-timer; a Beckman Liquid Scintillation Counter; 32 EG&G Alpha Spectrometers; 12 Ludlum Alpha Scintillation detectors; Tissue Oxidizer; and Geiger Counters.

Supporting equipment available in ESE's laboratories includes GPC autoprep units for extract cleanup, sonicators with sonaboxes for soil extractions, centrifuges, shaking devices, blenders, tissuemisers, balances, ovens, etc., and a full range of laboratory glassware necessary for all aspects of environmental analytical chemistry.

## 2. SAMPLE HANDLING CAPACITY

Table a.2-1 gives an overview of ESE's sample handling capacity for selected key analyte groups. The table lists by method category the estimated weekly sample analysis capacity of the ESE network of laboratories. As the table indicates, the

Table a.2-1. ESE's Sample Handling Capacity--Samples Per Week

| Analysis Type | Gainesville | Peoria | Denver |
|---|---|---|---|
| ORGANICS (EPA SW 846 Method): | | | |
| GC/MS Volatiles (8240/8260) | 200 | 50 | 130 |
| GC/MS Semivolatiles (8270) | 200 | 40 | 80 |
| GC Volatiles (8010-20/8021) | 200 | 40 | 80 |
| GC Organochlorine Pesticides/PCBs (8080) | 200 | 20 | 120 |
| GC Organophosphorus Pesticides (8141) | 100 | 10 | 0 |
| GC Chlorinated Herbicides (8150) | 100 | 30 | 50 |
| GC Phenols (8040) | 50 | 30 | 25 |
| HPLC Nitroaromatics/Amines (Explosives) (8330) | 200 | 10 | 100 |
| HPLC Polynuclear Aromatic Hydrocarbons (8310) | 100 | 20 | 50 |
| Total Organic Halides (9020) | 30 | 40 | 0 |
| Total Organic Carbon (9060) | 100 | 0 | 100 |
| Phenolics (9065) | 40 | 90 | 0 |
| Total Recoverable Petroleum Hydrocarbons (9070) | 50 | 60 | 50 |
| INORGANICS (EPA SW 846 Method): | | | |
| ICP Metals (6010) | 350 | 570 | 200 |
| AA Metals (7000s) | 500 | 300 | 100 |
| Mercury | 250 | 125 | 125 |
| Cyanide (9010) | 60 | 80 | 40 |
| Sulfide (9030) | 200 | 80 | 50 |
| Nitrate (9200) | 200 | 700 | 0 |
| WASTE CHARACTERIZATION (EPA SW 846 Method): | | | |
| Appendix IX | 50 | 50 | 40 |
| TCLP (Full, including ZHE) (3111) | 30 | 30 | 30 |
| pH (9040) | 250 | 250 | 200 |
| Corrosivity/Ignitability/Reactivity (9040/1010/9010) | 60 | 40 | 80 |
| RADIOCHEMISTRY (EPA SW 846 or other EPA Method): | | | |
| Gross Alpha/Gross Beta (9310) | 60 | 0 | 0 |
| Uranium (908.0) | 50 | 0 | 0 |
| Cesium (901.0) | 50 | 0 | 0 |
| Strontium (905.0) | 50 | 0 | 0 |
| Tritium (906.0) | 100 | 0 | 0 |
| Gamma Screen (901.1) | 50 | 0 | 0 |
| Radium Total (903.3) | 50 | 0 | 0 |
| AEC/USATHAMA SPECIAL METHODS: | | | |
| Tetrazine | 50 | 0 | 0 |
| Thiodiglycol | 50 | 0 | 0 |
| DIMP/DMMP | 200 | 0 | 150 |
| Organosulfurs | 100 | 0 | 90 |
| IMPA/MPA/EMPA/Fluoroacetic acid | 100 | 0 | 0 |
| VX Mustard Gas Precursors | 50 | 0 | 0 |
| AIR TOXICS (EPA TO Method): | | | |
| VOCs GC/MS (Tenax) (TO-1) | 50 | 0 | 50 |
| VOCs GC/MS (SUMMA Canister) (TO-14) | 50 | 0 | 0 |

Source: ESE.

laboratories have substantial capacity for organic, inorganic, and waste characterization methods, with considerable flexibility for assigning tasks where current capacity and technical ability best allow the client to be served in a quality and timely manner. This is especially important for large, multi-year projects.

## 3. EXISTING WORKLOAD

Tables a.2-2 and a.2-3 show percent availability of selected laboratory equipment based on projected workload at the Gainesville and Denver laboratories. There is adequate capacity and redundancy in instrumentation to handle all samples expected from this project.

The redundancy of staff and facilities provide alternate routes for producing sample data during equipment breakdowns.

An adequate reserve of qualified staff and redundant equipment are critical components affecting a laboratory's ability to meet holding times and turnaround times. ESE's extensive laboratory facilities and staff are fully described in other sections. With a staff of over 250 chemists, technicians, and support personnel, ESE's laboratories are equipped with ample cross-trained staff to meet the needs of this solicitation.

Another consideration in evaluating a laboratory's ability to meet critical holding time and turnaround times is its access to alternate laboratory capacity to allow shifting of workload and opening up capacity for ICF KE samples. ESE's network of three nationally recognized analytical laboratories has equivalent certifications, equivalent QA/QC, and networked data management systems that will ensure consistency within ICF KE's Program. This network also allows shifting on non-ICF KE samples to open up reserve capacity for ICF KE samples within a particular lab, thereby helping to further ensure consistency and chain-of-custody that might bed required for a typical work order. In addition, ESE has teaming agreements with other local laboratories that allow shifting of routine samples. The use of this non-ESE alternate

Table a.2-2. Selected Gainesville Laboratory Equipment

| Item | Manufacturer | Model | # | Available % |
|---|---|---|---|---|
| Gas Chromatograph/Mass Spectrometer (GC/MS) | Hewlett Packard | 5987 | 2 | 40 |
| | Hewlett Packard | 5988 | 1 | 80 |
| | Hewlett Packard | 5995 | 1 | 80 |
| | Hewlett Packard | 5970B | 3 | 30 |
| | Finnigan | INCOS 50 | 2 | 40 |
| Liquid Chromatograph/Mass Spectrometer (LC/MS) | Hewlett Packard | 1092 L2 | 1 | 10 |
| Magnetic Tape Recording System, 9 Track | Hewlett Packard | 7970E | 2x | 50 |
| Linus Tape System | Hewlett Packard | 1000E | 2 | 50 |
| | Hewlett Packard | Series A RTE A4000 | 2 | 50 |
| | Data General | 10-SP | 2 | 50 |
| 76K Compound Library (NIH/EPA MS Database) | NBS/Wiley/Stirs | NA | | |
| Gas Chromatograph (GC) | Hewlett Packard | 5890A ECD/NPD | 2 | 60 |
| | Hewlett Packard | 5890A ECD/ECD | 3 | 40 |
| | Hewlett Packard | 5890A ECD/FID | 2 | 40 |
| | Hewlett Packard | 5890A HECD/PID | 1 | 80 |
| | Tracor | 540(P10/HECP) | 5 | 80 |
| | Varian | 3400 (FPD) | 2 | 75 |
| | Hewlett Packard | 5890A (ECO/NPD) | 2 | 75 |
| | Hewlett Packard | 5890A (NPD/NPD) | 1 | 75 |
| | Hewlett Packard | 5890A (FID) | 1 | 75 |
| | Hewlett Packard | 5890 Series II | 1 | 75 |
| | Hewlett Packard | 5880 (ECD/ECO/ NPD/FID) | 1 | 75 |
| | Hewlett Packard | 5790 (FID) | 1 | 75 |
| | Hewlett Packard | 5730 (ECD/ECD/ ECD/FID) | 2 | 75 |
| | Hewlett Packard | 5710 (FID) | 1 | 75 |
| Inductively Coupled Plasma/ Mass Spectrometer (ICP/MS) | Perkin-Elmer | Elan 5000 | 1 | 5 |
| Inductively Coupled Plasma Spectrometer (ICP) | Thermo Jarrell Ash | 1100 | 1 | 25 |
| | Perkin-Elmer | PII | 1 | 25 |
| Atomic Absorption Spectrometer (AAS) | Perkin-Elmer | 3030 (Graphite) | 1 | 25 |
| | Perkin-Elmer | 4100 (Graphite) | 1 | 25 |
| | Perkin-Elmer | 5100 (Flame/Graphite) | 2 | 30 |
| | Buck Scientific | 400 (Mercury) | 1 | 20 |
| | Perkin-Elmer | 3100 (Mercury/Flame/ Hydride) | 1 | 20 |
| Ion Chromatograph | Dionex | 4000I | 2 | 20 |
| | Dionex | 2000I | 2 | 20 |
| Gel Permeation Chromatography System | ABC Labs | 1002A, B | 2 | 60 |
| High Performance Liquid Chromatograph (HPLC) | Shimadzu | LC-6A | 3 | 40 |
| | Shimadzu | LC-6 | 2 | 40 |
| | Beckman | 332, 342, 330 | 4 | 40 |
| | Altex | 312 MP, 322 MP | 2 | 40 |

Source: ESE.

## Table a.2-3. Selected Denver Laboratory Equipment

| Item | Manufacturer | Model | # | Available % |
|---|---|---|---|---|
| Gas Chromatograph/Mass Spectrometer (GC/MS) | Hewlett Packard | 5995 | 3 | 50 |
| | Hewlett Packard | 5890/5970 MSD | 2 | 30 |
| | Hewlett Packard | 5996 | 1 | 90 |
| Magnetic Tape Recording System, 9 Track | Hewlett Packard | 7970E | 2 | 50 |
| Linus Tape System | Hewlett Packard | 1000E | 4 | 50 |
| 76K Compound Library (NIH/EPA MS Database) | NBS/Wiley/Stirs | NA | 4 | 50 |
| Gas Chromatograph (GC) | Hewlett Packard | 5890A ECD/NPD | 2 | 30 |
| | Hewlett Packard | 5890A ECD/ECD | 2 | 50 |
| | Hewlett Packard | 5890A ECD/FID | 1 | 75 |
| | Hewlett Packard | 5890A HECD/PID | 1 | 20 |
| | Carlo Erba | MEGA ECD/FID | 1 | 20 |
| | Carlo Erba | MEGA ECD/ECD | 1 | 90 |
| | Carlo Erba | MEGA ECD/FPD | 2 | 90 |
| | Carlo Erba | MEGA HECD/PID | 2 | 90 |
| Inductively Coupled Plasma Spectrometer (ICP) | Thermo Jarrell Ash | 61 | 1 | 30 |
| Atomic Absorption Spectrometer (AAS) | Instrumentation Lab | S12 (Graphite/Hydride) | 1 | 30 |
| | Perkin-Elmer | 3030 (Graphite) | 1 | 50 |
| | Perkin-Elmer | 4100 (Graphite) | 1 | 50 |
| | Perkin-Elmer | 5100 (Flame/Graphite) | 1 | 25 |
| | Perkin-Elmer | 50B (Mercury) | 1 | 75 |
| Ion Chromatograph | Dionex | 4000i | 1 | 90 |
| Gel Permeation Chromatography System | ABC Labs | 1002A | 2 | 25 |
| High Performance Liquid Chromatograph (HPLC) | Shimadzu | SCL-6A | 2 | 30 |

Source: ESE.

laboratory capacity would be limited to non-ICF-KE samples. All ICF KE samples will be analyzed within ESE's analytical network except for dioxins/furans and asbestos. Dioxins/furans will be subcontracted to Quanterra, West Sacramento, CA, and asbestos will be subcontracted to Microanalytical Laboratories, Gainesville, FL.

## 4. BUSINESS AND TECHNICAL APPROACH

The approach proposed for this contract will be patterned after the approach used successfully on prior contracts. ESE believes the proposed structure for the contract demonstrates significant depth of personnel to ensure that all contract objectives, including performing multiple tasks simultaneously, will be met in a timely, cost-effective manner.

The successful performance of multi-parameter chemical analyses is dependent upon the combined administrative and technical expertise of the project management. The management structure has been designed to clearly assign authority and responsibility for specific tasks, and provide the close tracking of project progress for the timely completion of the analysis of each batch of samples.

ESE recognizes the need for clear and direct communication between all project team members. It is of the utmost importance that all project requirements are understood by each project team member, including such items as QA/QC requirements, holding times, reporting limits, turnaround, and budgetary constraints. ESE's Laboratory Information Management System (LIMS), called Chemical Laboratory analysis and Scheduling System (CLASS™), has been designed with these communication needs in mind. CLASS™ has been discussed in Section e. In terms of assisting the Project Manager, Project QA Supervisor, Department Managers (Instrument Managers), and analysts, the flexibility of CLASS™ allows for updating systems as project requirements change. The following subsections provide details of each task proposed for this contract.

## 4.1 QA/QC

The requirements which will be submitted to the ICF KE in the Laboratory Quality Assurance Project Plan (LQAPP) will be adhered to throughout the duration of this project. The LQAPP will be in accordance with EPA QAMS-005/80 and will contain the project specific requirements such as:

a. Analytical methods,
b. Calibration procedures,
c. Instrument maintenance procedures,
d. Description of statistical procedures to monitor precision and accuracy,
e. Corrective action procedures,
f. Description of performance audits,
g. Description of chain-of-custody procedures,
h. Description of record-keeping and storage,
i. Description of data reporting, and
j. Description of project organization.

## 4.2 PRE-SAMPLING ORGANIZATION/SAMPLE KITS

Prior to sample arrival, the Project Manager will set-up all the requirements for each sample in the CLASS™ data system. Sample bottles/containers, required preservatives, sample labels, packing materials, coolers, custody seals, trip blanks, and sample analysis request sheets will be provided to the ICF KE field team. The sample containers and preservatives will be equivalent to those described in the QAPjP.

## 4.3 SAMPLE RECEIPT/LOGIN

Samples will be received at ESE by the Sample Custodian who will unpack the samples and check the contents against the chain-of-custody (COC). All observations and any anomalies will be recorded on the COC and/or the cooler receipt form, including the temperature blank readings. ICF KE will be immediately notified of any unusual circumstances that might compromise the quality of the data. The COC will be signed by the Sample Custodian, and forwarded to Data Management for electronic login of all samples.

## 4.4 CHEMICAL ANALYSIS

All analyses will be performed according to the methods specified in the ICF KE QAPjP. These methods include USAEC, SW 846, and EPA CLP methodologies as appropriate and identified in the QAPjP . Method upgrades (such as the use of surrogates and the inclusion of retention time standards and detection limit verification samples) will be used, if necessary, to meet the Data Quality Objectives (DQOs).

All work performed by ESE will meet the requirements of the ICF KE QAPjP. Each analytical batch will contain the following QC samples:

| QC Sample | Frequency |
|---|---|
| Method Blank | One/batch or one/20 samples (for each analysis) |
| Spike | One/batch or one/20 samples (for each analysis) |
| Duplicate or Spike/Duplicate | One/batch or one/20 samples (for each analysis) |
| Reference Samples | One/10 samples (for each analysis) |
| Internal and Surrogate Standards | All samples, GC/MS analysis only |

## 4.5 ADHERENCE TO HOLDING TIMES

ESE will meet all holding times as stated in the ICF KE QAPjP. Holding times for all methods will be calculated from the date of sampling. ESE will use the CLASS™ data system as described in Section e to track and monitor holding times.

a. Any re-analysis or second-column confirmation will be completed within stated holding times.
b. ESE will meet both extraction and analytical holding time requirements.

## 4.6 DATA REPORTING

Hard copy analytical and QA/QC data will be delivered to ICF KE within 30 days of receipt of samples.

ESE will enter all data into the U.S. Army's Installation Restoration Data Management Information System (IRDMIS) and provide electronic data deliverables in the IRDMIS format within 45 days of receipt of samples.

Data packets as defined in the laboratory SOW will be prepared. For CLP methodologies, Level IV CLP data packets will be prepared. For other methodologies, Level IV CLP data packets will be prepared. For other methodologies, Level IV "CLP-like" data packets will be prepared.

## 4.7 ADHERENCE TO SCHEDULE

ESE is fully prepared to provide all of the necessary equipment, personnel, laboratory facility, and logistics to perform the work upon receipt of the samples. ESE will meet the following schedule for the turnaround of analytical results:

1. Standard turnaround will not exceed 30 calendar days from the date the sample is received at the laboratory.

2. Accelerated turnaround will not exceed 14 calendar days from the date the sample is received at the laboratory.

3. Urgent turnaround will not exceed 48 hours from the time samples are received at the laboratory.

## 4.8 REMEDY FOR EQUIPMENT FAILURE

ESE has redundant capacity in all key analytical areas (GC/MS, GC, HPLC, and Metals), as described in Section 3. Service and maintenance contracts are kept current for all major equipment allowing for expedited repair service or loaner parts,

etc. As a final backup, ESE's network of laboratories serves as an analytical backup in case of emergencies.

This network of three nationally recognized analytical laboratories has equivalent certifications, equivalent QA/QC, and networked data management systems that will ensure consistency within ICF KE's program. This network also allows shifting of non-ICF KE samples to other local laboratories to open up reserve capacity for ICF KE. The use of this non-ESE alternate laboratory capacity would be limited to non-ICF KE samples. All ICF KE samples will be analyzed within ESE's analytical network except for dioxins/furans and asbestos. Dioxins/furans will be subcontracted to Quanterra, West Sacramento, CA, and asbestos will be subcontracted to Microanalytical Laboratories, Gainesville, FL.

## 5. METHODS FOR THE DETERMINATION OF DEPLETED URANIUM

Analysis of depleted Uranium in soil samples will be determined by total dissolution of the sample by HF or HF/HNO3 digestion and analysis of the resulting digestate for isotopic ratio $U_{235}/U_{238}$ by inductively coupled plasma/mass spectrometry (ICP/MS).

The ICP/MS technique measures ions produced by a plasma source. The ions produced are entrained in the plasma gas and by means of a water cooled interface, are introduced into a quadrupole mass spectrometer. The mass spectrometer is capable of providing a resolution of at least 1 amu peak width at 10% of the peak height. The elemental ions and molecular ions produced in the plasma and those formed during the introduction of the ion beam into the mass spectrometer, are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Mass calibration and tuning of the ICP/MS are done each time the instrument is set up. The tune, response factor and mass calibration are verified and documented immediately before any sample analysis and at the end of each analytical run, or every eight hours, whichever is more frequent. Calibration of the ICP/MS is achieved by analyzing a calibration standard and a blank. Use of internal standards (Li-6, Sc-45, Y-89, In-115, and Bi-209) is required in all ICP/MS analyses to

compensate for interferences due to sample matrices. $U_{235}/U_{238}$ ratios are determined by comparing their concentrations in a given sample.

Natural uranium has a ratio of over 120 parts U-238 to U-235. Sample results showing in excess of this ratio implies a portion of the U-235 has been removed from the sample, i.e., the uranium has been depleted. If the ratio is less than the 120:1, then the U-235 has been enriched.

## 6. METHODS FOR THE DETERMINATION OF HYDRAZINE

ESE developed methods for the determination of unsymmetrical dimethylhydrazine for AEC. These methods utilized HPLC with electrochemical detection. ESE is prepared to develop and perform method demonstration for the determination of hydrazine, monomethylhydrazine, and dimethylhydrazines if these analyses are required. A literature search for available methods will be performed and the references evaluated for use with samples received from the Picatinny Arsenal. After consultation with ICF KE and AEC a method or methods will be chosen for laboratory evaluation and validation. The method chosen will be developed by ESE and validated according to AEC or EPA protocols as necessary.

## 7. DEVIATIONS FROM STANDARD METHODOLOGIES

ESE will follow the standard methodologies as described in the QAPjP. Should alternate methods be required ESE will get written approval from ICF KE before proceeding with analysis of samples.

ESE realizes that in order to meet some of the data quality objectives required that some methods may need to be modified in order to meet low level detection limits. ESE has had numerous years of experience developing methodologies to meet the AEC program requirements. ESE has been successful at developing low level methods for phosphanate esters, explosives, and phosphoric acids. Should method modifications be required, ESE will develop the SOPs, to include instrumentation maintenance, tuning, calibration, MDLs, QC criteria, blank requirements, and step wise procedures for analysis, and submit these to ICF KE for approval before proceeding.

## b. LABORATORY EXPERIENCE

REFERENCES

The following ongoing projects provide references for comparable work performed by ESE. Each task has been performed on time and within budget.

1. Project Title: RI/FS - Carroll Island/Graces Quarters, Aberdeen Proving Grounds, MD

   Client: Dames & Moore  Contact: Tim Llewellyn

   Contract No.: DACA87-90-D-0025  Amount: $615,926

   Contract Period: Ongoing

   Project Description: ESE was contracted in 1990 to analyze approximately 240 water and 5 sediment samples for volatile and semi-volatile organic compounds, total and dissolved metals, and cyanide following CLP 3/90 SOW and producing full electronic and hard copy CLP deliverables. The same samples were also analyzed for explosives and chemical agent byproducts (thiodiglycol, DIMP, DMMP, MPA/IMPA) utilizing AEC (USATHAMA) methodology. Most of the water samples were also analyzed for various water quality indicator parameters and selected radionuclides including gross alpha, gross beta, and tritium using EPA methods. Analytical data was input into the AEC database for all samples and analyses. This contract is ongoing.

2. Project Title: Analytical Support Contract for Rocky Mountain Arsenal (RMA)

   Client: Program Manager Rocky Mountain Arsenal  Contact: Douglas Stevenson

   Contract No.: DAAA05-92-D-0012  Amount:$4.7 million

   Contract Period: Ongoing

   Project Description: ESE conducted an extensive analysis program in support of the sampling activities at the RMA. The ESE laboratory was responsible for the chemical characterization of water, soil, air, and biological tissue at RMA. ESE offered a full-range of analytical services on this contract including GC/MS, GC, HPLC, ion chromatography, metals, thiodiglycol, explosives, and agent degradation products. Approximately 1,200 soil samples, 1,300 water samples, 1,000 air samples, and 1,500 biota samples are being analyzed as part of this contract.

3. Project Title: U.S. Army Total Environmental Program Support (TEPS)

Client: AEC Contact: Robert Turkletaub

Contract No.: DAAA15-90-D0017 Amount:$10 million

Contract Period: Ongoing

Project Description: ESE, as prime TEPS contractor, has conducted several confirmation surveys, remedial investigations/feasibility studies/remedial designs for several task orders. The descriptions that follow are highlights of the analytical portion of selected task orders.

Ft. Sheridan, RI/FS--ESE laboratory provided analytical services in support of an RI/FS at Ft. Sheridan, IL. The RI/FS verified and quantified the nature and extent of contamination, performed public health and environmental risk assessments, and evaluated remedial action alternatives prior to the closing of the installation. Analytical services included analysis of several hundred groundwater, surface water, soil, sediment, wood and wipe samples for TCL volatiles, semivolatiles, organochlorine pesticides, PCBs, herbicides, cyanide, TAL metals, and bulk samples for asbestos.

Defense Distribution Region-Sharpe Site, RI/FS--ESE has been contracted to provide services to comply with the most recent EPA guidance at this Defense Logistics Agency Superfund site in northern California. ESE has been providing environmental consulting, remediation, and analytical services for the RI/FS at the Sharpe site since 1984. This task involves the collection and analysis of soil and groundwater samples for total petroleum hydrocarbons (as gas and diesel), volatile organic compounds, organochlorine pesticides, PCBs, priority pollutant metals, bromacil, and water quality indicators. The active participants in this project include AEC, two California regulatory agencies, EPA, DLA, Sacramento District USACE, four local regulatory agencies, three subcontractors, four other consulting firms, and several offsite property owners.

Letterkenny Army Depot (LEAD), RI/FS/RD--ESE is currently conducting remedial investigations and will subsequently prepare endangerment assessment and feasibility study documents covering both of the defined NPL site portions at the 19,500-acre depot (Property Disposal Area and Southeastern industrial area). All work performed for AEC under this task order is in response to the requirements of an Interagency Agreement between the Army, EPA, and Pennsylvania Department of Environmental Resources.

The analyses included TCL volatiles, TCL semivolatiles, organochlorine pesticides, PCBs, cyanide, TAL metals, and water quality indicators. A 6-month effort of bi-weekly sampling and analysis of algae, surface water and fish tissue samples was conducted to determine source of mercury contamination. The

samples were analyzed for low-level mercury. All analyses were performed under AEC's rigorous QA/QC program using certified and approved methods. Control charts and comment/corrective actions were processed weekly for analytical lots and data were electronically transmitted to AEC's data system.

4. Project Title: Environmental Sample Analysis, CLASS

Client: USAEC Contact: Mary Stutz

Contract No.: DAAA15-87-D-0015 Amount: $5,050,000

Contract Period: 1987-1992

Project Description: ESE was responsible for 109 task orders which involved method certification; chemical analyses, formatting and transmitting all chemical data to USAEC computer. ESE one of five participating laboratories in USAEC's CLASS program. This project involved analysis of environmental samples in support of remedial investigations/activities. ESE offered a full range of analytical services on this contract including GC/MS, GC, HPLC, ion chromatography, and routine water quality tests using certified and approved methods. Standard requests included complete CLP target compound list (TCL) and target analyte list (TAL), explosives, and agent breakdown by-products.

All analyses were performed under USAEC's rigorous QA/QC program using certified and approved methods. Control charts and comment/corrective actions were processed weekly for analytical lots and data were electronically transmitted to USAEC's data system. Some of the installations for which ESE has provided analytical services include:

Rocky Mountain Arsenal, CO
Fort Campbell, KY
Picatinny Arsenal, NJ
Tobyhanna Army Depot, PA
Letterkenny Army Depot, PA
Redstone Arsenal, AL
Fort A.P. Hill, VA
Anniston Army Depot, NY
Seneca Army Depot, NY
Badger Army Ammunitions Plant, WI
Lonestar Army Ammunitions Plant, TX
Umatilla Army Ammunitions Plant, OR
Blossom Point, MD
Aberdeen Proving Ground, MD
Fort Richardson, AK
Phoenix Military Installation, MD
Cornhusker Army Ammunition Plant, NE
Milan Army Ammunition Plant, TN
Alabama Army Ammunition Plant, AL
Joliet Army Ammunition Plant, IL

5. Project Title: Environmental Sample Analysis in Support of Remedial Investigations and Feasibility Studies (RI/FS) at Aberdeen Proving Ground, Maryland

Client: ICF Kaiser Engineers, Inc. Contact: Mr. Gary Nemeth
Ms. Nora Okusu
Mr. Larry Theabeau

Contract No.: DAAA15-91-D-0014 Amount: $1,120,000

Contract Period: 1992 to present

Project Description: ESE has provided analytical support to ICF KE for four tasks (6, 10, 11, 12) associated with the RI/FS at Aberdeen Proving Ground, Edgewood and Aberdeen, Maryland. Over 500 samples of groundwater, surface water, soils, sediments, and biota have been submitted for analyses since 1992. The analytical categories for this project include: TCL organics (volatile organics, semivolatile organics, and pesticides/PCBs), TAL inorganics (metals and cyanide), explosives, chemical surety material (CSM) precursors and degradation products (including organosulfer compounds, DIMP/DMMP, Thiodiglycol, and IMPA/MPA/ Chloroacetic acid), herbicides, radionuclides (gross alpha, gross beta, gamma scan), and various water quality parameters including anions (chloride, bromide), nitrate/ nitrite, sulfate, ammonia, hardness, alkalinity, and phosphorus. ESE also developed an analytical method for the determination of Trichlorophenylurea (TCPU) in sediment for use on this project. All data generated for this project was entered into the AEC IRDMIS database. EPA CLP data validation deliverables were produced and transferred to ICF KE for the TCL and TCL analytes.

## c. PROJECT ORGANIZATION AND MANAGEMENT

This section discusses ESE's proposed program organization and structure; ESE's philosophy and approach to internal and external communications, scheduling techniques, and procedures; and ESE's approach to compliance with ICF KE's reporting requirements.

### 1. PROJECT ORGANIZATION AND STRUCTURE

Figure c.1-1 presents the proposed project organization. The goals of ESE's management approach are listed below:

1. Submitting quality products on time and on budget;
2. Committing qualified staff and resources with assurance of continuity on add-ons and the subsequent year's work; and
3. Utilizing cost-effective approaches, scheduling, and personal assignment to minimize cost to ICF KE while maintaining quality and schedule

ESE's approach offers the following features:

1. An organizational structure with clear lines of authority, accountability, and responsibility;
2. A Project Manager (PM) as the main point of contact and coordination throughout the contract;
3. Designated, experienced Department managers (Instrument Managers) who will lead the activities for each specific method;
4. Laboratory Field Sample Coordinator who will serve as a liaison between ICF KE field team and ESE PM;
5. A Quality Assurance Supervisor to ensure compliance with the project QA/QC Plan; and
6. Centralized support and oversight.

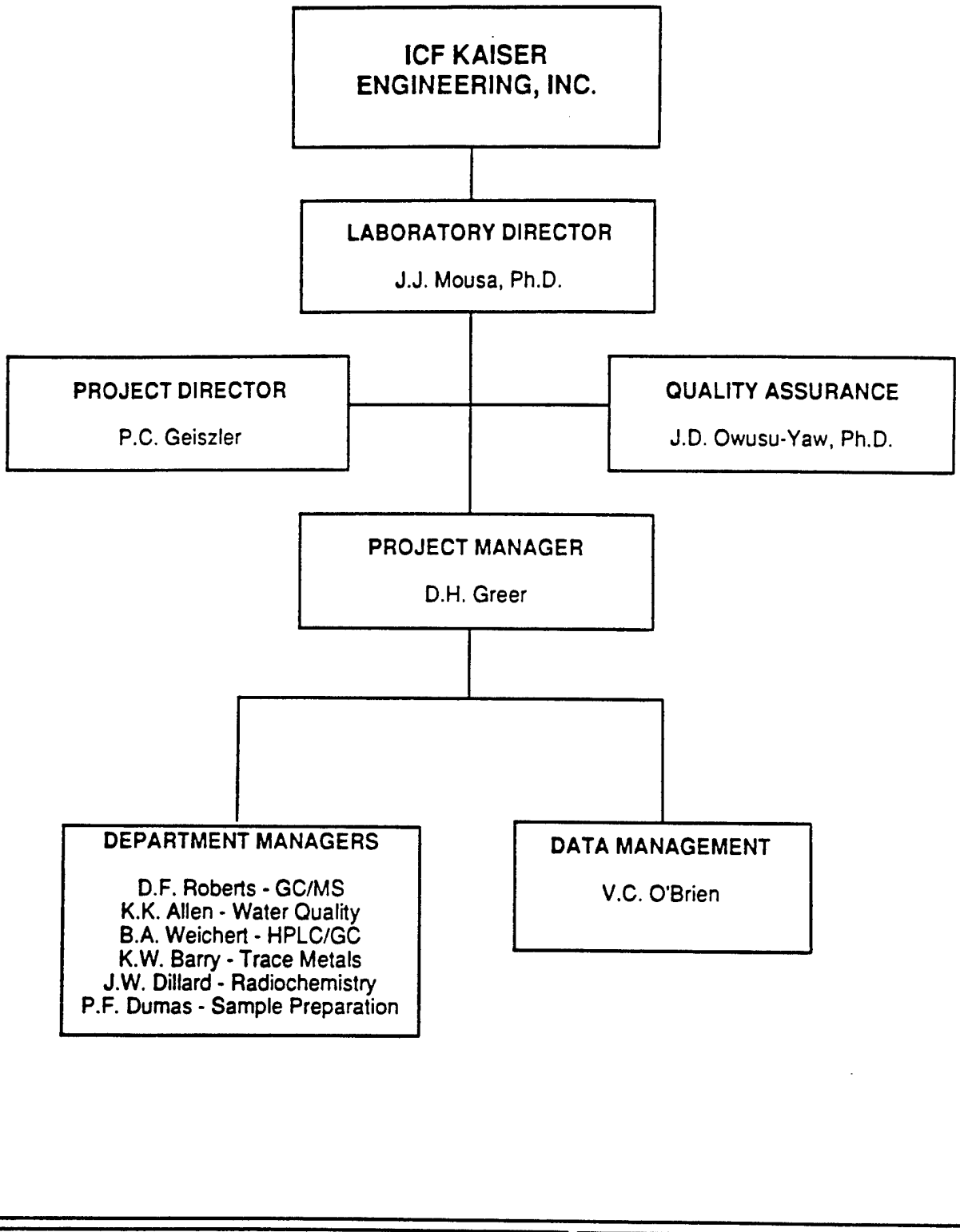



**Figure C.1-1**
**PROPOSED PROJECT ORGANIZATION**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

In order to better present ESE's organizational structure, the various titles of management personnel are provided below:

Laboratory Director -- Responsible for the overall performance of the laboratory.

Project Director -- Responsible for the performance of the ESE Project Team

Quality Assurance Supervisor -- Responsible for administration of the project QA/QC plan.

Project Manager (PM) -- Overall project management responsibility and authority.

Department Manager (Instrument Manager) -- Responsible for the performance of a specific analytical task within a project.

## 2. KEY PERSONNEL

ESE's laboratories have a solid record of employee retention and over the past 20 years have developed a highly skilled, experienced laboratory management and technical staff. Most of ESE's top laboratory managers have been with ESE for over 10 years. The high experience base and technical credentials of our staff ensure that ICF KE receives a high quality product and outstanding service. Laboratory personnel have the extensive scientific expertise needed to support technically complex analyses.

ESE staff of over 250 chemists, scientists, and technicians is extremely qualified to meet the demand of this contract. The Team has the trained personnel to perform CLP TCL and TAL analyses, herbicides, chemical agent degradation products, explosives, TOC, radiochemistry, as well as any required water quality parameters. Table c.2-1 provides a selected list of ESE personnel showing a broad range of methodology and instrumentation experience. Resumes of selected key project personnel are provided in Appendix A.

# Table c.2-1. Experience Summary for Analytical Chemists

| Personnel | Function | Highest Degree | Years of Experience at ESE | Methodology Experience | | | | | | | | | | | | Instrumentation Experience | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Volatiles | Semivolatiles | Pesticides | PCBs | Explosives | Metals | Radionuclides | Water Quality | CLP | SW-846 | Drinking Water | Wastewater | GC/MS | GC | HPLC | ICAP | AAS | IC | Technicon |
| P.F. Dumas | Extraction/Prep. | A.A. | 7 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | | | |
| B.W. Smith | GC | B.S. | 15 | ✓ | | | ✓ | | ✓ | | ✓ | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | ✓ | |
| V.S. Bauder | GC | B.S. | 8 | | | ✓ | ✓ | | | | | ✓ | ✓ | ✓ | ✓ | | ✓ | | | | | |
| J. Kostelnik | GC | B.S. | 7 | | | ✓ | ✓ | | | | ✓ | ✓ | ✓ | ✓ | ✓ | | ✓ | ✓ | | | | |
| B.R. Meek | GC | H.S. | 6 | ✓ | ✓ | ✓ | ✓ | | ✓ | | ✓ | | ✓ | ✓ | ✓ | ✓ | ✓ | | ✓ | ✓ | | ✓ |
| R.Y. Jaw | GC | M.S. | 5 | | | ✓ | ✓ | | ✓ | | | | ✓ | ✓ | ✓ | | ✓ | ✓ | | | | |
| R.R. Robertson | GC | B.A. | 4 | | | ✓ | ✓ | | | | ✓ | | ✓ | ✓ | ✓ | | ✓ | ✓ | | | | |
| D.F. Roberts | GC/MS | B.S. | 16 | ✓ | ✓ | ✓ | ✓ | ✓ | | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | | | ✓ |
| P.H. Chen | GC/MS | Ph.D | 9 | ✓ | ✓ | | | ✓ | | ✓ | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | | | |
| B. VanAusdale | GC/MS | M.S. | 8 | ✓ | ✓ | | ✓ | ✓ | ✓ | | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | | ✓ | | ✓ |
| G.C. Lamb | GC/MS | B.S. | 8 | ✓ | ✓ | ✓ | ✓ | | ✓ | | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | | | | | |
| W.C. Whitehill | GC/MS | B.S. | 4 | ✓ | ✓ | | | ✓ | | | | ✓ | ✓ | ✓ | ✓ | ✓ | | ✓ | | | | |
| R.D. Baker | HPLC | B.S. | 10 | | | ✓ | ✓ | ✓ | | | | ✓ | ✓ | ✓ | ✓ | | ✓ | ✓ | | | | |
| S.D. McMillen | HPLC | B.S. | 4 | ✓ | | | | ✓ | | | | ✓ | ✓ | | ✓ | ✓ | | ✓ | ✓ | | | |
| K. Waggoner | HPLC | B.S. | 4 | | | | | ✓ | | | | | ✓ | ✓ | ✓ | | | ✓ | | | | |
| B. Weichert | GC/HPLC | B.S. | 8 | ✓ | | ✓ | ✓ | ✓ | | | | ✓ | ✓ | ✓ | ✓ | | ✓ | ✓ | | | | |
| R.M. Kinney | Radiochemistry | B.S. | 6 | | | | | | | ✓ | ✓ | | | | ✓ | ✓ | | | ✓ | | | |
| S.D. Stewart | Radiochemistry | H.S. | 9 | | | | | | ✓ | ✓ | | | | | ✓ | | | | | ✓ | | ✓ |
| K.W. Barry | Trace Metals | B.S. | 8 | | | | | | ✓ | | | ✓ | ✓ | | | | | | ✓ | ✓ | ✓ | ✓ |
| M.R. Mansell | Trace Metals | A.S. | 8 | | | | | | ✓ | | ✓ | ✓ | ✓ | | ✓ | | | | ✓ | ✓ | ✓ | |
| G.L. Price | Trace Metals | A.S. | 7 | | | | | | ✓ | | | ✓ | ✓ | | ✓ | | | | ✓ | ✓ | ✓ | |
| L.L. Osborne | Trace Metals | B.S. | 4 | | | | | | ✓ | | | ✓ | ✓ | | ✓ | | | | ✓ | ✓ | ✓ | |
| J.R. Elliott | Water Quality | A.S. | 15 | | | | | | | | ✓ | | ✓ | | ✓ | | | | | ✓ | ✓ | |
| M.G. Travis | Water Quality | A.A. | 14 | | | | | | | | ✓ | | ✓ | | ✓ | | | | | ✓ | ✓ | ✓ |
| K.A. Allen | Water Quality | B.S. | 10 | | | | | | | | ✓ | ✓ | ✓ | | ✓ | | | | | ✓ | ✓ | ✓ |
| F.G. Wilson | Water Quality | A.A. | 7 | | | | | | | | ✓ | | ✓ | ✓ | ✓ | | | | | ✓ | ✓ | ✓ |
| E.D. Kennelley | Water Quality | M.S. | 4 | | | | | | ✓ | ✓ | | ✓ | ✓ | | ✓ | | | | | ✓ | ✓ | ✓ |

## 3. MANAGEMENT APPROACH

This section presents the roles and responsibilities of the Project Manager, Department Managers, and QA Supervisor. Designation of these roles and responsibilities are necessary to 1) provide responsiveness to the needs of ICF KE on this project, 2) maintain consistent and quality products, and 3) provide responsiveness as schedules and requirements change.

The following are the role and responsibilities of the Project Manager:

- Primary point of contact between ICF KE;
- Overall project management and control;
- Compliance with contract and assurance that the work order is staffed properly to achieve goals, objectives, schedule, and budgets;
- Preparation of overall project status report;
- Monitoring of each phase of the project for compliance with scope, scheduling, and budget requirements as well as adherence to QA plans and procedures and health and safety requirements;
- Through frequent monitoring, the PM will identify, communicate, and resolve problems. Corrective action reports will be prepared and transmitted to ICF KE as required;
- Allocate budgets among the tasks required for the project;
- Approve or disapprove of any labor, materials, or subcontractor charges to the project;
- Establish and enforce work element milestones to ensure timely completion of the work activities;
- Authorize support personnel to participate in the project as required;
- Approve or disapprove contributions to any technical deliverable for each work element;
- Communicate frequently with the ICF KE Project Manager with regard to day-to-day progress of the project; and
- Direct and monitor performance of subcontractors.

- Serve as the field sample coordinator to allow good communication between ICF KE field team and analytical laboratory.

The role of QA Supervisors will perform the following tasks:

- Monitor and periodically audit to ensure that QC procedures, as identified in the QAPjP, are followed by the project team;
- Review any and all project chemical analysis data for compliance with QA requirements and technical accuracy;
- Ensure that adequate QC documentation is provided for the analytical programs;
- Ensure that all QC problems are resolved in an expeditious manner and brought to the attention of the Project Manager and ICF KE as required;
- Coordinate and oversee QA/QC procedures employed by subcontractors; and
- Ensure that all observations, conclusions, and recommendations have had review by qualified and appropriate personnel.

To fulfill these responsibilities, the QA Supervisor has the following authority:

- Coordinate the preparation of QAPP's as required, and ensure consistency of all subsequent add-ons;
- Conduct unannounced audits of analytical laboratory procedures;
- Approve or disapprove of all laboratory data, based upon their compliance with project QC requirements;
- Require cessation of laboratory activities that are out of compliance with the QAPjP;
- Reject laboratory data not complying with project QC requirements; and
- Oversee review of reports for quality and conformance with contract and delivery order requirements.

The Data Management Supervisor will be responsible for transmission of all completed data coding forms to AEC through the Project Manager. Specific responsibilities follow:

- Supervising the operation of the data management system;
- Incorporating AEC QC and data management requirements into the ESE chemical data management system;
- Reviewing all complete field data coding forms for compliance with IR Data Management requirements;
- Instructing laboratory and field personnel in the proper procedures for coding data; and
- Transmitting approved level 1 data on a regular scheduled basis to the AEC IRDMIS.
- Providing support for EPA Level IV data package assembly and electronic data deliverables.

To fulfill his responsibility to transmit all completed data to the IRDMIS upon Project Manager approval, the following are the authorities of the Data Management Supervisor:

- Approving or disapproving laboratory and/or field data with regard to formatting as required by the Project Data Management Plan; and
- As point of control, directly communicating with AEC data management personnel and their designated contractors with regard to data transmittal and data transmittal problem resolution.

The laboratory Department Managers are responsible for satisfactory completion and technical quality of the assigned chemical analyses for their respective department. Specific responsibilities include the following:

- Scheduling of analysts and equipment for each assigned task;
- Reviewing analytical data;
- Redirecting technical priorities to ensure compliance with project technical criteria and schedule; and
- Supervising and directing analysts and technicians.

## MANAGEMENT OF SUBCONTRACTORS

ESE believes that it has an exemplary record of subcontractor performance, including cost control. This record will be sustained throughout this contract by careful supervision of the subcontractors. Close control will be maintained through a well-defined subcontract and through frequent reporting of budgetary, technical, and schedule tasks.

Concurrently, the Project Manager and the QA Supervisor will review subcontractor's technical activities to ensure satisfactory performance.

ESE will be subcontracting the dioxins/furans and asbestos analyses, since ESE does not perform these methods in-house. ESE will subcontract the dioxins/furans to Quanterrra, West Sacramento, CA. ESE has subcontracted dioxins/furans analysis to Quanterra over the last 10 years, including major contracts with the Department of Defense and the Department of Energy. Quanterra has an excellent reputation for dioxins/furans analyses and is presently under contract to ESE to provide these services for other ongoing contracts. ESE will subcontract the asbestos analysis to Microanalytical Laboratory located in Gainesville, FL. ESE has a successful relationship working with Microanalytical. Microanalytical has numerous years of experience in asbestos analysis by TEM. Quanterra and Microanalytical Laboratory will comply with the terms and conditions of the subcontract between ICF KE and ESE.

## RESPONSE TO PROBLEMS

If during the course of the project a problem arises in work performance, the Project Manager will be directly notified. Problems, depending upon their nature will be resolved by the PM by (1) securing additional staff, (2) authorizing repeat of analysis, (3) removing personnel or subcontractors from the project, or (4) requesting interpretation or guidance from the ICF KE Project Manager. ICF KE should likewise direct any problems to the ESE Project Manager for resolution.

Although ESE has proposed a mechanism for problem resolution, it is the company's intention to minimize problems through proper advance planning and by keeping all lines of communication open.

## SCHEDULING

### Work Order Scheduling

ESE recognizes that the schedule requirements issued by ICF KE result from regulatory compliance agreements between ICF KE's customers and state and federal regulatory agencies. Hence, ESE treats adherence to schedules seriously and utilizes appropriate management controls to plan and control its project performance.

### Compliance with Holding Times and Turnaround Times

The successful performance of a laboratory on this contract requires the availability of management structures and systems and adequate staff and equipment to meet required holding and turnaround times. This section describes the systems ESE has developed to ensure meeting of holding times and turnaround times.

### Meeting Holding Times and Turnaround Times

ESE understands the importance of maintaining task order analysis schedules and performing analyses within AEC, EPA and ICF KE defined holding times. Data generated in a timely manner is critical to decision making concerning remedial actions, for meeting regulatory reporting deadlines and for assessing potential personnel exposure. A coordinated functional mechanism for maintaining analytical

data reporting schedules and analytical holding times is critically important. Samples must be analyzed on time with no reruns for missed holding times. Missed holding times have the potential to involve additional sampling costs and can lead to severe political and logistical problems for ICF KE.

ESE currently has in operation tested mechanisms to minimize holding time problems and assure maximal compliance with turnaround times. These procedures are used to:

1. Notify analytical task managers and team leaders of the arrival of samples for analysis in a timely manner;
2. Track the status of samples through the laboratory analysis and review procedures;
3. Monitor compliance with sample holding times via a computerized tracking procedure; and
4. Communicate delays in the sample analysis and/or review chain to project management in a timely manner to implement effective corrective actions.

In addition, redundant staff and facilities and the availability of regional laboratory capacity provide added confidence in ESE's ability to meet critical holding times and turnaround times. These ongoing tracking and management procedures and ample reserve capacity have allowed ESE to meet critical turnaround time requirements on a number of high sample load contracts in the past year and has virtually eliminated holding time problems.

**Sample Holding Time Tracking**

As part of ESE's Chemical Laboratory Analysis and Scheduling System (CLASS™), each analytical team leader receives a daily listing of all samples requiring analysis by a particular method. Samples appear on this report within 24 hours of receipt in the laboratory and after log-in into the CLASS™ system. For emergency and fast turnaround samples (24 to 72 hrs.), samples are hand carried to the appropriate analytical team prior to appearing on the available numbers report. In addition to

providing notification of sample availability for analysis, the available numbers report also provides the team leader and analyst of the number of days left in order to meet the required holding times for the analysis and the number of days before the data is due.

In support of this program, the ESE laboratories have a full-time person designated as the Sample Tracking Assistant (STA) for each laboratory whose responsibilities include the daily checking of the status of samples which are nearing holding times for extraction and/or analysis. If a sample has only one day (three days if on a weekend) left before holding time will be exceeded, the STA notifies the analytical team leader and the laboratory task manager in writing. Follow-up and corrective action may then be instituted.

**Weekly Project Priorities Meeting**

ESE's analytical laboratory management holds a weekly project priorities meeting to review the status of all samples in the laboratory. Particular attention and emphasis is placed on sample lots which are nearing their due dates and or have potential to exceed turnaround times. Laboratory management reviews all projects and sets priorities for the analytical team leaders to assure meeting of turnaround times.

## 4. CORRECTIVE ACTION PROCESS

Rapid, effective, and thorough means of implementing the correction of QA problems and for noncomplying items, as well as followup reports, are essential to the implementation of a QA program. Items which may need corrective action range from a minor problem of a field team member failing to sign a field form to a major problem of an analyst using an improper analytical method. For this reason, corrective action protocols must be flexible.

In general, items needing corrective action fall into three "correction" categories: short-term, long-term, and QC; each requiring different action.

## SHORT-TERM CORRECTIVE ACTIONS

These actions consist of minor and major problems which can be corrected immediately. Examples include failure to date or sign a field form, and errors in data entry. Corrective action is initiated by verbally calling attention to the problem followed by written notification.

## LONG-TERM CORRECTIVE ACTIONS

The actions consist of minor and major problems which require a series of actions to resolve the problem. The actions to be taken are coordinated by the QA Supervisor, and a QA Corrective Action Report Form (Figure c.4-1) is used to track the action. These forms can be used as the means of notifying AEC that problems have arisen with samples. The CAR forms can document the actions taken by the laboratory to solve the problem, or they can be used to solicit guidance from ICF KE as to the appropriate action that should be taken by the laboratory. In either event, the CAR will serve as the means for documenting problem resolution. The Project QA Supervisor audits this process to assure that it is completed in an expeditious manner.

## QUALITY CONTROL CORRECTIVE ACTION

Consists of corrective action following a failure to meet QC criteria specified in this QA Plan and the analytical methods. Actions taken consist of two types: those resolved within each analytical department and those resolved outside the department. Examples outlining the differences between these two types of corrective action are as follows:

# QUALITY ASSURANCE CORRECTIVE ACTION FORM

1. Personnel identifying problem: _____

Nature of problem: _____

_____

_____

_____

_____

Signature _____ Date _____

2. Personnel determining corrective action: _____

Action to be taken: _____

_____

_____

_____

Signature _____ Date _____

3. Personnel responsible for implementing action: _____

Signature _____ Date _____

4. Personnel responsible for assuring the effectiveness of the action: _____

Action to be taken to assure effectiveness: _____

_____

_____

_____

_____

_____

Corrective action status: _____acceptable _____unacceptable

Signature _____ Date _____

**Figure C.4-1**
**QUALITY ASSURANCE CORRECTIVE ACTION FORM**

SOURCE: ESE.

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WITHIN DEPARTMENT ACTION

| QC Failure | Department Action |
|---|---|
| Tuning results for GC/MS fail criteria in Methods | Analyst re-tunes instrument |
| RPD and percent recoveries fail criteria and sample holding times have not expired | Analyst investigates problem and re-runs analyses |
| Standard curve correlation coefficient is less than 0.995 | Analyst investigates problem and re-runs curve and samples |
| Sample response falls outside calibration curve | Analyst dilutes sample into range of curve |

OUTSIDE DEPARTMENT ACTION

| QC Failure | Department Action |
|---|---|
| Holding times are exceeded | Notify Project Manager and QA Supervisor; resampling may be necessary |
| RPD and percent recoveries fail criteria and sample holding times have expired | Notify Project Manager and QA Supervisor; resampling may be necessary if a significant number of QA failures occur |

Corrective actions may be initiated for each measurement system (individual disciplines), by department managers, or other responsible individuals such as the Laboratory Coordinators or Laboratory Manager. On occasion, corrective actions will also be initiated at the request of the client. The QA Supervisor, will be responsible for approving the corrective action in the same fashion as if it had been initiated as a laboratory QA function.

## d. QUALITY ASSURANCE/QUALITY CONTROL PLAN

### 1. ESE LABORATORY QUALITY ASSURANCE/QUALITY CONTROL PLAN

A copy of the ESE Comprehensive Quality Assurance Plan (#860054-G) can be submitted, if required, under separate cover.

### 2. QUALITY ASSURANCE AS IT RELATES TO THE MANAGEMENT APPROACH

ESE subscribes to the Total Quality Management philosophy and has trained its staff in the principals of quality improvement and meeting client requirements.

It is the policy of ESE to maintain an active QA/QC program to provide analytical data of known and supportable quality, and ensure a high professional standard in analytical data generated in support of our customers' projects.

Each of ESE's laboratories operates under documented Laboratory Quality Assurance Plans approved by the various state and federal agencies for which the laboratories perform analytical services.

Within ESE's laboratory structure, QC activities are practiced and enforced by analysts, their immediate supervisors, laboratory project managers, and laboratory management. QC activities are the checks performed on a routine basis to ensure and document the quality of generated data. QA is comprised of those periodic audits and system/performance checks which ensure appropriate QC is being practiced. A regular system of QA audits and corrective actions is routinely employed in the laboratories to ensure data integrity.

ESE's laboratories utilize approved EPA and AEC procedures and methods for the analysis of environmental and hazardous waste samples. The exact methodologies utilized are specified in standard operating procedures and are selected based on the regulatory requirements of the client and/or projects (e.g., AEC, RCRA, Superfund,

NPDES, Industrial Hygiene), the ultimate purpose of the analytical data and its qualitative and quantitative aspects, and the required sensitivity of the analysis. For hazardous waste samples, AEC, SW-846 methods/QC or EPA/CLP protocols are observed. AEC validated methods (when required), EPA SW 846 series methods for evaluating solid wastes, EPA-600 series methods for wastewater, EPA-500 series for drinking water, and NIOSH methods for industrial hygiene surveys are also utilized. ESE's laboratories also have proven capabilities to incorporate and utilize client-specific methods.

QC samples analyzed with most sample lots include sample matrix spikes, standard matrix (control) spikes, and method blanks. Sample matrix replicates, standard matrix (control) spike replicates, post-digestion spikes, and surrogates are run as appropriate to the methodology or as otherwise required. Field QC samples, including equipment blanks, trip blanks, and field duplicates, are also included as required or specified for each task.

ESE laboratories are thoroughly familiar with the EPA and AEC requirements for the production of high-quality legally defensible data. These requirements include the responsibility and procedures necessary for chemical contamination investigative and remedial activities to assure that the analytical data obtained is of sufficient quality to meet the intended usages with each project. The QA Program philosophy and requirements as defined by EPA and AEC are clearly understood and have been successfully implemented by ESE on numerous projects since the late 1970's.

ESE understands that the analyses of samples from Picatinny Arsenal will require the implementation of various quality control procedures in order that all the requirements of this project are achieved. The TCL/TAL list of organics and inorganics will follow the methods and QC procedures as outlined in the appropriate AEC validated methods or the current CLP SOW, dioxins/furans will adhere to the methodology in DFLM 1.0, herbicides, explosives, and radiochemistry will follow the methods and QC required in SW 846 or AEC methods, water quality parameters will follow EPA

or AEC methods as appropriate, while chemical agent degradation products will follow the method and QC procedures as outlined in the respective AEC methods. All data will be high quality legally defensible data that can be accepted into the AEC IRDMIS data system.

## e. DATA MANAGEMENT

### 1. DATA MANAGEMENT, SAMPLE TRACKING, AND REPORTING

ESE's laboratories are equipped with CLASS™, which is an automated laboratory information management system (LIMS) that integrates information from sample collection, laboratory analyses, and QC requirements and calculates, checks, stores, and reports data in many different formats. The CLASS™ system resides on a Novell S-net personal computer (PC) network in each laboratory and connects more than 150 PCs in all ESE network laboratories. The CLASS™ system has the ability to combine analytical results, detection limits, QC, and site-specific data in data reports in a variety of formats for our customers. By linking all laboratories through CLASS™, ESE is able to shift workload assignments, transmit data reports, and track samples throughout its laboratory network system. Special features and capabilities of the CLASS™ system employed by ESE laboratories include:

- Automated sample log-in and tracking of samples throughout the laboratories;
- Sample holding time tracking system with manual follow-up;
- Automatic analyst notification of sample arrival and QC requirements;
- Interfacing of laboratory instruments such as AAS, GC/MS, and ICP to allow direct data transfer from instrument PCs to the CLASS™ system, thereby eliminating transcription errors and expediting data processing;
- Ability to transfer standard data files electronically via telephone modems and commercial software packages to link with client database systems;
- Specialized data report formats including Lotus 123® or dBASE® based outputs; and
- Complete QA/QC and chain-of-custody information outputs.

Our LIMS can produce AEC chemical data Transfer Files (TRN) directly from our data system without time consuming and error prone coding forms. The TRN files

can be transferred via modem to the AEC database manager for efficient manipulation of data.

AEC QC procedures have been integrated into ESE's CLASS™ system. The data system manages the flow of samples through the laboratory from sample arrival through final reporting. Prior to sampling, the Department Manager provides information on the number of samples, parameters to be analyzed, and sample arrival. This information is used to print logsheets, and sample labels. After sample collection, samples are returned to the laboratory accompanied by the logsheet with date and time of sample collection indicated so that holding times can be monitored and met. Samples are logged into the data management system by data management personnel, and laboratory analysts are notified of sample arrival, holding times, and Army Lot designations. Analysts then use the computer to reserve samples for analysis and to interactively check calibration curves, QC, and sample data. Immediate notification to the analyst of QC problems results; so that any necessary corrective action can be taken before more analyses are completed. When the analysts have entered the QC and sample data, the Army Lot, including strip charts, worksheets, and any other pertinent documentation, is turned into the Data Management Department. A program is run to achieve the following objectives:

- Check QC information such as precision and accuracy against documented data on control charts;
- Update sample records with final approved data; and
- Generate a data file formatted to AEC data reporting specifications containing field, sample, QC, analysis, and certification or performance standards data. The file is submitted to the local IRDMIS for data-checking, formatting, and transmittal to ICF KE. The way in which chemical data will be handled for this project is shown in Figure e.1-1. The files will be transferred via telephone line to the UNIX BBS for further checking and elevation (not later than 40 days after sample

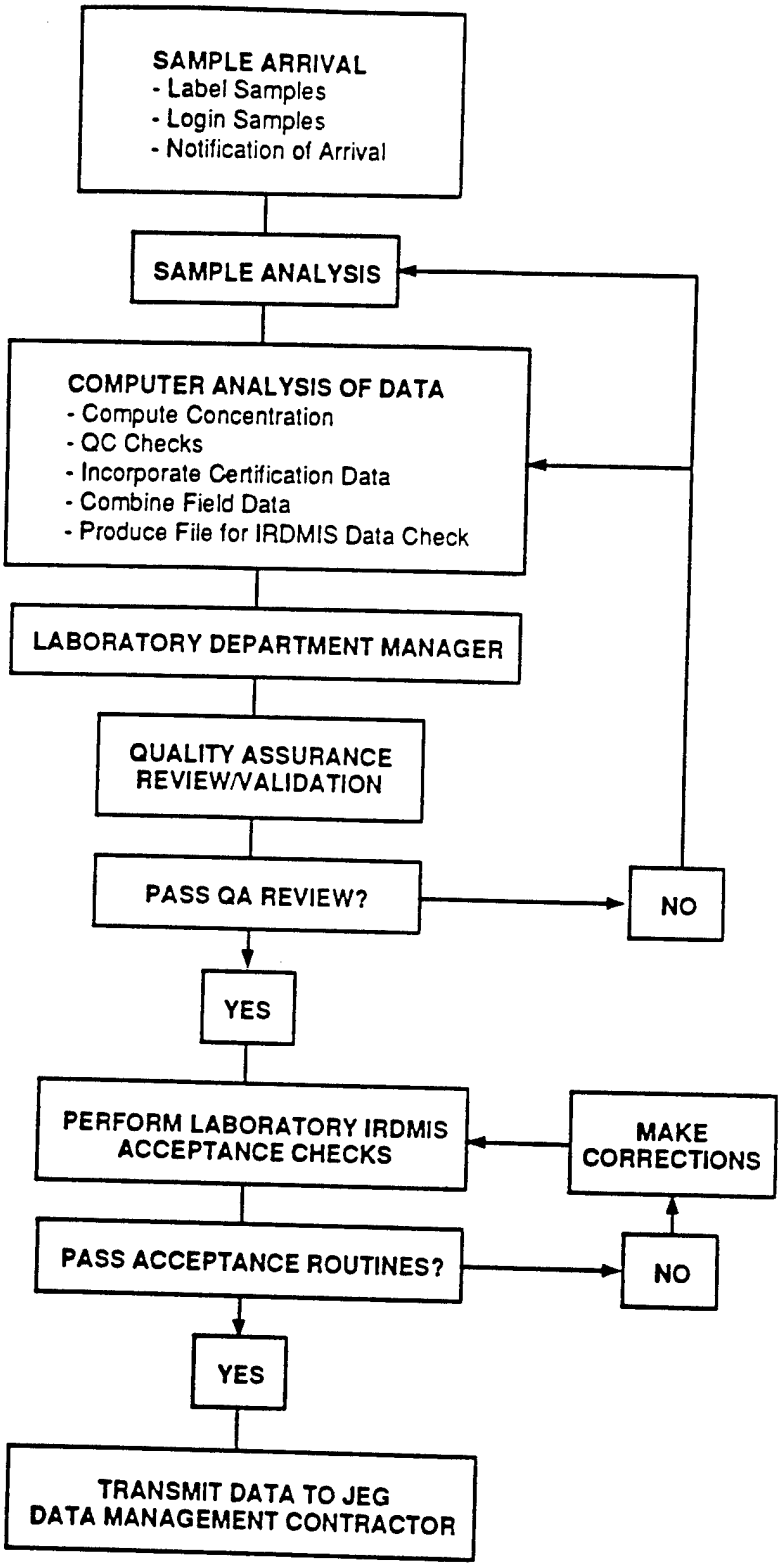


**Figure e.1-1**
**CHEMICAL DATA FLOW DIAGRAM**

SOURCE: ESE.

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

collection) to Levels 2 and 3. ESE will access Level 3 data for verification of correctness and for use in RI/FS reports.

## 2. DATA MANAGEMENT APPROACH

All laboratory data is managed using ESE's CLASS™ system. The QC reporting requirements for each type of EPA or AEC method and contract are built into the system and are automatically checked with every analytical data batch. The data system managed the flow of samples through the laboratory. Prior to sampling, the Project Manager provides information on the number of samples, site identifications, parameters to be analyzed, and estimated collection date. This information is entered into CLASS™ and is used to produce samples labels and chain-of-custody logsheets. A unique ESE number is assigned to each sample, and labels with that number and site identification are placed on each container for that sample.

At each site, samples are collected and placed in the appropriate containers. The logsheet is marked by indicating the analysis requested; noting the collection date and time; and recording the sample type, site type, sample depth, field sample number, and sampling technique.

After collection, samples accompanied by the logsheet are sent to the laboratory and received by the sample custodian. The custodian checks the sample containers against the information recorded on the logsheet and notes any discrepancies. The custodian submits the logsheet to data management personnel, who log the samples into the CLASS™ system. Sample collection date and time are entered so that holding times can be readily monitored.

ESE uses a combination of EPA Storage and Retrieval (STORET) numbers and method codes to designate parameters required for analysis. For example, benzene in water would be denoted by 34030 * 8240, since the STORET number for benzene is 34030 and the EPA method code is 8240. A list of all required analyses is logged into the computer with each sample. Each STORET-method combination has its own

QC requirements specific to that analysis. These requirements include extraction and analytical holding times, significant figures, decimal places, and spikes required.

Each list of required STORETs that is set up on the system includes the following data entries to facilitate converting data into the IRDMIS format.

| ESE and EPA Storet Numbers | Parameters |
|---|---|
| 71999 | Sample Type |
| 99759 | Site Type |
| 72015 | Sample Depth (ft) |
| 72005 | Sample Technique |
| 99720 | Installation Code |
| 29 | Field Sample Number |

The information is taken from the field log sheet and entered into CLASS™ so that it can be incorporated into the chemical data record.

The sampling information is entered into the computer to activate the parameter list for the samples collected and received by the laboratory. A notice of sample arrival and short holding times is immediately sent to the analytical team leaders. A report (available numbers) of samples requiring each analysis with the number of days left before the holding time is exceeded is distributed daily to all analytical departments. The information also can be assessed readily from CLASS™ by the Project Manager or any analyst in the Chemistry Division.

## 2.1 SAMPLE ANALYSIS AND IDENTIFICATION

ESE uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a batch of samples, the analyst will designate a specified group of samples in the computer and the sample-parameter status will be updated to IL (in laboratory). The basis for this grouping technique is that samples are analyzed in a group for the same method that can be extracted or analyzed (whichever is the most limiting factor) in a 24-hour period. The analytical batch is assigned a unique

batch control number, which is stored with all final data, so data can be checked and original documentation can be retrieved. For AEC projects, a 3 or 4-character alphabetical lot designation is also assigned to each analytical batch. Each sample in the lot is assigned a numerical suffix, starting with 001, to the 3 or 4-character alphabetical lot prefix. Using this scheme, there will be a direct relationship between ESE's unique sample number and the ICF KE sample identification for each analytical lot.

## 2.2 REQUIREMENTS FOR SAMPLE ANALYSIS AND DATA INTERPRETATION

Analytical data is entered into CLASS™ using either direct interfacing with instrumentation (such as GC, GC/MS, HPLC) or manually for other analyses. Regressions are calculated and curve correlation coefficients are tested for acceptability for each parameter. The consistency of the calibration response is checked by comparing pre-analysis and post analysis standard responses. Method blank and control spike information is then entered and results are calculated and checked against control limits as calculated for that method by certification and prior lots. Sample responses are entered into the batch, and final concentrations are calculated for each sample.

If the data fail the QC checks, appropriate actions (such as reanalyzing the samples or correcting transcription or data reduction errors) are taken.

Analysts use the CLASS™ computer to reserve samples for analysis and to interactively check calibration curves and QC results. By allowing the analyst to enter data directly and check QC and sample results, the analyst is notified immediately of QC problems. Thus, any necessary corrective actions can be taken before more analyses are completed. The CLASS™ system produces a hard copy batch/lot summary report listing raw data entry, curve values, QC results, and final sample concentrations

When the analyst has checked the QC and sample data, the Army lot, including worksheets and any other pertinent documentation (such as chromatograms), is placed in a file folder and submitted to the Data Management Department. The batch is then processed to verify QC results and finalize data in the CLASS™ system. By incorporating chemical, field, and certification data, the computer program produces a file formatted so that it can be read into and checked by the IRDMIS data checking routines. This file printout, along with the data package from the laboratory analyst, are placed in a folder and attached to the Army data transmittal form for review approval by the Department Manager, Project Manager, and QA Supervisor.

Data management personnel enter the formatted file into the IRDMIS data acceptance routines for record and group checking. A record check verifies that:

1. Data are correctly formatted,
2. The laboratory is certified for the method,
3. Test names are valid for the method,
4. Concentrations are within certified range or property diluted within range, and
5. Extraction and analysis holding times are met.

A group check verifies that:

1. A lot contains the correct number of QC samples,
2. All analyses for the method are present, and
3. All sites in a lot have a map record in the database.

When all checks are run, a transfer file is created from the system and a hard copy is placed in the lot folder along with the results of group and record checks. Chemical data are electronically reported within 45 days after sampling.

The lot folder will be reviewed by the Department Manager and Project Manager and validated by the QC Supervisor by checking selected data points against raw data. When review and validation are complete, the transfer file will be submitted to ICF KE/AEC.

## 2.3 DATA TRACKING

ESE understands the importance of maintaining task order analysis schedules. Data generated in a timely manner are critical to decision making concerning remedial actions and also for meeting regulatory reporting deadlines on monitoring efforts. With the expected multiple task awards on this contract, each with a variable number of samples, a coordinated functional mechanism for maintaining analytical data reporting schedules is critically important. Samples must be analyzed on time, with no reruns for missed holding times.

For this contract, ESE will implement tested mechanisms to accomplish the following tasks:

- Notify Project Managers and Department Managers of the arrival of samples for analysis in a timely manner;
- Track the status of samples through the laboratory analysis and review procedures;
- Monitor compliance with sample holding times via a computerized tracking procedure; and
- Communicate delays in the sample analysis and/or review chain to project management in a timely manner to implement effective corrective actions.

## Available Numbers Report

As part of ESE's CLASS™, each Department Manager receives a daily listing of all samples requiring analysis by a particular method. Samples appear on this report within 24 hours of receipt in the laboratory and after login to CLASS™. In addition to providing notification of sample availability for analysis, the available sample numbers report also provides the Department Manager and analyst a list of the number of days left in order to meet the required holding times for the analysis and the number of days before the data are due.

In addition, the ESE laboratory has a full-time person designated as the Sample Tracking Assistant (STA) whose responsibilities include the daily checking of the status of samples which are nearing holding times for extraction and/or analysis. If a sample has only 1 day (3 days if received on a weekend) left before holding time will be exceeded, the STA notifies the Department Manager in writing. Follow-up and corrective action may then be instituted.

## Chemtrak

AEC lots are tracked through the analytical laboratory and data review chain using a PC-based computer program in CLASS™ called Chemtrak. This program was especially written for AEC projects by ESE to provide a management tool for Project Managers to identify problem areas causing schedule slippage. The program functions as follows:

- As soon as lot assignments are made, the following information is entered into the computer: lot number, analytical method, installation, and date of earliest sample in the lot;

- From that information, the program calculates the date the completed analytical lot is due from the laboratory to the Data Management Supervisor for processing and the dates due for completion of supervisory

and QA review based on the required number of days for the data to be through the IRDMIS data checking routine and submitted to ICF KE/AEC;

- Weekly reports are produced by method and by installation to indicate the status of lots not completely through the system. These reports are circulated to Analytical Department Managers, Project Managers, and QA staff; and

- Daily reports are produced by project or task to indicate the status of all lots for a particular data set in order to monitor status of lots, submittal of QC charts, and completeness of task.

## 2.4 DATA REPORTS

The laboratory will submit the following reports to ICF KE:

1. Monthly Status Reports--The monthly report will be submitted by the 7th day of each month for the previous month's efforts and will contain the following information:
   a. Status as to the work accomplished and to be performed;
   b. Chronological listing of all samples received, analyzed, and in process;
   c. Difficulties or problems encountered;
   d. Significant issues or concerns; and
   e. The laboratory's ability to meet present and forecasted capacities.

2. Weekly Performance Status Reports--The weekly report will be submitted by Friday for the previous week's efforts and will contain the following items:
   a. ID of COC forms for samples in batch;
   b. Date and total number of samples received for each COC;
   c. Number of samples on which each analysis will be performed;

d. Work completed on the samples for each test (i.e., number of samples prepared/extracted, analyzed, and date each was last performed);
e. Critical out-of-control QA/QC occurrences for each test (e.g., missed holding times, lost sample, etc.) and number of samples affected by each occurrence;
f. Estimated batch completion data for each test;
g. Updates for batches received in previous weeks which are still active (i.e, in progress) during the reporting week;
h. Status of all outstanding electronic deliverables and estimated completion; and
i. Copies of COC forms.

## 2.5 LABORATORY ANALYTICAL DATA REPORT

Hard copies of the analytical data will be submitted within 30 days of receipt of samples and electronic copies within 45 days of receipt of samples.

Hardcopy results shall include standard CLP Level IV packages for CLP methods and CLP-like packages for other methods including drinking water, SW-846 and AEC methods. Excluded are raw data, chromatograms, run logs, and supporting data which shall be maintained in project-specific files for future use including possible onsite audits and Level IV data validation. All pages shall be sequentially numbered.

Electronic results will include an "error-free" IRDMIS data file which will be electronically transmitted to Potomac Research Institute (PRI) along with an MS-DOS formatted diskette.

## 3. EXPERIENCE WITH AEC IRDMIS

ESE's laboratories have been utilizing the IRDMIs PC Data Entry and Validation Subsystem for loading and validation of data since 1979. ESE has had experience with more than 60 projects that have required the use of IRDMIS. Many of these efforts can be described as multimedia projects, conducted during multiple years,

involving the analysis of large numbers of samples for full scope. Some of the larger projects include Rocky Mountain Arsenal, Letterkenny AD, Joliet AAP, Umatilla Depot Activity, Aberdeen Proving Ground, and Alabama AAP.

The data manager for this project is Virginia O'Brien, Manager, Gainesville Laboratory Information Services. Ms. O'Brien has 15 years of experience in the use of IRDMIS and has served as task manager/project coordinator for data management for the projects described above.

ESE has developed standard operating procedures (SOPs) for using IRDMIS. These SOPs are included in Appendix B. The laboratory shall follow these SOPs to load and validate data through IRDMIS. The laboratory will print and review the data from this system and forward an error-free EDD and printout via mail to ICF KE within 45 days of receipt of samples. Upon authorization from the ICF KE Project Manager, the laboratory will transmit the electronic data directly to PRI via modem.

## f. HEALTH AND SAFETY

### 1. ESE HEALTH AND SAFETY PLAN

A copy of the ESE Health and Safety Plan can be provided, if required, under separate cover.

### 2. HEALTH AND SAFETY RECORD OF ESE'S LABORATORY

ESE adheres to a broad-based health and safety program for all its employees, in order to protect them from workplace-related illnesses or injuries, as well as to be in compliance with applicable health and safety regulations. The ESE Health and Safety Manual in conjunction with the Chemistry Department Health and Safety Manual are designed to outline OSHA requirements and to help maintain safe working environments.

The following table summarizes the health and safety record for the Gainesville laboratory. This information was gathered from the OSHA 200 logs kept at the Gainesville laboratory for the period of January 1, 1993, through August 31, 1994.

Gainesville Laboratory Health/Safety Record

| OSHA 200 INFORMATION | 1993 | 1994 |
|---|---|---|
| Number of injury-related fatalities | 0 | 0 |
| Number of injuries with lost/restricted workdays | 1 | 2 |
| Number of injuries without lost/restricted workdays | 9 | 3 |
| Number of illness-related fatalities | 0 | 0 |
| Number of illnesses with lost/restricted workdays | 0 | 0 |
| Number of illnesses without lost/restricted workdays | 0 | 0 |
| Total number of injuries and illnesses | 10 | 5 |
| Total number of employee hours worked | * | * |
| Total restricted days | 7 | 0 |
| Total days away from work | 1 | 7 |

*Information not known at this time.

Source: ESE.

## 3. AGREEMENT TO FOLLOW ICF KE'S HEALTH AND SAFETY PLAN

ESE certifies that it will follow the procedures set forth in the ICF KE H&S plan if required.

CERTIFICATE OF INSURANCE

# ACORD. CERTIFICATE OF INSURANCE

ISSUE DATE (MM/DD/YY) 03/15/94

PRODUCER

JOHNSON & HIGGINS OF ILLINOIS, INC.
500 WEST MADISON STREET, SUITE 2100
CHICAGO IL 60661-2595

THIS CERTIFICATE IS ISSUED AS A MATTER OF INFORMATION ONLY AND CONFERS NO RIGHTS UPON THE CERTIFICATE HOLDER. THIS CERTIFICATE DOES NOT AMEND, EXTEND OR ALTER THE COVERAGE AFFORDED BY THE POLICIES BELOW.

INSURED

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
ATTN JIM BOURAZEK
8901 N. INDUSTRIAL RD.
PEORIA, IL 61615

## COMPANIES AFFORDING COVERAGE

| | | | |
|---|---|---|---|
| COMPANY LETTER | A | ILLINOIS NATIONAL INS. CO. | Rating: A++ Class: XV |
| COMPANY LETTER | B | NATIONAL UNION FIRE INS. CO. | Rating: A++ Class: XV |
| COMPANY LETTER | C | PLANET INS. CO. | Rating: A- Class: XI |
| COMPANY LETTER | D | N/A | |
| COMPANY LETTER | E | | |

## COVERAGES

THIS IS TO CERTIFY THAT THE POLICIES OF INSURANCE LISTED BELOW HAVE BEEN ISSUED TO THE INSURED NAMED ABOVE FOR THE POLICY PERIOD INDICATED. NOTWITHSTANDING ANY REQUIREMENT, TERM OR CONDITION OF ANY CONTRACT OR OTHER DOCUMENT WITH RESPECT TO WHICH THIS CERTIFICATE MAY BE ISSUED OR MAY PERTAIN. THE INSURANCE AFFORDED BY THE POLICIES DESCRIBED HEREIN IS SUBJECT TO ALL THE TERMS, EXCLUSIONS AND CONDITIONS OF SUCH POLICIES. LIMITS SHOWN MAY HAVE BEEN REDUCED BY PAID CLAIMS.

| CO LTR | TYPE OF INSURANCE | POLICY NUMBER | POLICY EFFECTIVE DATE (MM/DD/YY) | POLICY EXPIRATION DATE (MM/DD/YY) | LIMITS | | |
|---|---|---|---|---|---|---|---|
| A | GENERAL LIABILITY<br>X COMMERCIAL GENERAL LIABILITY<br>CLAIMS MADE [X] OCCUR<br>OWNER'S & CONTRACTOR'S PROT | GL3404599 | 3/16/94 | 4/01/95 | GENERAL AGGREGATE | $ | 1,000,000 |
| | | | | | PRODUCTS-COMP/OP AGG. | $ | 1,000,000 |
| | | | | | PERSONAL & ADV. INJURY | $ | 1,000,000 |
| | | | | | EACH OCCURRENCE | $ | 1,000,000 |
| | | | | | FIRE DAMAGE (Any one fire) | $ | 50,000 |
| | | | | | MED. EXPENSE (Any one person) | $ | 5,000 |
| B | AUTOMOBILE LIABILITY<br>X ANY AUTO<br>X ALL OWNED AUTOS<br>SCHEDULED AUTOS<br>HIRED AUTOS<br>X NON-OWNED AUTOS<br>GARAGE LIABILITY | CA1188525 | 3/16/94 | 4/01/95 | COMBINED SINGLE LIMIT | $ | 1,000,000 |
| | | | | | BODILY INJURY (Per person) | $ | |
| | | | | | BODILY INJURY (Per accident) | $ | |
| | | | | | PROPERTY DAMAGE | $ | |
| B | EXCESS LIABILITY<br>X UMBRELLA FORM<br>OTHER THAN UMBRELLA FORM | BE3090994 | 3/16/94 | 4/01/95 | EACH OCCURRENCE | $ | 14,000,000 |
| | | | | | AGGREGATE | $ | 14,000,000 |
| C | WORKER'S COMPENSATION AND EMPLOYERS' LIABILITY | NWA010257702<br>NWA010263802 (CA) | 3/16/94<br>3/16/94 | 4/01/95<br>4/01/95 | X STATUTORY LIMITS | | |
| | | | | | EACH ACCIDENT | $ | 500,000 |
| | | | | | DISEASE-POLICY LIMIT | $ | 500,000 |
| | | | | | DISEASE-EACH EMPLOYEE | $ | 500,000 |
| A | OTHER<br>PROFESSIONAL/<br>POLLUTION LIABILITY | C72961 | 2/23/94 | 4/01/95 | $3,000,000 EACH CLAIM<br>$3,000,000 AGGREGATE | | |

DESCRIPTION OF OPERATIONS/LOCATIONS/VEHICLES/SPECIAL ITEMS FOR PROPOSAL PURPOSES ONLY.

CERTIFICATE HOLDER

PROPOSAL CERTIFICATE

CANCELLATION

SHOULD ANY OF THE ABOVE DESCRIBED POLICIES BE CANCELLED BEFORE THE EXPIRATION DATE THEREOF, THE ISSUING COMPANY WILL ENDEAVOR TO MAIL 30 DAYS WRITTEN NOTICE TO THE CERTIFICATE HOLDER NAMED TO THE LEFT, BUT FAILURE TO MAIL SUCH NOTICE SHALL IMPOSE NO OBLIGATION OR LIABILITY OF ANY KIND UPON THE COMPANY, ITS AGENTS OR REPRESENTATIVES.

AUTHORIZED REPRESENTATIVE

Donald J. Price

DONALD J. PRICE/KG

ACORD 25-S (7/90)

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First of America Bank - Illinois, N.A.
301 S.W. Adams Street
Peoria, Illinois 61652-0749
Telephone 309-655-5000

FIRST OF AMERICA

March 16, 1993

To Whom It May Concern:

Environmental Science and Engineering, Inc. is a valued customer of First of America Bank-Illinois. In our experience with the company, they have handled all accounts in a satisfactory manner. The company maintains several depository accounts with First of America Bank-Illinois, with aggregate balances averaging in low seven figures. Credit commitments in low eight figures have been made available to the company, with usage in low seven figures at this time.

We have found Environmental Science and Engineering, Inc. to be a well managed and well capitalized business. First of America Bank-Illinois has had occasion to utilize the services provided by Environmental Science and Engineering, Inc., and has been pleased with their service and professionalism.

If you have any further questions, please do not hesitate to call me at (309) 655-5479.

Sincerely,

Timothy T. Fogerty
Vice President

# APPENDIX A

# RESUMES OF KEY PERSONNEL

JOHN J. MOUSA, Ph.D.
LABORATORY DIRECTOR

## AREAS OF SPECIALIZATION

Laboratory Management, Environmental Analytical Chemistry, Environmental Project Management, Trace Organic Residue Analysis, Environmental Contamination Assessment, Quality Assurance, Industrial Hygiene Analyses, Laboratory Design, Environmental and Hazardous Waste Chemicals Analysis, Fluorescence and Phosphorescence Analytical Techniques, Gas-Liquid Chromatography, High Pressure Liquid Chromatography, Hazardous Waste Safety

## EXPERIENCE

Dr. Mousa has 22 years of experience in managing and conducting environmental analytical efforts for industrial and governmental clients. He serves as director of 3 major environmental and industrial hygiene laboratories with a staff of 250 chemists and technicians involved in chemical analysis of environmental and hazardous waste samples for inorganic and organic parameters, asbestos, radiochemistry, and in methods development and applied research and development. His recent experience follows:

**Rocky Mountain Arsenal Contamination Assessment, U.S. Army Environmental Center (formerly USATHAMA), Laboratory Director**--Supervised and directed over $12 million of laboratory analysis for a large, multidisciplinary task order contract for the Program Manager's Office, Rocky Mountain Arsenal, Colorado. The project value was in excess of $30 million and involved 16 separate tasks. Remedial investigation tasks include contamination studies of the Arsenal's soil, groundwater, surface water, air, and biota.

**Remedial Investigation/Feasibility Study (RI/FS), Webb Air Force Base (AFB) and Schilling AFB, U.S. Army Corps of Engineers (USACE), Kansas City District, Laboratory Director**--Overall director of chemical analysis for organics, inorganics and metals.

**RI/FS Activities, Maxwell AFB, Patrick AFB, Hickam AFB, Dobbins AFB, Cape Canaveral AFB, and Keesler AFB, U.S. Air Force Occupational and Environmental Health Laboratories (USAFOEHL), Laboratory Director**--Provided corporate direction and support to project team and interacted with client to assure good performance on multimillion dollar environmental analyses projects.

**RI/FS at Multiple USAF Facilities, USAF Occupational and Environmental Health Laboratories, Brooks AFB, Texas, Laboratory Director**--Directed laboratory activities for a multi-year contract with the Air Force to conduct RI/FSs in accordance with CERCLA and SARA. ESE conducted these programs for over 12 Air Force bases. The analytical support provided by ESE's chemistry laboratory involved the analysis of over 4,000 samples according to the latest EPA SW846 and 600-series methods protocols for volatile and semi-volatile organics, petroleum hydrocarbons, heavy metals including leach and pesticides and PCB's.

**Total Quality Management, Instructor**--Served as course instructor for Quality Education System course for over 100 environmental professionals. Course covered four absolutes of quality and problem solving and identifying customer requirements.

**Corporate Manager, Quality Assurance/Safety**--Supervised Quality Assurance staff involved in preparation of Project Quality Assurance Plans, auditing departmental quality control, implementation of group and department level QC programs for sampling and analysis, and provision of independent peer review system of all reports generated within ESE. Duties involved interaction with clients and regulatory agencies to resolve quality control problems associated with analytical chemistry data.

## EDUCATION

Ph.D. Analytical Chemistry, University of Florida, 1973
B.S. Chemistry, University of Houston, 1970

## REGISTRATIONS AND AFFILIATIONS

Phi Beta Kappa
Phi Kappa Phi
American Chemical Society
American Industrial Hygiene Association

PAUL C. GEISZLER, M.S.
PROJECT DIRECTOR

## AREAS OF SPECIALIZATION

Hazardous Waste Analysis, Environmental Chemistry, Gas Chromatography/Mass Spectrometry, Remedial Investigation

## EXPERIENCE

Mr. Geiszler has more than 19 years of experience in environmental chemistry. He is responsible for the project management of all analytical tasks and compliance with program requirements. He has directed the analytical portion of projects under several federal programs including U.S. Army Environmental Center (AEC, formerly USATHAMA), U.S. Army Corps of Engineers (USACE), U.S. Navy, U.S. Air Force (USAF), U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) and REM III, and HAZWRAP. Relevant projects are provided below.

**Contamination Evaluation, Installation Restoration Program (IRP), Phase II, USACE, Mobile District, U.S. Naval Station, Lee Field and Turner Air Force Base**--Sampling and analysis in accordance of CE QA/QC requirements of soil, sediment, groundwater and surface water samples to determine presence or absence of chemical contamination. Analysis included priority pollutant volatiles, acid/base/neutral extractables, metals, pesticides and PCBs, and petroleum hydrocarbons.

**Remedial Investigation/IRP, Former Webb Air Force Base (AFB), Big Springs, Texas, USACE, Kansas City District, Analytical Services Project Director**--Sampling and analysis was performed in accordance with the USACE QA/QC requirements to define nature and extent of contamination in four study areas. Several hundred soil samples from each area were collected and analyzed for heavy metals, cyanide, polynuclear aromatics, TCL pesticides and PCBs, TCL volatiles and extractables, and identification of non-target compounds. Soil gas effort involved analysis of 150 samples for benzene, toluene, n-pentane, n-hexane, and trichloroethane.

**Remedial Investigations/Feasibility Studies (RI/FS) at Multiple U.S. Air Force (USAF) Facilities, USAF Occupational and Environmental Health Laboratories (USAFOEHL), Brooks AFB, Texas, Chemistry Program Manager**--ESE has a multi-year contract with the Air Force to conduct RIFSs in accordance with CERCLA and SARA. ESE is conducting these programs for the following Air Force bases:

Keesler AFB, Mississippi
Dobbins AFB, Georgia
Patrick AFB, Florida
Plant 78, Utah
Maxwell AFB, Alabama
Eileson AFB, Alaska
Hickam AFB, Hawaii
Tyndall AFB, Florida
Cape Canaveral AFS, Florida

The analytical support provided by ESE's analytical services laboratory involved the analysis of over 4,000 samples according to the latest EPA SW846 and 600-series methods protocols for volatile and semi-volatile organics, petroleum hydrocarbons, heavy metals (including leach and pesticides), and PCB's. Strict QA/QC protocols were followed in these analyses which were conducted under an approved QA project plan. ESE's laboratory data was audited extensively by the USAFOEHL for this work.

**Confirmation Surveys, USAFOEHL IRP, Brooks AFB, Texas, Chemistry Program Manager**--Task order contract to design and implement IRP Phase II Confirmation Surveys for OEHL. Studies include assessment of groundwater and surface water with toxic and hazardous substances.

ESE performed a confirmation study at Cape Canaveral Air Force Station and Patrick Air Force Base, Florida, to confirm the existence of potential contaminants at 43 former disposal and storage sites identified in the Phase I record search. Ten sites at Cape Canaveral AFS and 13 sites at Patrick AFB were investigated. These sites include 10 chemical disposal areas, eight landfills, a fuel spill area, two potential PCB-contaminated sites, and four firefighter training areas.

Geophysical surveys were conducted at several disposal areas, and groundwater monitor wells were installed and sampled. Surface waters, groundwaters, soil borings, and sediments were sampled and analyzed for various screening parameters, including total organic carbon (TOC), total organic halogens (TOX), phenols, oil and grease, pH, specific conductance, PCBs, and selected metals and pesticides.

## EDUCATION

M.S. Chemistry/Biology, University of Southern Mississippi, 1976
B.S. Biology/Chemistry, Southern Illinois University, 1972

## REGISTRATIONS AND AFFILIATIONS

American Chemical Society
American Society of Mass Spectrometry

DAVID H. GREER, JR., B.S.
PROJECT MANAGER

## AREAS OF SPECIALIZATION

Project Management, Analytical Chemistry, Gas Chromatography, HPLC, GC/MS, Water Quality, Environmental Assessment, Toxicity Testing

## EXPERIENCE

Mr. Greer has over 18 years of experience in environmental analytical chemistry, aquatic toxicology, water quality, environmental assessment, and project management. His experience includes analytical determinations of organic compounds in various matrices by gas chromatography (GC) utilizing a variety of detection systems, gas chromatography/mass spectrometry (GC/MS), high performance liquid chromatography (HPLC), and thin layer chromatography (TLC). Mr. Greer has performed aquatic toxicology studies using fresh and saltwater algae, fish, and invertebrates. He has managed numerous analytical tasks associated with aquatic toxicological studies in support of registration for various agrochemical and other organic compounds under FIFRA and TSCA, Good Laboratory Practices (GLP). Mr. Greer has also managed analytical projects designed to determine the effects of various organic contaminants in the environment including the determination of the magnitude of residues in water, soil/sediment, vegetation, and animal tissues.

**Project Manager**--Responsible for the planning, staffing, and coordination of projects dealing with method development, validation, and analysis of organic analytes. Other responsibilities include interpretation, documentation, and report preparation; and technical conduct of the following studies:

Environmental Sample Analysis in Support of RI/FS at Aberdeen Proving Ground, Maryland, ICF Kaiser Engineers, Inc., Abingdon, MD.

Determination of Pesticide Residues in Citrus Foliage Samples in Support of EPA Data Call-In (DCI) of September 12, 1986, Versar, Inc., RiskFocus Division, Springfield, VA.

Development of Standard Analytical methods for Organic Compounds and Metals, USATHAMA, Aberdeen, Maryland.

Determination of Ethylene Dibromide (EDB) in Soil and Water by GC/ECD in Support of Soil Transport Studies, Huey, Guilday, Kuersteiner and Tucker, PA, Tallahassee, Florida.

Method Development, Validation and Analysis of Proprietary Cationic Compounds in Water, Soil and Sediments by HPLC, The Procter & Gamble Company, Cincinnati, Ohio.

Determination of Aldicarb and Metabolites in Water by Direct Injection HPLC Post Column Derivitization and Fluorescence Detection, Technical Services, Inc., Jacksonville, Florida.

Determination of Proprietary Anionic Compounds by GC/MS, The Proctor & Gamble Company, Cincinnati, Ohio.

Determination of Butyltin Compounds in Water in Support of Environmental Testing, Envirosphere Company, Bellview, Washington.

Determination of Fenamiphos (Nemacur) in Water, Soil and Fish Tissue, Nutri-Turf Inc., Jacksonville, Florida.

Determination of Carbaryl and Alpha-Naphthol by HPLC in Support of Development of an Aquatic Toxicity Test using Freshwater Mollusks, KBN Engineering and Applied Science, Gainesville, Florida.

Determination of Various Pesticide Residues in Support of Aquatic Toxicity Tests for New Compound Registration, Rhone-Poulenc Ag Company, Monmouth Junction, New Jersey.

Determination of Thiobencarb by GC/NPD in Support of Aquatic Toxicity Studies using Freshwater Mussels, Young-Morgan and Associates, Inc., Franklin, Tennessee.

## EDUCATION

B.S. Biology/Pre-Professional Sciences, University of West Florida, 1975

## REGISTRATIONS AND AFFILIATIONS

American Chemical Society (ACS)
Society of Environmental Toxicology and Chemistry (SETAC)

JOE D. OWUSU-YAW, Ph.D.
QUALITY ASSURANCE

## AREAS OF SPECIALIZATION

Toxicology, Analytical Chemistry, Food Technology, and Quality Assurance (QA)

## EXPERIENCE

Dr. Owusu-Yaw has 4 years of professional experience in toxicology and quality assurance/quality control (QA/QC). His primary areas of focus include data review/validation, method development, preparation of project specific quality assurance plans, standard operating procedures (SOPs) and good laboratory practice (GLP) compliance issues and audits. As Laboratory QA Supervisor, Dr. Owusu-Yaw is responsible for laboratory and field QA procedures, audits, documentation, certification, and system performance samples. He writes, reviews and edits QA/QC reports for several projects and is responsible for teaching/training and management of personnel and budgets. His recent experience is highlighted in the following paragraphs.

**Environmental Surveys, for U.S. Army Environmental Center (formerly USATHAMA), Project QA Coordinator**--Responsible for analytical data acquisition audits for the Joliet Army Ammunition Plant, Radford Army Ammunition Plant and West Virginia Ordinance Works. Performed field sampling and QA/QC audits for the above military installations.

**Environmental Surveys for U.S. Army, Project QA Scientist**--Responsible for analytical data acquisition audits for Alabama Army Ammunition Plant (AAAP), Sharpe, Phoenix, Tobyhanna, Ft. Dix, Lonestar, Milan, Louisiana Army Ammunition Plant (LAAP), Letterkenny Army Depot, and Umatilla Army Depot.

**Environmental Assessment and Toxicology, Good Laboratory Practice Compliance Monitor**--QA supervisor responsible for in-phase, laboratory, protocols and reports audits for compliance with FIFRA, TSCA and FDA GLP regulations. Responsible for writing and reviewing SOPs, and interpreting, training and advising personnel on the good laboratory practice (GLP) regulations.

**Navy CLEAN Program Data Validation Services, Project QA Coordinator**--Responsible for CLP data review/validation for U.S. Marine Corps Logistics Base, Barstow, California, and U.S. Marine Corps Air Station El Toro Phase I RI/FS, California.

## EDUCATION

Ph.D. Food Chemistry and Toxicology, University of Florida, 1989
M.S. Food Science and Technology, University of Florida, 1984
B.S. Food Science and Biochemistry, University of Ghana, 1977

## REGISTRATIONS AND AFFILIATIONS

Institute of Food Technologists
American Chemical Society
Society of Quality Assurance

DWIGHT F. ROBERTS, B.S.
GC/MS

## AREAS OF SPECIALIZATION

GC/LC/MS, Gas Chromatography, HPLC, Organic Chemistry, Quality Control, Analytical Chemistry

## EXPERIENCE

Mr. Roberts has 19 years of analytical chemistry experience and 14 years of gas chromatography/mass spectrometry experience. Mr. Roberts has been actively involved in the analysis of air, gas, water, soil, sludges, and sediments for the last 15 years, involving duties and responsibilities that varied from a technician to a department manager. Mr. Roberts is currently responsible for GC/LC/MS analysis for volatile and semi-volatile organic compounds, supervising 17 chemists and maintaining 11 GC/MSs. Sources of samples for analytical determination have included: raw and finished drinking waters, groundwaters, surface waters, industrial effluents, hazardous waste samples, including waters, sludges, sediments, and soil, TCLP, landfills, munitions, and pesticide manufacturers, indoor and ambient air samples, and industrial proprietary methodologies. Currently responsible for GC/LC/MS environmental analytical chemistry, providing detailed results of target and non-target compounds and consultation related to source of or associated risks.

**Chemical Contamination Evaluation, Ft. McCoy Air Force Base, U.S. Corps of Engineers (USACE), Mobile District, GC/MS Manager**--Actual work being performed includes review of available records, sampling and analysis of soil and groundwater, and reports, conclusions and recommendations for future work. Responsible for GC/MS analyses. Analyses include HSL volatiles, HSL semivolatiles, organochlorine pesticides and PCBs, TRPH, and RCRA metals using EPA SW 846 Methods and adhering to strict QA/QC protocols.

**RI/FS at Multiple Air Force Installations, U.S. Air Force Occupational and Environmental Health Laboratories (USAFOEHL), GC/MS Manager**--Responsible for GC/MS analysis in support of remedial investigations at Patrick AFB, Cape Canaveral AFB, Hickam AFB, Eileson AFB, Keesler AFB, Dobbins AFB, Maxwell AFB, and Plant 78.

**Analyses of Various VOC and SOC Analytes in Drinking Water, Municipal SOC Program, Various Florida Municipalities, Sr. Associate Chemist**--Responsible for GC/MS analyses to comply with Florida Department of Environmental Regulation (FDER) requirements for VOC and SOC determinations in municipal drinking water supplies for over 50 municipalities in the State of Florida.

**Synthetic Organic Chemicals in Drinking Waters for Various Residential Municipalities, Task Manager**--Monitored drinking waters for trace levels of chemicals by capillary GC/MS per Safe Drinking Water Act.

**Contract Laboratory Program, U.S. Environmental Protection Agency (EPA) Department Manager**--Supervise and review data for solids and liquids for EPA Regions. Pass quarterly performance evaluations samples. Provide special analytical services.

**Environmental Survey Analysis for Trace Organics in Groundwaters, Soils, and Sediments from Various Army Installations, Task Manager**--Environmental monitoring of previous U.S. Army munitions facilities for chemical characterization of representative sites, VOA and SVO priority pollutants. Involved with sample preparation, screening of extracts by GC-FID, and analysis by capillary GC/MS, as well as Department Manager supervision and review.

## EDUCATION

B.S. Chemistry, University of Florida, 1973

## REGISTRATIONS AND AFFILIATIONS

American Society for Mass Spectrometry (ASMS)
American Chemical Society (ACS), Environmental Division
Certificate of Recognition, 1990 Technical Paper Award, ESE
Hazardous Materials Control Research Institute, 1992

KATHLEEN K. ALLEN, B.S.
WATER QUALITY

## AREAS OF SPECIALIZATION

Petroleum Hydrocarbon Analysis by Scanning Infrared Spectroscopy; Classical Water Quality Parameters including Demand, Titrametric, Colorimetric, Potentiometric and Gravimetric Procedures; Cyanide and Phenol Analysis by Manual and Semiautomated Color Development

## EXPERIENCE

Ms. Allen has 9 years of professional experience in the inorganic areas of environmental labs. She has done analytical analysis, as well as managerial tasks, in the areas of classical, semi-automated, metals and wastewater analysis. The following projects represent Ms. Allen's relevant experience.

**Air Toxics Analysis in Ambient Air, Rocky Mountain Arsenal (RMA), U.S. Army Environmental Center (formerly USATHAMA)**--Monitored air quality for semivolatile and volatile organics, pesticides, and metals for sites at the Rocky Mountain Arsenal. Methods were certified (USATHAMA/PMRMA) by ESE following TO-series methods (TO-1, TO-4).

**Clean Air Status and Trends Network (CASTNet), U.S. Environmental Protection Agency (EPA), Various Nationwide Locations**--ESE is responsible for coordinating all aspects of this major study to determine the effectiveness of new air quality regulations, specifically, the Clean Air Act Amendments of 1990 (CAAA). CASTNet is the largest air quality monitoring project ever awarded by EPA. ESE is managing a team of subcontractors and consultants in establishing a 375-station monitoring network across the country. Data will be collected, analyzed, and presented to EPA to satisfy the CAAA mandate for research and monitoring of the status, trends, and effects of hazardous air pollutants (HAPS), ozone, acid deposition, acid aerosols, and visibility.

**National Dry Deposition Study, EPA**--Under a 5-year contract, ESE assumed management of a 6-station prototype dry deposition network and expanded the network to a 55-station nationwide network. Weekly samples are collected to measure for aerometric concentrations of particulates and gases. Analyses are performed for: particulate sulfate, nitrate, and ammonium, gaseous nitric acid; and, sulfur dioxide. Earlier phases sampled day and night weekly samples and included analyses for base cations (Ca, Mg, Na, K). At 11 stations weekly wet deposition samples are collected and analyzed for pH, specific conductivity, acidity, cations (Ca, Mg, Na, K, $NH_4$), and anions ($SO_4$, Cl, $NO_3$, $NO_2$). ESE's laboratory is responsible for preparation and

distribution of sampling materials, sample receipt, analysis, data management, and reporting. Meteorological data are collected at each site to allow for calculation of dry deposition fluxes.

## EDUCATION

B.S. Agriculture, University of Florida, 1978

## REGISTRATIONS AND AFFILIATIONS

Florida Society of Environmental Analysts

BRADLEY A. WEICHERT, B.S.
HPLC/GC

## AREAS OF SPECIALIZATION

Gas Chromatography, High Performance Liquid Chromatography, Pesticide Analysis, Trace Organics Method Development, Air Toxics and Industrial Hygiene Analyses

## EXPERIENCE

Mr. Weichert has 13 years experience in the analysis of environmental samples. His primary duties are management of analyses by gas and liquid chromatography and methods development for organic analyses in environmental matrices under EPA, AEC/USATHAMA, and FIFRA/TSCA GLP guidelines. Recent experience follows.

**U.S. Army Environmental Center (formerly USATHAMA), Task Manager**--Responsible for managing the certification and analysis of organic compounds by GC and HPLC using USATHAMA protocols. Analyses included explosives, organochlorine pesticides and chemical warfare agent precursors.

**U.S. Navy Comprehensive Long-Term Environmental Action Navy (CLEAN) Program and U.S. Army Corps of Engineers (USACE), Task Manager**--Responsible for the management of the analysis of water and soils for fuel products at military installations in support of underground storage tank (UST) remedial investigation/feasibility studies (RI/FS) and remediation activities throughout the United States including Hawaii, Alaska and Puerto Rico. Various fuels were fingerprinted including aviation gasoline, JP-4, JP-5, Bunker C residual fuel oils and crude oil.

**Chemical Support of Toxicity Testing, United States Air Force Office of Environmental Health Laboratories (USAFOEHL), Task Manager**--Responsible for the management of the analysis of environmental samples for pesticides and herbicides using EPA methodologies including SW 846 and CLP protocols.

**National Pesticide Survey (NPS), U.S. Environmental Protection Agency (EPA), Task Manager**--Department contracted to perform analyses for carbamates by HPLC on samples from 1500 potable water supplies throughout the United States.

**EPA, Special Analytical Services, Task Manager**-Managed the effort for the analysis of sediment samples from Puget Sound for polynuclear aromatic hydrocarbons (PNA) using high performance liquid chromatography. PNAs

were quantified by both ultraviolet and fluorescence detectors. Approximately 250 samples were analyzed as part of an interlaboratory study.

**EPA-Contract Laboratory Program (CLP), Task Manager**--Managed the analysis of pesticides using CLP protocols, project quality assurance review, data package submission and technical liaison.

**American Cyanamid, Task Manager**--Managed chemical support activities by HPLC for toxicity tests for Terbufos($C^{14}$), Phorate($C^{14}$) and AC801,757 using Mysidopsus bahia, Cyprinodon variegatus and Crassostrea virginica.

**Dow Chemical Company, FMC Corp., Rhone-Polenc and NPC, Task Manager**--Responsible for management of chemistry support for various proprietary pesticides for pesticide registration toxicity tests under FIFRA GLP guidelines.

**Searle Pharmaceutical Company, Task Manager**--Managed chemical support activities for three drugs-Oxaprozin, Misoprostal and Diclofenac. Toxicity tests were performed using Daphnia Pulex, Pimephales promelas and Selanastrum capricornutum.

EDUCATION

B.S. Chemical Oceanography, Florida Institute of Technology, 1979

KATHERINE W. BARRY, B.S.
TRACE METALS

## AREAS OF SPECIALIZATION

Metals Analysis of Water, Wastewater, Soils and Sediments, Biological Materials and Ambient Air Utilizing ICP/MS, Furnace, Cold Vapor, Hydride, and Flame Spectroscopy; Analysis of Water, Soil, and Sediments, Biological Materials, and Ambient Air for Various Chemical Constituents and Characteristics; Bioassay

## EXPERIENCE

Ms. Barry has 12 years of professional experience in the environmental field primarily in analysis of water wastewater, soils, and sediments; biological materials; and ambient air. Her relevant experience is highlighted below.

**Chemical Contamination Evaluation, Former Camp Sibert, U.S. Army Corps of Engineers, Laboratory Analyst**--ESE was contracted to determine the extent of chemical contamination at the former site of Camp Sibert. Performed analysis for furnace, cold vapor, hydride and flame on groundwater and soil samples for selected metals. Analyses were performed under USATHAMA and SW846 methods, as appropriate.

**Chemical Contamination Evaluation, Ft. Pickett, U.S. Army Corps of Engineers, Laboratory Analyst**--Supervised Furnace Group analysts in determining the amount of chemical contamination in soil and groundwater at the fire training area, Ft. Pickett. Analytical requirements included analysis of the RCRA metals.

**RI/FS at Multiple USAF Facilities, Laboratory Analyst**--Scheduled and performed Furnace Group analysis on this multi-year contract with the Air Force to conduct RI/FSs at multiple USAF facilities in accordance with CERCLA and SARA. The metals data provided by the Gainesville laboratory involved the analyses of over 4,000 samples according to methods within the EPA's third edition of SW846. Strict QA/QC protocols were followed under an Air Force approved QA project plan.

**Contamination Assessment, Rocky Mountain Arsenal, U.S. Army Environmental Center (formerly USATHAMA), Laboratory Analyst**--Supervised Furnace Group Analyst during ESE's recently completed large, multidisciplinary contract for the Program Manager's Office, Rocky Mountain Arsenal, Colorado. The Gainesville Spectroscopy Department certified 16 analytical methods. The laboratory successfully provided quality data on over 2,000 environmental samples collected at RMA.

**Contract Laboratory Analytical Support Services Program (CLASS), U.S. Army, Laboratory Analyst**--Performed and supervised Furnace Group analysis on a 5 year $10 million contract as one of five laboratories participating in the CLASS Program. This project involves the analysis of environmental samples on a task order basis in support of remedial investigations. ESE is offering a full range of analytical services including ICAP, GFAA and Cold Vapor Spectroscopy. (All analyses are performed using USATHAMA certified methods.)

**Ambient Air Analysis, City of Detroit, Laboratory Analyst**--ESE performed daily ambient air monitoring for one month at several points around the city of Detroit. Select elements were analyzed from PM-10 filters by ICP and mercury was analyzed from sorbent tubes by Cold Vapor Spectroscopy.

**Acid Deposition Study for Various Networks Throughout the United States and Canada, Laboratory Analyst**--Analyzed wet, dry and bulk precipitation samples and lake samples to study the atmospheric deposition of acidity, nutrients, and minerals for networks in Florida, Ohio, Wisconsin, Idaho and Canada and for NOAA and USGS.

**Florida Acid Deposition Monitoring Program, Florida Electric Power Coordinating Group, Laboratory Analyst**--Responsible for the analyses of Ca, Mg, and NA by ICP Spectroscopy, as well as K by Flame Spectroscopy, for this seven station wet deposition network.

## EDUCATION

B.S., 1991, Chemistry, University of Florida
1991, ICP/MS Workshop, Society for Applied Spectroscopy
1989, GFAA users workshop, Perkin-Elmer
1988, Advanced GFAA workshop, Perkin-Elmer
1987, GFAA seminar, Florida Power and Light
1986, GFAA workshop, Perkin-Elmer
1984, GFAA/FLAA training school, Allied/IL Video 22
1984, GC training school, Perkin-Elmer
1982, GFAA/FLAA training school, Perkin-Elmer
A.A., 1982, Pre-Engineering, Santa Fe Community College
A.S., 1981, Environmental Science, Santa Fe Community College

## ASSOCIATIONS AND CERTIFICATIONS

Florida Society of Environmental Analysts
Society for Applied Spectroscopy

JAMES W. DILLARD, Ph.D.
RADIOCHEMISTRY

## AREAS OF SPECIALIZATION

Radiochemistry, Environmental Analysis, Laboratory Management, Technical Direction, Program Assessment, Analytical Chemistry, Electrochemistry, Project Management, Method Development, and Data Quality Assessment

## EXPERIENCE

Dr. Dillard is a senior technical associate and laboratory department manager with more than 17 years of experience in environmental radiochemical and mixed waste analysis. Relevant experience follows:

**Site Characterization, Loring Air Force Base, Radiochemist**--Performed a baseline study of soil and water samples for gamma spectroscopy, alpha spectroscopy (plutonium, uranium, thorium), radium-$^{226}$, and radium-$^{228}$.

**Site Characterization, Los Alamos National Laboratory, Radiochemistry**--Provided analysis of soil and water samples for alpha spectroscopy (plutonium, thorium) and strontium-$^{90}$.

**International Technology Corporation, Oak Ridge, Tennessee, Senior Scientist**--Served in a technical and management capacity with International Technology (IT) Corporation and most recently as a Senior Technical Associate of IT's Oak Ridge Laboratory Facility (a radiochemical and mixed-waste analysis laboratory). Provided technical overview of the laboratory activities and directed method development, reviewed procedure modifications, contributed to technical preparation of proposals and contracts, and provided technical training. Of special interest is method optimization utilizing new technologies such as open-vessel microwave digestion of samples in preparation for radiochemical analysis. Supported regional and national technical programs such as laboratory automation, protocol standardization, and establishment of nationally accepted performance based methods for environmental radiochemical analysis.

Managed the radioanalytical laboratory and served as a technical and project contact to clients during a period of rapid expansion at the IT Oak Ridge Facility.

**Tennessee Valley Authority, Senior Scientist**--Served at TVA's Eastern and Western Radiological Laboratories in both technical and management positions. Provided technical direction for research and development of radiochemical procedures and provided direction for design and

implementation of a computer laboratory information management system. Coordinated the design, construction, and equipping of TVA's Eastern Area Radiological Laboratory and managed the analytical section at that facility. Also served as manager of TVA's Western Area Radiological Laboratory quality control section.

Performed evaluations of nuclear plant chemical, radiochemical, and health physics programs and activities as an ANSI qualified Lead Auditor for TVA's nuclear power program.

## EDUCATION

Ph.D. Analytical Chemistry, North Carolina State University, 1976
B.S. Chemistry, University of Arizona, 1970

## REGISTRATIONS AND AFFILIATIONS

American Chemical Society
ACS Division of Analytical Chemistry
Sigma XI, The Scientific Research Society of North America

PRESTON F. DUMAS, A.A.
SAMPLE PREPARATION

## AREAS OF SPECIALIZATION

Organic Extractions and Sample Preparation Using Standard EPA Methodologies Including EPA 600 Series Methods, SW 846 Methods, and EPA Contract Laboratory Program Protocols for Both Pesticides and GCMS Sample Preparation

## EXPERIENCE

Mr. Dumas has 11 years of laboratory experience including over 6 years as a supervisor for organic sample preparation. Over the past 6 years, he has had considerable experience with the following methods for water, soil, sediment, and tissue (both plant and animal) sample preparation for organic analysis: EPA Contract Lab Program (CLP) (Semivolatile and Pesticide/PCB) and U.S. Army methods. Relevant experience follows:

**Contamination Evaluation, Installation Restoration Program (IRP), Phase II, U.S. Naval Station, Lee Field and Turner Air Force Base, U.S. Army Corps of Engineers (USACE), Mobile District, Department Manager - Supervised all extractions and sample prep**--Sampling and analysis in accordance with USACE QA/QC requirements of soil, sediment, groundwater and surface water samples to determine presence or absence of chemical contamination. Analysis included priority pollutant volatiles, acid/base/neutral extractables, metals, pesticides and PCBs, and petroleum hydrocarbons.

**Chemical Contamination Evaluation, Ft. McCoy Air Force Base, USACE, Mobile District, Department Manager - Supervised all extractions and sample prep**--ESE has been contracted to determine the presence of chemical contamination at Ft. McCoy Air Force Base. Actual work being performed includes review of available records, sampling and analysis of soil and groundwater, and reports, conclusions and recommendations for future work. Analyses include HSL volatiles, HSL semivolatiles, organochlorine pesticides and PCBs, TRPH, and RCRA metals using EPA SW 846 Methods and adhering to strict QA/QC protocols.

**Remedial Investigation/IRP, Former Webb Air Force Base (AFB), Big Springs, Texas, USACE, Kansas City District, Technician/Department Manager - Involved in and/or supervised all extractions and sample prep**--Sampling and analysis was performed in accordance with the USACE QA/QC requirements to define nature and extent of contamination in four study areas. Several hundred soil samples from each area were collected and analyzed for heavy metals, cyanide, polynuclear aromatics, TCL pesticides and PCBs, TCL

volatiles and extractables, and identification of non-target compounds. Soil gas effort involved analysis of 150 samples for benzene, toluene, n-pentane, n-hexane, and trichloroethane.

**Remedial Investigations/Feasibility Studies (RI/FS) at Multiple U.S. Air Force (USAF) Facilities, USAF Occupational and Environmental Health Laboratories (USAFOEHL), Brooks Air Force Base (AFB), Texas, Department Manager - Supervised all extractions and sample prep**--ESE has a multi-year contract with the Air Force to conduct RIFSs in accordance with CERCLA and SARA. ESE is conducting these programs for the following Air Force bases:

Keesler AFB, Mississippi
Dobbins AFB, Georgia
Patrick AFB, Florida
Plant 78, Utah
Cape Canaveral AFS, Florida
Maxwell AFB, Alabama
Eileson AFB, Alaska
Hickam AFB, Hawaii
Tyndall AFB, Florida

The analytical support provided by ESE's analytical services laboratory involved the analysis of over 4,000 samples according to the latest EPA SW846 and 600-series methods protocols for volatile and semi-volatile organics, petroleum hydrocarbons, heavy metals (including leach and pesticides), and PCB's. Strict QA/QC protocols were followed in these analyses which were conducted under an approved QA project plan.

EDUCATION

A.A. Chemistry/Engineering, University of Florida, 1983

40-Hour Hazardous Waste Site Training Course, February 1990
ACS Short Course "New Sample Preparation Methods for Chemical Analysis", 1991
EAS Short Course "Supercritical Fluid Extraction and Chromatography", 1991

VIRGINIA C. O'BRIEN, B.A.
DATA MANAGEMENT

## AREAS OF SPECIALIZATION

Laboratory Database Management, Project Coordination, Data Management, Data Retrieval, Data Processing

## EXPERIENCE

Ms. O'Brien has 13 years of experience in laboratory database management including the following areas: development of the in-house Laboratory Information Management System (LIMS); coordinating, processing, storing, retrieving, reporting, archiving, and transferring laboratory and geotechnical data; and coordinating the exchange of data between the laboratory and client databases.

**Chemical Contamination Evaluation, Ft. McCoy Air Force Base, U.S. Corps of Engineers (USACE), Mobile District, Laboratory Data Manager**--ESE has been contracted to determine the presence of chemical contamination at Ft. McCoy Air Force Base. Actual work being performed includes review of available records, sampling and analysis of soil and groundwater, and reports, conclusions and recommendations for future work. Analyses include HSL volatiles, HSL semivolatiles, organochlorine pesticides and PCBs, TRPH, and RCRA metals using EPA SW 846 Methods and adhering to strict QA/QC protocols.

**Analytical Services in Support of Contamination Assessment at Eielson Air Force Base (AFB), Harding, Lawson & Associates, Task Manager, Data Management**--ESE was contracted to provide analytical services to Harding, Lawson & Associates in support of an Eielson Air Force Base IRP, Stage 4. ESE provided chemical analyses for environmental soil and water samples, and laboratory and field QC reports in accordance with USAFOEHL format requirements. Analyses included metals, volatiles, semi-volatiles, organochlorine pesticides and PCBs, and TRPH and selected nutrients.

**Contamination Assessment at Dobbins AFB, U.S. Air Force (USAF), Georgia, Task Manager, Data Management**--ESE performed chemical analyses for various inorganics, metals, pesticides and PCBs, and volatiles and BNAs in water and solid matrix from Dobbins AFB in Georgia.

**Remedial Investigation/Installation Restoration Program (IRP), Former Webb AFB, Big Springs, Texas, USACE, Kansas City District, Laboratory Data Coordinator**--Sampling and analysis was performed in accordance with the USACE QA/QC requirements to define nature and extent of contamination in

four study areas. Several hundred soil samples from each area were collected and analyzed for heavy metals, cyanide, polynuclear aromatics, TCL pesticides and PCBs, TCL volatiles and extractables, and identification of non-target compounds. Soil gas effort involved analysis of 150 samples for benzene, toluene, n-pentane, n-hexane, and trichloroethane.

**Environmental Sample Analysis, Contractor Laboratory Analytical Support Services (CLASS), U.S. Army Environmental Center (formerly USATHAMA), CLASS• Task Manager for Data Management**--Responsible for processing all chemical data, formatting, and transferring to USATHAMA computer. ESE has a 5-year, $10 million contract as one of five participating laboratories in USATHAMA's Contractor Laboratory Analytical Support Services (CLASS) program. This project involves analysis of environmental samples on a task order basis in support of remedial investigations and remediation activities.

**Drummed Hazardous Waste Sampling, Homestead AFB, Florida, Laboratory Data Coordinator**--In charge of processing and storing all laboratory data. Drummed liquid hazardous wastes were tested for shipping and disposal parameters for Homestead AFB, Florida. Drums were opened, sampled, and resealed or overpacked, and contents tested onsite by a compatibility protocol. Samples were tested for reactivity, corrosivity, ignitability and to allow for proper compositing. Confirmatory analyses on 20 composite samples were subsequently analyzed in ESE's chemical laboratories.

**Remedial Investigations/Feasibility Studies (RI/FS) at Multiple USAF Facilities, USAFOEHL, Brooks AFB, Texas, Task Manager, Data Management**--In charge of processing all data. ESE has a multi-year contract with the Air Force to conduct RIFSs in accordance with CERCLA and SARA. ESE is conducting these programs for the following Air Force bases: Keesler AFB, Mississippi; Dobbins AFB, Georgia; Patrick AFB, Florida; Plant 78, Utah; Maxwell AFB, Alabama; Eileson AFB, Alaska; Hickam AFB, Hawaii; Tyndall AFB, Florida; and Cape Canaveral AFS, Florida.

The analytical support provided by ESE's analytical services laboratory involved the analysis of more than 4,000 samples according to the latest EPA SW846 and 600-series methods protocols for volatile and semi-volatile organics, petroleum hydrocarbons, heavy metals (including leach and pesticides), and PCB's. Strict QA/QC protocols were followed in these analyses which were conducted under an approved QA project plan. ESE's laboratory data was audited extensively by the USAFOEHL for this work.

## EDUCATION

B.A. Mathematics, Vanderbilt University, 1955

# APPENDIX B

# IRDMIS SOPs

# PROCESSING AND TRANSMITTAL OF PMRMA CHEMICAL DATA USING IRDMIS

STANDARD OPERATING PROCEDURE

SOP4152-06

This Standard Operating Procedure (SOP) describes the process followed to Process and Transmit PMRMA chemical data to Level II.

DATE:_____ AUTHORIZED BY:_____

DISTRIBUTION:

# PROCESSING AND TRANSMITTAL OF PMRMA CHEMICAL DATA USING IRDMIS

SOP

## 1.0 OBJECTIVES

The objectives of this standard operating procedure are to ensure that the proper procedures are followed in:

1.1 Preparing PMRMA lot data files for processing using IRDMIS.

1.2 Processing PMRMA lot data files using IRDMIS.

1.3 Transmitting PMRMA transfer files to PRI.

1.4 Definition: IRDMIS - Installation Restoration Data Management Information System - is the menu-driven PC program group used to process PMRMA chemical, geotechnical and map data for transmittal to Potomac Research, Inc. (PRI) and PMRMA.

Further detailed information may be obtained by referencing the official **USER'S MANUAL for the IRDMIS PC Data Entry & Validation Subsystem**, and the **PMRMA USER'S GUIDE, VOL. II (Data Dictionary)**.

## 2.0 SCOPE

This standard operating procedure applies to all PMRMA data.

## 3.0 PROCEDURES

There are seven steps involved in processing and transmitting data using IRDMIS.

They are as follows:

1) Preparing PMRMA lot files for upload into IRDMIS.

2) Uploading lot files into IRDMIS.

3) Running Record Check on the lots.

4) Running Group Check.

5) Creating IRDMIS transfer files for the lots being processed.

6) Transmitting transfer files to PRI (Level U).

7) Documentation for the lots which have been transferred.

### 3.1 Preparing PMRMA lot files for upload into IRDMIS.

To prepare PMRMA lot files for upload, go to 0: \ARMY and call up the ARMY validation file in brief. The ARMY validation filename is written on the lot cover sheet in the 'comments' section of the 'Data Coordinator' box. See Exhibit A titled 'Chemical Data File Formats' to resolve any questions about the format of the data in the validation file. Make the following alterations to the validation file.

3.1.1 Make sure that there is a 'O' after MO.00 (correct to MO.00 0) in column 36, for the method blank. Do the same for Site Types FBLK, RNSW, and TRIP (ie. FO.00 0, RO.00 0, TO.00 0).

3.1.2 On line 1, add '88.1' starting in column 59. This code assures that units for depths are read as 'feet.'

3.1.3 On line 1, in columns 31-32, add the correct code for prime contractor (reference the PMRMA users guide, section 9.15)

3.1.4 On the sample lines (i.e. S001, S002, etc.) in column 72, add the correct base closure code (Y or N). This information should be supplied by the Lab Coordinator).

3.1.5 On the sample lines, in columns 73-76, add the Sample Delivery Order Number (to be supplied by the Lab Coordinator).

3.1.6 Correct any values (except depths) which are reported as 10.0 to 01.0 ... (ie 10.0-02 = 01.0-01).

3.1.7 Make sure that all required field data is present.

### 3.2 Uploading PMRMA files into IRDMIS.

Before loading a new group of lots into IRDMIS for processing, 'ZAP' lot files already transferred out of IRDMIS. To do this, go to C:\ and type 'ZAP'. This will clean out the database files.

From C:\ type 'IR.'

3.2.1 When prompted to 'Press any key to continue,' do so.

3.2.2 After 'Zapping' the data base files, it is necessary to reindex the files. To do this, choose number 4 from the menu - Utilities. Then choose 3 - Index all data bases.

To begin uploading PMRMA files:

3.2.3 Choose number 1 from the menu - Chemical Data.

3.2.4 Choose number 1 from the menu - Enter New Data.

3.2.5 At the prompt for 'mode of entry,' press 'F' for file input.

3.2.6 Enter the name and location of the file to upload (O:\ARMY\FILENAME).

3.2.7 Press <enter> when done to return to the menu.

### 3.3 Running IRDMIS Record Check.

Before running Record Check, go to a network directory and set up the PrinTamer so that any Record Check errors can be captured and printed so we have a hard copy of them. PrinTamer will not be able to read the PDF file and load the fonts from the c: drive.

3.3.1 From the IRDMIS Chemical Data Main Menu, choose number 4-Record check existing data.

3.3.2 Enter the lots to be processed by either manually typing in the Installation code, Laboratory, and Lot number, or by pressing <page down> to call up a window which displays the lots. In the window, press the number (1 to 5) of the lot to process, and press <page down> again. If the number of lots is greater than 5, pressing the <space> bar will show the second, third, etc, group of 5 lots.

3.3.3 Press 'N' when all lots to be processed are loaded in.

3.3.4 IRDMIS will either report that a lot is dean, or that it is in error and ask if you wish to edit the data found in error. If it is clean, simply record the lot and the number of analysis records in the Lot Transfer Verification Book. If it is in error, press 'Y' to edit. Using the PrinTamer, make a hardcopy of the screen showing the error(s). Then follow the procedure in appendices A and B for correcting and/or documenting errors.

### 3.4 Running IRDMIS Group Check.

After lots have been run through Record check and are clean, or which contain errors deemed acceptable:

3.4.1 Chose number 5 - Group check existing data - from the menu.

3.4.2 Load the lots to be checked, as in Step 3.

3.4.3 If a lot contains an error, IRDMIS will report that 'This lot contains invalid data - Do you wish to confine (Y/N).' Press 'Y' if the error has been deemed acceptable.

3.4.4 When all lots have been loaded, IRDMIS will show the filename which the results will be written to: \IRSCC\SCC91001.GRP. The numeric part of the filename is the Julian date, in this case 91001.

3.4.5 When processing is complete, print the SCC file. Review the results for errors. Once all errors are deemed acceptable, or all lots check clean, go to step 5.

## 3.5 Creating IRDMIS transfer files.

3.5.1 Choose number 6 - Output existing data - from the menu.

3.5.2 Press 'T' to create a transfer file.

3.5.3 IRDMIS will show the default subdirectory and filename for the TRN file.

3.5.4 Change the subdirectory from \IRSCC to \TRNIR.

3.5.5 Once all desired lots have had transfer files created, go to C:\TRNIR.

3.5.6 Type 'PACKES' to create an archive file of the transfer files.

3.5.7 Create a README file (READMonthDay.Year - READ414.91, etc) listing the transfer files being transmitted, and their status as clean or in error. (See previous README files for details).

3.5.8 PACKES will change the *.trn ending to *.trr so that once a trn file has been put in an archive file, it will not be picked up again.

3.5.9 Copy the *.arc and the readme file onto a disk.

## 3.6 Transmitting PMRMA transfer files to DPA.

Send the diskette containing the transfer files and readme file to DPA along with a letter of transmittal listing all lots on the diskette via U.S. mail or as directed.

## 3.7 Documentation of transferred lots.

Documentation consists of recording the lot, date of transfer, number of analysis records, and lot status (clean or not) in the Lot Transfer Verification Book. It also consists of printing out a hardcopy list of the lots, archived filename, and readme filename.

3.7.1 At C:\TRNIR, type DIR >C:\TRNIR\RECORD\RECORD.xxx, where the extension is the month and day of the transmittal. The DIR > command will list the entire contents of the directory to the file Record.xxx, which will be located in the directory C:\TRNIR\RECORD. By editing this file in brief, a list of the transferred files can be easily obtained.

3.7.2 Print out all of the lot transfer files, two copies of the readme file and two copies of the Record.xxx file. This may be done easily by using a batch file.

3.7.3 Copy all of the lot transfer files and the readme file to the current disk of 'Files Sent to DPA.

3.7.4 Upload the readme file into the Lot Verification screen. At the main CLASS menu, choose the USATHAMA/PMRMA submenu. Choose the UNISYS submenu, and then

choose Lot Verification. Enter the directory and filename of the readme file, and the date of the readme file. This will add the DPA submittal date to CHEMTRAK.

3.7.5 Move the readme file to the directory C:\TRNIR\README, and delete the readme file and the lot transfer files from the C:\TRNIR directory.

This completes the process. See appendices A, B, and exhibit A for additional information.

Appendix A

## 1.0 OBJECTIVES

The objectives of this appendix are to ensure that the proper procedures are followed in correcting and/or determining the acceptability of errors occurring in IRDMIS processing.

## 2.0 SCOPE

This appendix applies to all PMRMA lot data files encountering errors in IRDMIS processing.

## 3.0 PROCEDURES

3.1 If the error lies with the field data and/or sampling dates, first check the logsheet to assure that the correct field data and sampling data have been entered into the lot. If the error is with the chemical data, go to step #3.

3.2 If the field data and sampling date on the logsheet match the data in the lot file, use the IRDMIS data dictionary to find the correct site types which are allowable for the filename give (CSO, CSE, CSW, CGW, etc.).

3.3 Then consult with the lab coordinator to verify the correct data which can then be entered upon obtaining a written correction notice from the lab coordinator. In the case of chemical data, verify that the data are in error and obtain a written explanation which can be submitted upon transmission data to Level II.

3.4 If all the data seem to be correct, yet the error message persists, contact Virginia O'Brien so that she can contact PRI in order to determine whether and how the data can be submitted, and if the error lies with the data or with the software.

3.5 When errors have been corrected, rerun the lots in question through Record and Group check again to clean them up. (IRDMIS flags errors internally so that lots in error must be run through again when clean in order to remove the error flag.)

Appendix B

## 1.0 OBJECTIVE

The objectives of this appendix are to ensure that the proper procedures are followed in documenting PMRMA lot errors which occur in IRDMIS processing.

## 2.0 SCOPE

This appendix applies to all PMRMA data encountering errors in IRDMIS processing.

## 3.0 PROCEDURES

3.1 PMRMA lots arrive from Quality Assurance. The lots are uploaded into the IRDMIS system and processed.

3.2 Lots without Record and Group Check errors are sent to DPA according to regular procedure.

Lots which contain errors are dealt with in the following manner:

3.3 Lots which contain errors which can be resolved by consulting with the project and/or lab coordinator shall be corrected as per their instructions and reprocessed. (Errors of this type include incorrect Site ID's, Missing Map data, etc.) If the lot the checks clean, it will be sent to DPA according to regular procedure.

3.4 Lots which contain errors which are due to lab / analysis / other situations which cannot be corrected shall be handled as follows:

3.4.1 A hardcopy screen printout of the errors as they occur in IRDMIS will be obtained to document the exact error message and the data in error.

3.4.2 This printout shall be attached to the IRDMIS PROCESSING COMMENT FORM, which will contain explanations of the data in error, the reason for the error, and any other documentation pertaining to the error. The Group Check results will also be attached. This documentation shall then be added to the lot folder. A copy shall be made for ESE files, so that ESE can retain documentation of the errors independent of the lot folder.

3.4.3 Upon submittal to DPA, a README file will be included in the transmittal which will

include the explanations included in the IRDMIS Processing Comment Form.

Note: Lots which pass both Record and Group checks will not have an IRDMIS Processing Comment Form included in the lot folder. The Group check results printout will serve as documentation of the lot having checked cleanly.

3.5 Lots which fail only because of missing map information will be dealt with as follows:

3.5.1 If PMRMA has authorized the submission of lots for which we are missing map information, these lots shall be sent to DPA. These lots will not have an IRDMIS Processing Comment Form, as the Group check record shall be sufficient documentation.

3.5.2 If PMRMA has not authorized the submission of lots for which we do not have map information, these lots shall not be sent. The project manager or lab coordinator will be notified so that map information can be obtained. These lots will be held pending reception of map information, or authorization to sent without maps.

Exhibit A

Chemical Data File Formats

**Lot Record**

| Columns | |
|---|---|
| 1 | "L" |
| 2-3 | Installation Code |
| 4-5 | Laboratory Code |
| 6-8 | Lot Number |
| 9-12 | Method Number |
| 13-16 | Units |
| 17-19 | Initials |
| 20 | Class Indicator |
| 24 | Delivery Order Indicator |
| 31-32 | Prime Contractor |

**Sample Record**

| Columns | |
|---|---|
| 1 | "S" |
| 2-4 | Sample Number |
| 5-7 | File Name |
| 8-11 | Site Type |
| 12-21 | Site Identification |
| 22-29 | Field Sample Number |
| 30-37 | Sample Date (MM/DD/YY) |
| 38-40 | Sample Program |
| 41-46 | Sample Depth |
| 47 | Sample Technique |
| 48-55 | Laboratory Sample Number |
| 56-63 | Sample Preparation Date (19YYMMDD) |
| 64-71 | Analysis Date (19YYMMDD) |
| 72 | Base Closure |
| 73-76 | Delivery Order Number |

**Analysis Record**

| Columns | |
|---|---|
| 1 | "A" |
| 2-7 | Test Name |
| 8-9 | Boolean |
| 10-13 | Uncorrected Mantissa (#.##) |

| | |
|---|---|
| 14-16 | Uncorrected Exponent |
| 17-19 | Dilution Mantissa (#.##) |
| 20 | Dilution Exponent |
| 21-24 | Moisture (##.#) |
| 25-28 | Accuracy |
| 29 | Flagging Code |
| 30 | Qctest Code |
| 31-34 | QC Mantissa (#.##) |
| 35-37 | QC Exponent |

# APPENDIX C

# LICENSES AND CERTIFICATIONS

DEPARTMENT OF THE ARMY
MISSOURI RIVER DIVISION, CORPS OF ENGINEERS
P.O. BOX 103, DOWNTOWN STATION
OMAHA, NEBRASKA 68101-0103

REPLY TO ATTENTION OF

January 12, 1993

Environmental, Hazardous, Toxic
and Radioactive Waste Division

Environmental Science, and Engineering, Inc.
5 Miles West I-75 on State Road 26
Gainesville, Florida 32607

Gentlemen:

This correspondence addresses the recent evaluation of your laboratory by the U.S. Army Corps of Engineers (USACE) for hazardous and toxic waste analysis in support of the USACE hazardous and toxic waste program and the Technical Support Division of U.S. Army Toxic and Hazardous Materials Agency (USATHAMA).

Your laboratory has successfully analyzed the audit samples as listed below:

| METHOD | PARAMETERS | MATRIX |
|---|---|---|
| 8240 | Volatile Organics | Water |
| 8010 | Halogenated Volatile Organics | Water |
| 8020 | Aromatic Volatile Organics | Water |
| 8270 | Semivolatile Organics | Water |
| 8270 | Semivolatile Organics | Sediment |
| 8080 | Organochlorine Pesticides | Water |
| 8080 | PCBs | Water |
| 8080 | PCBs | Sediment |
| 8150 | Herbicides | Water |
| 8040 | Phenols | Water |
| 8310 | PAHs | Water |
| 8330 | Explosives | Water |
| 8330 | Explosives | Sediment |
| SW-846 | TAL Metals (2) | Water |
| SW-846 | TAL Metals (2) | Sediment |
| 8015 | TPH | Water |
| 8015 | TPH | Sediment |
| 418.1 | TRPH | Water |
| 9071 | TRPH | Sediment |

| | | |
|---|---|---|
| 300 series | Anions (1) | Water |
| 9010 | Cyanide | Water |
| 9060 | TOC | Water |

Remarks: (1) Anions: chloride, fluoride, sulfate, nitrate and ortho-phosphate.

(2) TAL metals include: aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.

Enclosed for your information is a copy of the Laboratory Inspection and Evaluation Report. Your laboratory has responded to the majority of the deficiencies and recommendations made. However, responses are required within ten working days, to the items outlined in paragraph 5.d. of the report.

Based on the successful analysis of the project specific audit samples indicated in the table in Paragraph 2 above and the results of the laboratory inspection, your laboratory is validated for multimedia sample analysis by the methods listed above. The period of validation is eighteen (18) months and expires on June 8, 1994.

USACE reserves the right to conduct additional laboratory auditing or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of analytical samples from USACE Contracting Officer Representatives. If you have any questions or comments, please contact Paulette Lewis at (402) 221-7494.

Sincerely,

Thomas L. Donaldson

for Marcia C. Davies
Chief, Environmental, HTRW Division
HTRW and Engineering Directorate

Enclosure

№ 11

STATE OF MARYLAND

# DEPARTMENT OF HEALTH AND MENTAL HYGIENE
# LABORATORIES ADMINISTRATION

*Certifies That*

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.

14220 W. NEWBERRY RD, GAINESVILLE, FL. 32607

*having duly met the requirements of the*

*Regulations Governing Laboratory Certification*
*And Standards Of Performance In Accordance With*
*The Annotated Code of Maryland,*
*is hereby approved as a*

## *State Certified Water Quality Laboratory*

*To perform the analyses indicated on the Annual Certified Parameter List,*
*which must accompany this certificate.*

Certification # 228

Date Issued FEBRUARY 7, 1994

Expiration Date MARCH 31, 1995
(Not Transferable)

J. Melvin Joseph
Director, Laboratories Administration

This certification is subject to unannounced laboratory inspections

CONSPICUOUSLY DISPLAY IN THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST

*State of Florida*

*Department of Health and Rehabilitative Services*

# *OFFICE OF LABORATORY SERVICES*

# *SAFE DRINKING WATER*

*This is to certify that*

**HRS # 82138**
Env. Science & Eng, Inc. (ESE)
5 miles West of I-75 on S.R. 26
Gainesville, FL 32602

*has complied with Florida Administrative Code Section 10D-41.050 pertaining to safe drinking water testing in the following categories:*

Primary Inorganic Contaminants, Secondary Inorganic Contaminants, Pesticides and PCB's, Other Regulated Contaminants, Group I Unregulated Contaminants, Group II Unregulated Contaminants

*Specific certified analytes and methodologies within these categories are listed on the analyte sheets with this laboratory and the DHRS Office of Laboratory Services*

*EFFECTIVE* July 1, 1994 *through* June 30, 1995

*1994 - 1995*

*No.* 94114
*Non-Transferable*

*Eldert C. Hartwig, Jr., ScD., M.P.H.*
*Chief, Laboratory Services*
*Office of Laboratory Services*
*HRS Form 1697, May 94*

© GOES 445

# State of Florida

## Department of Health and Rehabilitative Services

# OFFICE OF LABORATORY SERVICES
# ENVIRONMENTAL WATER

*This is to certify that*

HRS # E82067
Environmental Science & Engineering, Inc. - Gainesville
5 miles west of I-75 on SR 26
Gainesville, FL 32602-3053

*has complied with Florida Administrative Code Section 10D-41.100 for the examination of environmental water in the following categories:*

Bioassay, Metals, Nutrients, Demands, General Category I, General Category II, Extractables (GC, GC/MS), Purgeables (GC, GC/MS), Hazardous Waste Characterization, Pesticides/Herbicides/PCB's (GC, GC/MS)**********

*Specific methods, parameters, and analytes certified are on file at the Office of Laboratory Services, P. O. Box 210, Jacksonville, Florida 32231.*

*EFFECTIVE* July 1, 1994 *through* June 30, 1995

*1994 - 1995*

*No.* 94021
Non-Transferable

Eldert C. Hartwig, Jr., ScD., M.P.H.
Chief, Laboratory Services
Office of Laboratory Services
HRS Form 1697, May 94

© GOES 442

STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES

ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
14220 Newberry Road
Gainesville, FL 32607

*I.D. #82138*

Based on the authority mandated by Florida Statute 403.863 and Florida Administrative Code 10D-41, it is hereby declared that

# ENVIRONMENTAL SCIENCE & ENGINEERING

is granted certification to perform the following measurements on compliance drinking water samples:

**GROSS ALPHA**
**GROSS BETA**
**NATURAL URANIUM**
**PHOTON EMITTERS:**
**(Cs-134, Cs-137, Co-60, Ba-133, Zn-65, Ru-106)**
**RADIUM-226**
**RADIUM-228**
**STRONTIUM-89**
**STRONTIUM-90**
**TRITIUM**

Compliance in this case specifies that Environmental Science and Engineering has met the minimum requirements for a radiochemistry laboratory as set forth in Florida Administrative Code 10D-41.050-062.

This certification is effective as of July 1, 1994 and shall be in effect until June 30, 1995 or until revoked by the Florida Department of Health and Rehabilitative Services.

Joseph Escalante
Certification Officer
Safe Drinking Water Laboratory

Benjamin Prewitt
Certification Officer
Safe Drinking Water Laboratory

Any party whose substantial interests are affected by this determination has a right to request an administrative proceeding pursuant to Section 120.57, Florida Statute, and rules promulgated pursuant thereto, within 30 days of receipt of this notice. Failure to timely request a hearing in writing shall be deemed a waiver of any right to a Section 120.57, Florida Statute proceeding and this decision shall become final agency action.

HRS STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES

ENVIRONMENTAL SCIENCE & ENGINEERING, INC. *I.D. #E82067*
14220 Newberry Road
Gainesville, FL 32607

Based on the authority mandated by Florida Statute, Section 403.0625 and Florida Administrative Chapter 10D-41, it is here by declared that

# ENVIRONMENTAL SCIENCE & ENGINEERING

is granted certification to perform measurements on ENVIRONMENTAL WATER SAMPLES in the following category:

## RADIOLOGICAL:

**Total Alpha** **Radium-226**
**Total Beta** **Total Radium**

Compliance in this case specifies that Environmental Science & Engineering has met the minimum requirements for a radiochemistry laboratory as set forth in Chapter 10D-41.100-113, Florida Administrative Code.

This certification is effective as of July 1, 1994 and shall be in effect until June 30, 1995 or until revoked by the Florida Department of Health and Rehabilitative Services.

Joseph Escalante
Certification Officer
Safe Drinking Water Laboratory
Office of Radiation Control

Benjamin Prewitt
Certification Officer
Safe Drinking Water Laboratory
Office of Radiation Control

Any party whose substantial interests are affected by this determination has a right to request an administrative proceeding pursuant to Section 120.57, Fla. Stat., and rules promulgated pursuant thereto, within 30 days of receipt of this notice. Failure to timely request a hearing in writing shall be deemed a waiver of any right to a Section 120.57, Fla. Stat. proceeding and this decision shall become final agency action.

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

Pursuant to Chapter 404, Florida Statutes, and Chapter 10D-91, Florida Administrative Code (F.A.C.), and in reliance on statements and representations heretofore made by the licensee designated below, a license is hereby issued authorizing such licensee to receive, acquire, possess and transfer the radioactive material(s) designated below and to use such radioactive material(s) for the purpose(s) and at the place(s) designated below. This license is subject to all applicable rules, regulations and orders of the state of Florida, Department of Health and Rehabilitative Services now or hereafter in effect and to any conditions specified below.

| Licensee | |
|---|---|
| 1. Name: ENVIRONMENTAL SCIENCE & ENGINEERING, INC. | With reference to correspondence dated 05/11/94, State of Florida Radioactive Materials 3. License Number: 2119-1 is hereby amended in its entirety to read as follows: |
| 2. Address: P.O. Box 1703 Gainesville, FL 32602-1703 | 4. Expiration Date: December 31, 1995<br>5. Category: 3K |

| Radioactive material (element and mass number) | 7. Chemical and/or physical form | 8. Maximum quantity licensee may possess at any one time |
|---|---|---|
| A. Atomic numbers 1 through 96 excluding special nuclear materials or as licensed below | A. Any form except gas | A. No isotope to exceed 0.1 millicurie. Total not to exceed 100 millicuries |
| B. Carbon 14 | B. Aqueous, sealed ampules | B. 2 millicuries |
| C. Nickel 63 | C. Sealed source (Hewlett-Packard Detector Cell Part Number 18713A) | C. 7 sources; not to exceed 15 millicuries each |
| D. Nickel 63 | D. Sealed source (Perkin-Elmer Detector Cell Part Number 330-0119) | D. 5 sources; not to exceed 15 millicuries each |
| E. Cesium 137 | E. Sealed source (ICN Pharmaceuticals Model 375) | E. 5 millicuries |
| F. Barium 133 | F. Disc source | F. 0.3 microcurie |

(See Page 2)

Licensee copy

License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

| 6. Radioactive material (element and mass number) | 7. Chemical and/or physical form | 8. Maximum quantity licensee may possess at any one time |
|---|---|---|
| G. Cesium 137 | G. Disc source | G. 0.3 microcurie |
| H. Cobalt 60 | H. Disc source | H. 0.3 microcurie |
| I. Uranium 238 | I. Alpha standard | I. 0.001 microcurie |
| J. Barium 133 | J. Gamma solution | J. 5 microcuries |
| K. Cesium 137 | K. Gamma solution | K. 5 microcuries |
| L. Iodine 131 | L. Standard solution | L. 10 microcuries |
| '. Americium 241 | M. Sealed source (Eberline Instrument Corp. Model Number DNS-5) | M. 5 microcuries |
| N. Phosphorous 32 | N. Solution | N. 5 millicuries |
| O. Nickel 63 | O. Foils (U.S. Radium Lab 784 or NEN Model Number NER-004 in Tracor Part Number 23604-0001) | O. No single source to exceed 15 microcuries each |
| P. Polonium 208 | P. Solution | P. 0.1 microcurie |
| Q. Radium 228 | Q. Solution | Q. 5 microcuries |
| R. Uranium 232 | R. Solution (EPA) | R. 5 microcuries |
| S. Thorium 229 | S. Solution | S. 5 microcuries |
| T. Radium 226 | T. Solution (EPA) | T. 5 microcuries |
| U. Americium 241 | U. Sealed source (New England Nuclear NES 302S) | U. 50 nanocuries |
| '. Technetium 99 | V. Solution (NBS) | V. 5 microcuries |

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

| 6. Radioactive material (element and mass number) | 7. Chemical and/or physical form | 8. Maximum quantity licensee may possess at any one time |
|---|---|---|
| W. Strontium 89 | W. Solution (EPA) | W. 5 microcuries |
| X. Strontium 90 | X. Solution (EPA) | X. 5 microcuries |
| Y. Nickel 63 | Y. Sealed source (Hewlett-Packard Detector Cell Part Number 19282) | Y. 15 sources; not to exceed 15 millicuries each |
| Z. Nickel 63 | Z. Sealed source (Hewlett-Packard Detector Cell Part Number 19312) | Z. 7 sources; not to exceed 15 millicuries each |
| AA. Carbon 14 | AA. Labeled organic chemicals | AA. 50 millicuries |
| BB. Plutonium 236 | BB. Aqueous solution | BB. 5 microcuries |
| CC. Nickel 63 | CC. Sealed source (Hewlett-Packard Detector Cell Part Number 19233) | CC. 15 sources; not to exceed 15 millicuries each |
| DD. Hydrogen 3 | DD. Tritium labeled organic compounds | DD. 100 millicuries |
| EE. Iron 55<br>Iron 59<br>Cesium 137<br>Bismuth 207<br>Polonium 207<br>Lead 210<br>Plutonium 236<br>Plutonium 239 | EE. Any form except gas | EE. Not to exceed 1 millicurie for each radionuclide |
| FF. Polonium 209<br>Chromium 51<br>Zinc 65<br>Ruthenium 106<br>Cesium 134 | FF. Solution | FF. Not to exceed 5 microcuries for each radionuclide |

(See Page 4)

License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

| 6. Radioactive material (element and mass number) | 7. Chemical and/or physical form | 8. Maximum quantity licensee may possess at any one time |
|---|---|---|
| GG. Hydrogen 3 | GG. Sealed source (G-cubed Detector Cell Part Number 200-ECD) | GG. 300 millicuries |
| HH. Radioactive material distributed to a General Licensee per 10D-91.306(1) and (4), F.A.C. | HH. Sealed source(s) and/or contained source(s) | HH. No single source to exceed that quantity authorized for the general license device |
| II. Uranium 238 with daughters natural or depleted | II. Any form except gas | II. No sample to exceed 50 grams/16.8 micro-curies of U 238. Total not to exceed 1 kilogram/335.1 microcuries U 238 |
| JJ. Thorium 232 with natural daughters only | JJ. Any form except gas | JJ. No sample to exceed 50 grams/5.5 microcuries of Th 232. Total not to exceed 1 kilogram/109.5 microcuries Th 32 |
| KK. Plutonium (excluding Pu 238 and Pu 241) | KK. Any form except gas | KK. No radionuclide to exceed 10 micro-curies |
| LL. Plutonium 238 | LL. Any form except gas | LL. No sample to exceed 10 microcuries or 0.6 micrograms. Total not to exceed 600 micrograms |

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

| 6. Radioactive material (element and mass number) | 7. Chemical and/or physical form | 8. Maximum quantity licensee may possess at any one time |
|---|---|---|
| MM. Plutonium 241 | MM. Any form except gas | MM. No sample to exceed 10 microcuries or 0.1 micrograms. Total not to exceed 100 micrograms |
| NN. Uranium 233 | NN. Any form except gas | NN. No sample to exceed 10 microcuries or 1.04 milligrams. Total not to exceed 100 microcuries or 46.3 milligrams |
| OO. Uranium 235 as contained in enriched uranium | OO. Any form except gas | OO. No single sample to exceed 10 microcuries or 4.63 grams. Total not to exceed 100 microcuries or 46.3 grams |
| PP. Americium 241/ curium 244/ plutonium 239 | PP. Plated source (North American Scientific, Inc.) | PP. 4 sources; not to exceed 2.0 nanocuries of each radionuclide for each source. |

9. Authorized use

A. Samples received for the purpose of analytical isotopic determination.

B. For storage only.

C. To be used in Hewlett-Packard Model 5739 gas chromatographs.

D. To be used in Perkin-Elmer Sigma Gas chromatographs.

E. To be used in the calibration of monitoring equipment.

(See Page 6)

License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

9. Authorized use (continued)

F. through L. Source standards.

M. To be used for instrument calibration.

N. To be used in calibration and spiked sample analysis.

O. To be used in Tracor Model 560 gas chromatographs.

P. To be used as a tracer for Po-210 analysis.

Q. To be used for instrument calibration.

R. To be used as a tracer for isotopic uranium determinations.

S. To be used as a tracer for isotopic thorium determinations.

T. and U. To be used for instrument calibration.

V. through X. To be used for instrument calibration and chemical yield.

Y. To be used in Hewlett-Packard Model 5790 gas chromatographs.

Z. To be used in Hewlett-Packard Model 5880 gas chromatographs.

AA. To be used in laboratory aquatic and/or bioaccumulation studies

BB. To be used in chemical yield determination in alpha spectrometry and spectrometer calibration.

CC. To be used Hewlett-Packard Model 5890 gas chromatographs.

DD. To be used in laboratory aquatic and/or bioaccumulation studies.

EE. To be used as calibration and reference standards and for recovery and/or tracer studies.

FF. To be used as calibration and reference standards and for tracer studies.

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

9. Authorized use (continued)

GG. To be used in a S-Cubed Model 215BCG trace gas analyzer.

HH. To be used in devices approved for receipt under general license provisions as described in Items 6, 7 and 8.

II. and OO. Samples received for the purpose of analytical isotopic determination.

PP. To be used as a calibration/reference source in an alpha spectrometer.

CONDITIONS

10. The authorized place of use and storage is the licensee's facility located 5 miles west of I-75 at 14220 Newberry Road (State Road 26), Gainesville, Florida.

11. Failure to comply with the provisions of this license is a felony of the third degree pursuant to section 404.161, Florida Statutes. Also, violations may warrant an administrative fine of up to $1,000.00 per violation per day pursuant to section 404.162, Florida Statutes.

12. A. Licensed materials shall be used by, or under the supervision of Daniel D.A. Duncan, III, Kenneth U. Erondu, Richard M. Kinney, Sandra Mihocik, Sarah Stewart, or Andrew Weitz.

B. The radiation safety officer is Richard M. Kinney.

13. The licensee shall comply with the provisions of Chapter 10D-91, F.A.C., Part X, "Notices, Instructions and Reports to Workers; Inspections" and Part IV, "Standards for Protection Against Radiation."

14. The licensee shall not transfer possession or control of radioactive material, or products containing radioactive material as a contaminant except:

A. By transfer to a specifically licensed recipient; or

B. As provided otherwise by specific provision of his license pursuant to the requirements of Chapter 10D-91, F.A.C.

(Page 8)

License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
OFFICE OF RADIATION CONTROL

RADIOACTIVE MATERIALS LICENSE

15. Sealed sources containing licensed material shall not be opened nor removed from their respective source holders by the licensee

16. Detector cells containing licensed material shall not be opened nor the foil source removed from the detector cell by the licensee

17. Detector cells containing (hydrogen-3, scandium tritide, titanium tritide, nickel-63, or strontium-90) shall only be used in conjunction with a properly operating temperature control mechanism which prevents the temperature from exceeding 350 degree Celsius.

18. A. Maintenance and repair of devices containing radioactive material shall be performed only by persons specifically authorized by the U.S. Nuclear Regulatory Commission or an Agreement State to perform such services.

B. Installation, replacement and disposal of sealed sources in the source holder shall be performed only by the sealed source manufacturer or by other persons specifically authorized to perform such services.

19. Radioactive material transported on public thoroughfares shall be packaged, prepared for shipment and transported in accordance with Title 49, Code of Federal Regulations and Chapter 10D-91, F.A.C.

20. The licensee shall assure that each sealed source is tested for leakage or contamination and follow the appropriate actions as required by section 10D-91.1404, F.A.C. Licensed material other than Hydrogen 3, with a half-life greater than thirty days, shall be tested at least semiannually, except licensed material designed for the purpose of emitting alpha particles which shall be tested at least quarterly. The test sample (smear) shall be taken by the licensee using an approved leak test kit. Analysis of the test sample shall be performed by individuals who are licensed by the department, NRC, agreement state, or licensing state to provide these services. The licensee is required to retain leak test records containing the manufacturer's name, model and serial number of each sealed source tested, identity of each sealed source radionuclide and its estimated activity, the measured activity of each test sample expressed in microcuries, the date of the test and signature of the radiation safety officer or designee. The records shall be maintained for 3 years for inspection by the department.

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
OFFICE OF RADIATION CONTROL

RADIOACTIVE MATERIALS LICENSE

21. The licensee shall conduct a physical inventory and inspection at least semiannually to account for all sealed sources received and possessed under this license as required by section 10D-91.1405, F.A.C. Inventory records shall be maintained for 3 years from the date of the inventory for inspection by the department and shall include the manufacturer's name, model and serial numbers of each sealed source, the identity of each sealed source radionuclide and its estimated activity, the location of each sealed source, the date of the inventory and the signature of the radiation safety officer or designee.

22. The licensee shall not use radioactive material in or on human beings nor in field applications where radioactive material is released to the environment, except as provided otherwise by a specific provision of this license.

23. Animals and plants administered radioactive materials and their products, shall not be used for human consumption.

24. The licensee shall comply with section 10D-91.1410, F.A.C., listing additional requirements for device(s) received under general license provisions as described in items 6, 7, 8 and 9, subitem HH. The licensee shall conduct a physical inventory and inspection at least annually to account for all sealed sources received and possessed under this license as required in section 10D-91.1405, F.A.C. Inventory records shall be maintained for 3 years from the date of the inventory for inspection by the department, and shall include the manufacturer's name, model and serial numbers of each sealed source, the identity of each sealed source radionuclide and its estimated activity, the location of each sealed source, the date of the inventory and the signature of the radiation safety officer of designee.

25. Individuals involved in operations which utilize, at any one time or over a 3 month period, tritium in an unsealed form that exceeds the activities specified in Table 1 of the U.S. Nuclear Regulatory Commission's Regulatory (NRC) Guide 8.32 shall have bioassays performed at the following frequency and follow the corresponding actions:

A. (I) A bioassay shall be taken within 72 hours of initial use of tritium and every 2 weeks thereafter. When work with tritium is on an infrequent basis (less frequent than every 2 weeks), a bioassay shall be taken within 10 days of the last day of use.

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
OFFICE OF RADIATION CONTROL

RADIOACTIVE MATERIALS LICENSE

25. A. (II) The licensee shall take the corresponding actions according to the action levels listed below:

(a) If the intake of tritium within any 40-hour work period exceeds the amount that would be taken into the body from uniform exposure for 40 hours at the air concentration of 5 X 10-6 microcuries per milliliter, the licensee is required to make evaluations, take necessary corrective actions, and maintain records required by section 10D-91.404(5), F.A.C.

(b) If the urinary excretion rates exceed 5 microcuries per liter but are less than 50 microcuries per liter, the following actions shall be taken:

(1) An investigation of the operations involved, including air and surface contamination surveys, shall be carried out to determine the causes of exposure and to evaluate the potential for further exposures or possible involvement of other employees;

(2) If the investigation indicates that further work in the area might result in exposure of a worker to concentrations that would cause the limiting intakes established in section 10D-91.404, F.A.C., to be exceeded, the licensee shall restrict the worker from further exposure until the source of exposure is discovered and corrected;

(3) Any reasonable corrective actions that will eliminate or lower the potential for further exposures shall be implemented;

(4) A repeat bioassay shall be taken within 1 week of the previous measurement and shall be evaluated within 1 week after the measurement. Internal dose commitments shall be estimated using at least two bioassays and other survey data, including the probable times of intake of tritium; and

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License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

STATE OF FLORIDA
DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
OFFICE OF RADIATION CONTROL

RADIOACTIVE MATERIALS LICENSE

25. B. (I) (c) The working conditions during the 3 month period, with respect to the potential for tritium exposure, are representative of working conditions during the period in which the quarterly urinalysis frequency is employed, and there is no reasonable expectation that the criteria given in B.(I)(a) and B.(I)(b) will be exceeded.

(II) If the urinary excretion rate exceeds 5 microcuries per liter, the following actions shall be taken:

(a) Carry out all steps as described in A.(II)(b) and A.(II)(c) of this condition; and

(b) Reinstitute bioassays every 2 weeks for at least the next 16 months, (even if the urinary excretion falls below 5 microcuries per liter,) before reestablishing quarterly bioassays.

26. A. Except as specifically provided otherwise by this license, the licensee shall possess and use licensed material described in items 6, 7, 8, and 9 of this license in accordance with statements, representations and procedures contained in the licensee's application dated April 17, 1990, signed by Daniel Moore, Department Manager, and correspondence dated:

May 31, 1990, signed by Daniel Moore, Department Manager;
July 23, 1990, signed by Richard M. Kinney, Radiochemist;
July 1, 1991, signed by Kenneth U. Erondu, Division Manager Inorganic Chemistry;
September 6, 1991, signed by John J. Mousa, Ph.D., Director Analytical Services;
November 26, 1991, signed by Kenneth U. Erondu, Division Manager, Inorganic Analytical Chemistry;
March 19, 1992, signed by Richard M. Kinney, Radiation Safety Officer;
April 13, 1994; and
May 11, 1994, both signed by Kenneth U. Erondu, Associate Vice President.

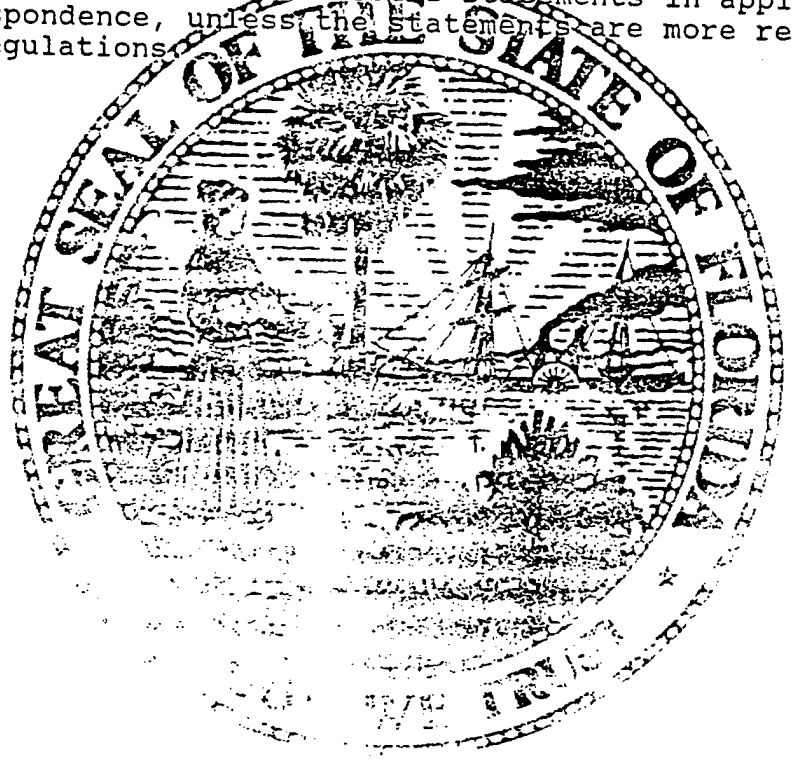
(See Page 13)

License Number 2119-1
AMENDMENT NO. 6
Category: [3K]
Expiration date: 12/31/95

# STATE OF FLORIDA
# DEPARTMENT OF HEALTH AND REHABILITATIVE SERVICES
# OFFICE OF RADIATION CONTROL

## RADIOACTIVE MATERIALS LICENSE

26. B. The licensee shall comply with all applicable requirements of Chapter 10D-91, Florida Administrative Code, and these regulations shall supersede the licensee's statements in applications or correspondence, unless the statements are more restrictive than the regulations.



For the Office of Radiation Control

Issuance Date JUN 14 1994

Joy Stephenson
Public Health Physicist
1317 Winewood Blvd.
Tallahassee, FL 32399-0700
(904) 487-2437

# STATE OF NEW JERSEY
# DEPARTMENT OF ENVIRONMENTAL PROTECTION

Let's protect our earth

*Certifies That*

Environmental Science & Engineering, Inc.
PO Box 1703
Gainesville, FL. 32602-1703

*having duly met the requirements of the*

*Regulations Governing Laboratory Certification*
*And Standards Of Performance N.J.A.C. 7:18 et. seq.*

*is hereby approved as a*

## *State Certified Water Laboratory*

*To perform the analyses as indicated on the Annual Certified Parameter List*
*which must accompany this certificate to be valid*

#49529
PERMANENT CERTIFICATION NUMBER

April 24, 1990
DATE

Judith N. Yeatman
COMMISSIONER
DEPARTMENT OF ENVIRONMENTAL PROTECTION

**This certification is subject to unannounced laboratory inspections as specified by N.J.A.C. 7:18-2.11(d) and agreed to by the Laboratory Manager on filing the application**

**) BE CONSPICUOUSLY DISPLAYED AT THE LABOR' 'RY WITH THE ANNUAL CERTIFIED PARAMETER L'**

# APPENDIX F

# APPENDIX F
# IT LABORATORY STATEMENT OF QUALIFICATIONS AND QA PROGRAM

California Analytical
Laboratory

# QUALITY ASSURANCE
# PROGRAM PLAN
# FOR
# ENVIRONMENTAL CHEMICAL ANALYSIS

Enseco
A CORNING Company

Prepared By:
Enseco Incorporated

Revision: 3.5
May, 1992

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## 1. INTRODUCTION

Enseco Incorporated (Enseco) of Corning Lab Services Incorporated (CLSI) comprises the largest and most experienced network of environmental testing laboratories in the United States. The Enseco facilities are organized into four geographic regions:

Eastern Region:

- Enseco-East in Somerset, New Jersey
- Enseco-Erco Laboratory in Cambridge, Massachusetts

Central Region:

- Enseco-Rocky Mountain Analytical Laboratory in Arvada, Colorado
- Enseco-Mixed Waste Laboratory in Arvada, Colorado
- Enseco-Houston Technical Service Center in Houston, Texas

Western Region:

- Enseco-California Analytical Laboratory in Sacramento, California
- Enseco-CRL in Garden Grove, California
- Enseco-Air Toxics Laboratory in City of Industry, California
- Enseco-Mobile Laboratories in Garden Grove, California

Wadsworth/ALERT Region:

- Wadsworth/ALERT Laboratories in North Canton, Ohio
- Wadsworth/ALERT Laboratories in Pittsburgh, Pennsylvania
- Wadsworth/ALERT Laboratories in Tampa, Florida

Addresses and telephone numbers for these facilities are listed in Table 1-1.

This document describes the Enseco Quality Assurance policies and procedures related to chemical analysis for environmental pollutants in water, soil, and waste.

## TABLE 1-1

## ENSECO FACILITIES

Eastern Region

Enseco-East
2200 Cottontail Lane
Somerset, NJ 08875
(908) 469-5800
Facsimile (908) 469-7516

Enseco-Erco Laboratory
205 Alewife Brook Parkway
Cambridge, MA 02138
(617) 661-3111
Facsimile (617) 354-5258

Central Region

Enseco-Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611
Facsimile (303) 431-7171

Enseco-Mixed Waste Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611
Facsimile (303) 467-9136

Enseco-Houston Technical Service Center
1420 East North Belt, Ste. 120
Houston, TX 77032
(713) 987-9767
Facsimile (713) 987-9769

Western Region

Enseco-California Analytical Laboratory
2544 Industrial Boulevard
West Sacramento, CA 95691
(916) 372-1393
Facsimile (916) 372-1059

Enseco-CRL
7440 Lincoln Way
Garden Grove, CA 92641
(714) 898-6370
Facsimile (714) 891-5917

Enseco-Air Toxics Laboratory
18501 East Gale Ave, Ste. 130
City of Industry, CA 91748
(818) 965-1006
Facsimile (818) 965-1003

Enseco-Mobile Laboratories
7440 Lincoln Way
Garden Grove, CA 92641
(714) 898-6370
Facsimile (714) 891-5917

**TABLE 1-1**

**ENSECO FACILITIES**

**(Continued)**

Wadsworth/ALERT Region

Wadsworth/ALERT Laboratories
4101 Shuffel Drive, NW
North Canton, OH 44720
(216) 497-9396
Facsimile (216) 497-0772

Wadsworth/ALERT Laboratories
5910 Breckenridge Parkway
Breckenridge II, Suite H
Tampa, FL 33610
(813) 621-0784
Facsimile (813) 623-6021

Wadsworth/ALERT Laboratories
450 William Pitt Way, Bldg. 6
Pittsburgh, PA 15238
(412) 826-5477
Facsimile (412) 826-5571

## 2. QUALITY ASSURANCE POLICY

Enseco's commitment is to meet the client's requirements by providing quality environmental analytical services to both the public and private sectors. The quality management system at Enseco stresses process improvement and error prevention through training and planning. It provides for detection of errors that occur through quality control and auditing. The goal of each laboratory is to generate error-free work through the support of personal standards of performance, the attitude that errors can be prevented, and to devise permanent solutions for problems which are detected. A comprehensive system of measurement and display of key characteristics of the laboratory provides opportunity for continuous improvement. The extensive Quality Assurance program, as part of the quality management system, ensures the production of scientifically sound, legally defensible data of known, documentable and verifiable quality. This program relies on clearly defined objectives, well-documented procedures, a comprehensive audit system, and management support, both Corporate and Regional for its effectiveness.

All work at Enseco is conducted under this QAPP unless another approved program plan, project plan or contract is in place which describes a quality management system appropriate to the client's requirements to generate scientifically sound, legally defensible data of known, documentable and verifiable quality.

## 3. PURPOSE AND SCOPE OF DOCUMENT

### Purpose

This QA Program Plan presents an overview of the essential elements of the Enseco QA program. This plan is modeled along EPA guidelines as outlined in "Interim Guidelines and Specifications for Preparing Quality Assurance Program Plans," QAMS-004/80, December 29, 1980 and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80, February, 1983. Both of these documents have been issued by the Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency (U.S. EPA). Elements above and beyond those specified in these two documents have been included in this QA Program Plan in order to completely describe the Enseco QA/QC system.

### Scope

The Enseco QA program is designed to control and monitor the quality of data generated in Enseco laboratories. The program has four key elements.

- Demonstrating laboratory capability by providing information which documents the overall qualifications of the laboratory to perform environmental analyses;
- Establishing procedures for controlling laboratory operations which measure laboratory and instrument performance on a daily basis;
- Measuring matrix effects to determine the effect of a specific matrix on method performance, and
- Reporting appropriate QC information with the analytical results to enable the end-user to assess the quality of the data.

The specific procedures involved in implementing each aspect of the program are described in this document. An overview of these QC procedures, along with the section number in which each is discussed, is given in Table 3-1.

The QA/QC policies and procedures described herein are designed to eliminate systematic errors and minimize the occurrence of other errors. The QA program forms the framework for minimizing errors, identifying those errors which do occasionally occur, and correcting them at their source. These QA/QC policies and procedures must be coupled with the professional judgment of the technical staff in interpreting the events surrounding the generation of the final result to ensure that quality data is consistently produced, and decisions and corrective actions are fully documented.

In many instances, Enseco participates with its clients in the preparation and evaluation of project-specific Quality Assurance Project Plans (QAPjP). Typically the elements of the Enseco QAPP are incorporated into these documents. In some instances other requirements may be specified. Each QAPjP must be reviewed and signed by the QA Director or his/her designee of the Enseco facility entering into the client agreement to assure that minimum standards of quality exist by which the work can be evaluated as to its scientific and legal integrity. The QA Director must assure that both the analytical testing objectives and regulatory requirements of the project are described in the project plan. In the presence of an approved QAPjP, Enseco laboratories must follow the specific requirements of that project plan which supersedes the Enseco QAPP for any work explicitly associated with that QAPjP.

## Table 3-1

## ELEMENTS OF QA PROGRAM PLAN

| Evaluation Criteria | Operational Elements | Section of QA Plan |
|---|---|---|
| LABORATORY QUALIFICATIONS | Facilities/equipment/staff | * |
| | Written SOPs for all laboratory procedures, including: | |
| | Sample custody | 17 |
| | Calibration procedures | 7 |
| | Analytical procedures | 8 |
| | Data validation | 9 |
| | Documented QA program | 10 |
| | Laboratory certifications | 1-15 |
| | | 12 |
| LABORATORY PERFORMANCE | Check samples | 12 |
| | Laboratory Control Samples | 11 |
| | Calibration data/calibration verification | 8 |
| | Method detection limits | 14 |
| MATRIX EFFECTS | Matrix spike/matrix duplicate/ matrix spike duplicate analyses | 11 |
| | Sample surrogate recoveries | 11 |
| | Standard additions | 11 |
| | Field blanks | 11 |
| | Method detection limits (determined with specific sample matrix) | 11 |
| DATA REPORTING | Data reduction and validation | 10 |
| | Data reporting | 10 |
| | Reporting Limits | 14 |

* Described in a separate document available from each facility.

## 4. DEFINITION OF TERMS

Quality Assurance (QA): the total integrated program for assuring the reliability of data generated in the laboratory.

Quality Control (QC): the routine application of specific, well-documented procedures to ensure the generation of data of known and accepted quality, thus fulfilling the objectives of the QA program.

Quality Assurance Program Plan (QAPP): an assemblage of management policies, objectives, principles, and general procedures outlining the techniques by which the laboratory produces data of known and accepted quality.

Quality Assurance Project Plan (QAPjP): an assemblage of detailed procedures describing how the laboratory will generate data that meet the Data Quality Objective (DQOs) of a specific project.

Standard Operating Procedure (SOP): a detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.

Legally Defensible Data: data which are supported by a QAPP and documentation adequate to reconstruct the analytical process. Legal defensibility is not dependent on the level of deliverables.

Holding Time: the period of time during which a sample can be stored after collection and preservation according to method or client requirements.

Initiate Preparation: the point in time at which the separation of organic extractable compounds or metals from the sample matrix by solvent extraction, acid digestion, or leachate generation is begun.

Initiate Analysis: the point in time at which the sample, extract or digestate is introduced into an instrument or process which complies with the SOP for analysis of the parameter of interest.

Standard Additions (SA): the practice of adding a series of known amounts of an analyte to an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. The practice of SA's is generally used with metal analyses to compensate for the effect of the sample matrix on the accuracy of the analyses.

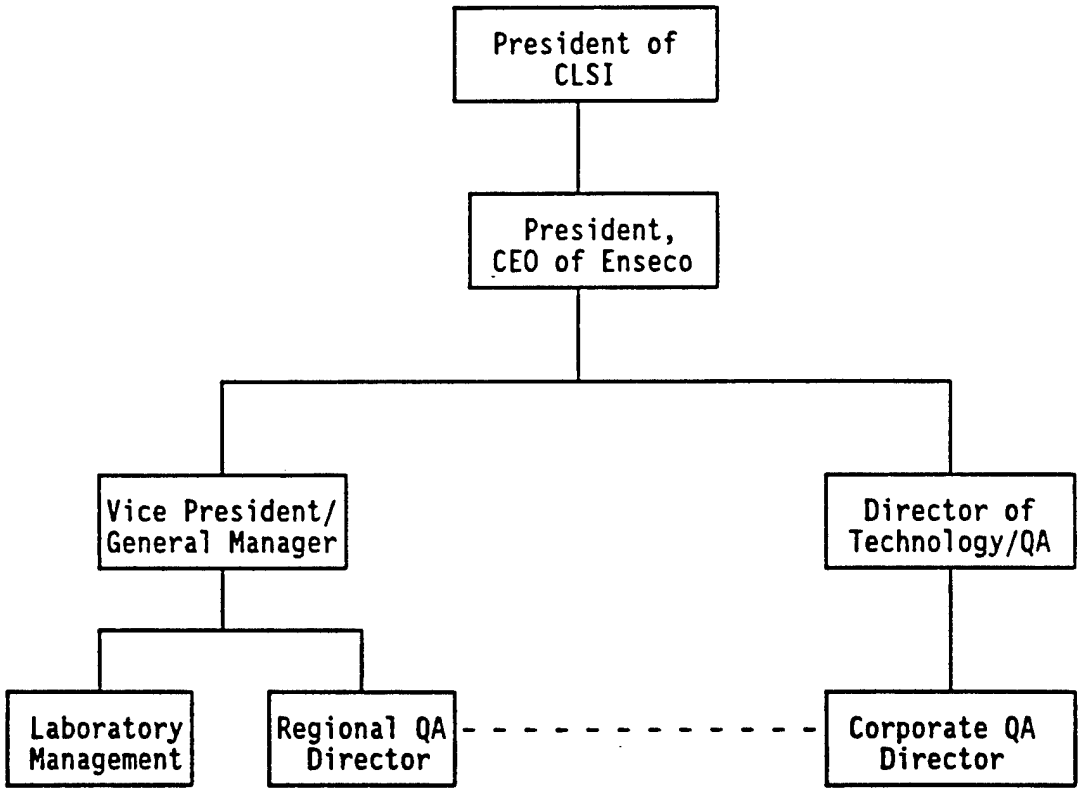
## 5. RESPONSIBILITIES AND AUTHORITIES

Executing an effective QA program in a large and complex multi-laboratory system demands the commitment and attention of both management and staff. The QA effort is administered by the Director of Technology/Quality Assurance who manages the Corporate Quality Assurance Office. The Director of Technology/Quality Assurance reports directly to the President and Chief Executive Officer (CEO) and has the responsibility for overseeing and regulating all laboratory functions (see Figure 5-1). The Corporate QA Director reports to the Director of Technology/Quality Assurance and has the responsibility of the day-to-day functions of the QA office. The QA Office operates independently of all areas generating analytical data to ensure complete objectivity in the evaluation of laboratory operations.

The implementation of the QA program within each region is administered by the Regional QA Director. The Regional QA Director reports to both the Corporate QA Director and to the Vice President/General Manager or the Assistant General Manager who manages the region. Each facility has a QA Director who monitors the day to day QA activities at that facility. The QA Director participates in the facility Quality Improvement Team (QIT) and management team meetings as a full partner of the management team to ensure the policies of the organization with respect to client service and quality are met. In addition, all scientists within the organization play a vital role in assuring the quality of their work. We believe that the success of Enseco is dependent upon the continued commitment of all within the organization to a strong and viable QA Program. The responsibilities and levels of authority within the organization are described below. The descriptions which follow are intended to address the functions required of these positions. Actual position titles may vary among the facilities.

**FIGURE 5-1**

QA ORGANIZATIONAL CHART



## Corporate Quality Assurance Office

### Members

The QA effort within Enseco is directed by the Corporate QA Director under the management of the Director of Technology/ Quality Assurance to carry out the responsibilities of the department.

### Responsibilities

The Corporate QA Director is responsible for:

- Developing and implementing a Corporate QA program that ensures that all data generated in Enseco laboratories are scientifically sound, legally defensible, and of known precision and accuracy;
- Monitoring the QA Plan to ensure compliance with QA objectives in all Enseco laboratories;
- Developing and implementing new QA procedures within the corporation to improve data quality;
- Conducting audits and inspections of all Enseco laboratories on a regular basis, reporting the results of those audits to Regional and Corporate management, and recommending corrective actions as needed to ensure compliance with the Enseco QA Program Plan and/or applicable QA Project Plan;
- Establishing databases that accurately reflect the performance of each of the Enseco laboratories;

- Assisting facility QA Directors in the implementation of the Enseco QA Plan;
- Chairing the Enseco QA Committee, a working committee which includes all of the Regional and facility QA Directors and deals with QA issues on an ongoing basis;
- Monitoring the status of facility certifications;
- Conducting seminars on QA issues for both clients and staff; and
- Promoting sound QA practices within the environmental regulatory and analytical communities.

Authority

Both the Director of Technology/Quality Assurance and the Corporate QA Director have the authority on issues dealing with data quality and have the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. The Director of Technology/Quality Assurance and the Corporate QA Director have the authority to recommend appropriate disciplinary action up to or including suspension or termination of employees on the grounds of dishonesty, incompetence, or repeated non-compliance with QA procedures. In addition, these Corporate Directors have the authority to overrule decisions and actions of the Regional and facility QA Directors and must approve the termination or transfer of any Regional or facility QA Director. The authority of the Corporate QA Director and the Director of Technology/Quality Assurance comes directly from the President of CLSI.

## Regional Quality Assurance Departments

### Members

Each Region has a Regional Quality Assurance Director who reports directly to the Vice President/General Manager and indirectly to the Corporate QA Director.

### Responsibilities

The Regional QA Director is responsible for:

- Overseeing the implementation of the QA Plan within the Region to ensure compliance with the QA objectives;
- Assisting staff in maintaining regulatory analytical compliance;
- Overseeing and assisting the facility QA staff in meeting the responsibilities of the facility Quality Assurance Departments at each facility in the Region as described below;
- Reporting the status of the facility QA programs within the Region to the Corporate QA Director with formal and informal communications;
- Providing training opportunities relating to QA for both QA and laboratory staff;
- Conducting seminars on QA issues for clients;
- Assisting facility QA Directors and managers in resolution of data quality inquiries;
- Assisting the Corporate QA office in the writing of QA policies and procedures;

- Serving as a member of the QA Committee;
- Serving as a channel of communications between the Vice President/General Manager and the facility QA groups; and
- Meeting client requirements.

Authority

The Regional QA Director is the final authority within each region on all issues dealing with data quality. He/she has the authority to require that procedures be amended or discontinued or analyses suspended or repeated. In addition, the Regional QA Director has the authority to overrule decisions and actions of the facility QA Directors and must approve the termination or transfer of any facility QA Director. He/she can make recommendations to the Vice President/General Manager and the Corporate Director of QA regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The Regional QA Director reports to the Vice President/General Manager. The authority of the Regional QA Director comes directly from the Corporate QA Director.

## Facility Quality Assurance Departments

Members

Each facility QA Department is managed by a QA Director. The QA Director reports directly to the laboratory management and the Regional QA Director. The QA Director is supported by a QA staff within the facility.

Responsibilities

The facility QA Director or his/her designee is responsible for:

- Implementing QA policies;
- Actively supporting the implementation of the QA Plan within the laboratory to ensure compliance with QA objectives;
- Assisting in maintaining regulatory analytical compliance;
- Conducting in-house audits to identify potential problems and ensure compliance with written SOPs;
- Establishing databases that reflect the performance of the laboratory and review data;
- Prescribing and monitoring corrective actions;
- Serving as the in-house client representative on all project inquiries involving data quality issues;
- Monitoring the preparation and verification of analytical standards;
- Assisting analysts in the writing of SOPs;
- Approving SOPs in concurrence with management;
- Reporting the status of the laboratory QA program to management and the Regional and Corporate QA Director with formal and informal communications;
- Maintaining records and archives of all QC data, PE results, audit comments, and customer inquiries concerning data quality;
- Assuring that the laboratory staff has access to current SOPs;
- Monitoring laboratory performance including holding times, PE performance, and meeting program and project specific requirements.

- Conducting seminars on QA issues for clients;
- Training laboratory staff on QA principles and requirements;
- Approving QA Project Plans;
- Assisting the Corporate QA office in the writing of QA policies and procedures;
- Serving as a member of the QA Committee;
- Auditing subcontractors; and
- Meeting client requirements.

Authority

The facility QA Director is the final authority within each facility on all issues dealing with data quality. He/she has the authority to require that procedures be amended or discontinued or analyses suspended or repeated. He/she can make recommendations to the Vice President/General Manager and the Regional QA Director regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the facility QA Director comes directly from the Corporate Director of QA through the Regional QA Director.

**Facility Management**

Members

The managers and supervisors who direct the analytical work at each facility are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.

Responsibilities

Facility management is responsible for:

- Actively implementing the Enseco QA Plan within the facility;
- Responding to and implementing corrective actions;
- Following the Corporate ethics statement;
- Maintaining accurate SOPs and enforcing their use in the laboratory;
- Providing training for laboratory staff;
- Maintaining a work environment that emphasizes the importance of data quality;
- Providing management support to the Corporate, Regional, and facility QA departments; and
- Meeting client requirements.

Authority

The managers and supervisors of the facility have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgment, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the facility management comes directly from the Vice President/General Manager.

## Laboratory Personnel

### Members

All facility personnel involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan.

### Responsibilities

Laboratory personnel are responsible for:

- Having a working knowledge of the Enseco QA Plan;
- Ensuring that all work is generated in compliance with the Enseco QA Plan;
- Following the Corporate ethics statement;
- Performing all work according to written SOPs and client-specific QAPjPs;
- Ensuring that all documentation related to their work is complete and accurate;
- Providing management and QA with immediate notification of quality problems; and
- Meeting client requirements.

### Authority

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance of data that fall outside of established QC guidelines or rejection of data for technical reasons that meet established QC guidelines

must be approved by laboratory management and the QA department. Laboratory personnel have the authority to recommend a stop-work order due to quality problems. This recommendation can be made either to their supervisor or to the QA Department. The authority of the laboratory personnel flows from the Vice President/General Manager.

## 6. SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters and polyethylene containers for inorganic/metal parameters (see Appendix I);
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization (see Appendix I);
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed (see Appendix I); and
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met (see Appendix I).

### Sample Containers and Preservatives

Enseco can make available to the client sample containers that are properly cleaned and preserved for use in sample collection. Appropriate containers and preservatives, and minimum sample volumes required for analyzing routine organic, metal, and wet chemistry parameters are listed in Appendix I.

## Holding Times

EPA has established holding time requirements for some analyses. These holding time requirements are listed in Appendix I, along with container and preservative requirements. As indicated in Appendix I, holding time requirements differ depending on the regulatory program. Enseco follows the holding times given in SW-846, Update I of SW-846, 40 CFR Part 136, or Methods of Chemical Analysis of Water & Waste, based on the method source, unless otherwise instructed by the client. CLP holding times are followed when CLP protocols are requested by the client.

## Sample Disposition

Sample disposition procedures, including disposition of empty sample containers, meet Federal and State regulations.

## 7. SAMPLE CUSTODY

Upon receipt by the laboratory, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

All samples are received by the facility's sample control group and are carefully checked for label identification, and completed, accurate chain-of-custody records. Each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

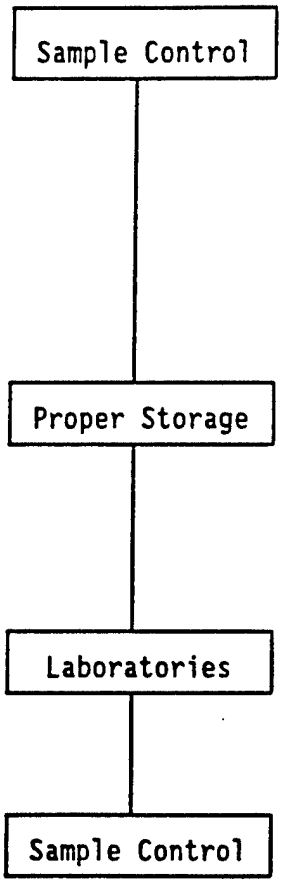
Samples must be transmitted under chain-of-custody both between the field and laboratory and between the laboratory and any subcontractor laboratory as documentation of sample possession. Samples are not transferred to subcontractor laboratories without prior approval of the client.

An example of a Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2. An example of a Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples to subcontractor laboratories is given in Figure 7-3.

Sample bottles provided to the client by Enseco are transmitted under custody.

**FIGURE 7-1**

ENSECO SAMPLE PROCESSING FLOW CHART




* Check and document physical condition of sample
* Log into LIMS
* Verify documentation and parameter assignment

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

* Document analytical work
* Return unused samples to Sample Control

* Return sample to client or arrange for sample disposal

## FIGURE 7-2

Enseco
A Coming Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada CO 80002
303/421 6611 FAX 303/431 7171

CHAIN OF CUSTODY

| | | | | | | |
|---|---|---|---|---|---|---|
| ENSECO CLIENT | | | PACKED BY | | SEAL NUMBER | |
| PROJECT | | | SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY | | CONDITION OF CONTENTS | |
| SAMPLING COMPANY | | | SEALED FOR SHIPPING BY | | INITIAL CONTENTS TEMP | °C |
| SAMPLING DATE | | | SEAL NUMBER | | SAMPLING STATUS ☐ Done ☐ Continuing Until | |
| TEAM LEADER | | | SEAL INTACT UPON RECEIPT BY LAB ☐ Yes ☐ No | | CONTENTS TEMPERATURE UPON RECEIPT BY LAB | °C |

SAMPLE SAFE™ CONDITIONS

| DATE | TIME | SAMPLE ID/DESCRIPTION | SAMPLE TYPE | # CONTAINERS | ANALYSIS PARAMETERS | REMARKS |
|---|---|---|---|---|---|---|
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |

CUSTODY TRANSFERS PRIOR TO SHIPPING

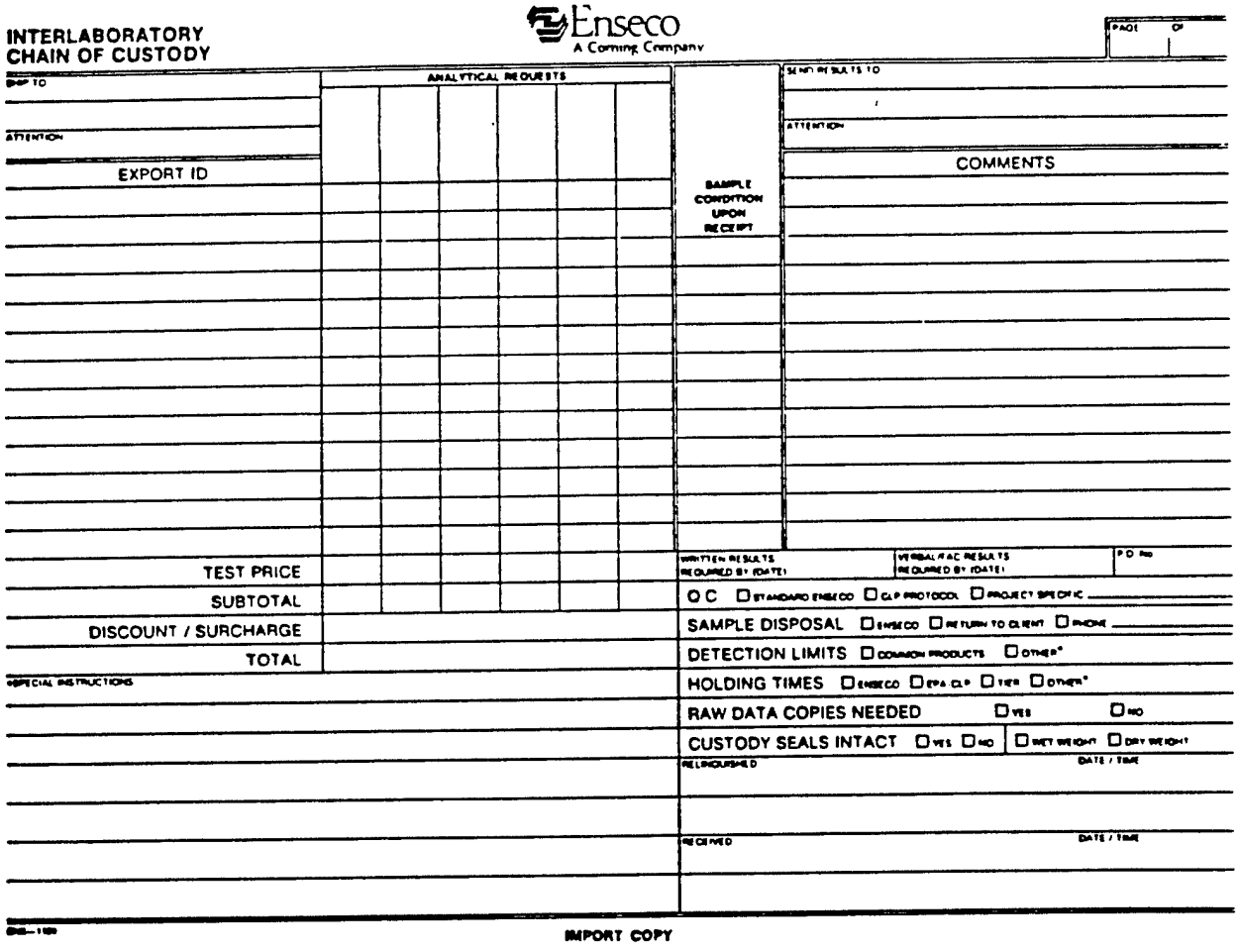
| RELINQUISHED BY (SIGNED) | RECEIVED BY (SIGNED) | DATE | TIME |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

SHIPPING DETAILS

| | | |
|---|---|---|
| DELIVERED TO SHIPPED BY | | |
| METHOD OF SHIPMENT | | AIRBILL NUMBER |
| RECEIVED FOR LAB | SIGNED | DATE/TIME |
| ENSECO PROJECT NUMBER | | |

White - CLIENT Pink - LAB

## FIGURE 7-3



INTERLABORATORY
CHAIN OF CUSTODY

Enseco
A Corning Company

PAGE OF

SHIP TO

ATTENTION

ANALYTICAL REQUESTS

EXPORT ID

SAMPLE CONDITION UPON RECEIPT

SEND RESULTS TO

ATTENTION

COMMENTS

TEST PRICE

SUBTOTAL

DISCOUNT / SURCHARGE

TOTAL

WRITTEN RESULTS REQUIRED BY (DATE)

VERBAL/FAC RESULTS REQUIRED BY (DATE)

P O NO

QC ☐ STANDARD ENSECO ☐ CLP PROTOCOL ☐ PROJECT SPECIFIC

SAMPLE DISPOSAL ☐ ENSECO ☐ RETURN TO CLIENT ☐ PHONE

DETECTION LIMITS ☐ COMMON PRODUCTS ☐ OTHER*

HOLDING TIMES ☐ ENSECO ☐ EPA CLP ☐ YES ☐ OTHER*

RAW DATA COPIES NEEDED ☐ YES ☐ NO

CUSTODY SEALS INTACT ☐ YES ☐ NO ☐ WET WEIGHT ☐ DRY WEIGHT

*SPECIAL INSTRUCTIONS

RELINQUISHED DATE / TIME

RECEIVED DATE / TIME

IMPORT COPY

## 8. CALIBRATION PROCEDURES AND FREQUENCY

### Standard and Reagent Preparation

A critical element in the generation of quality data is the purity or quality and traceability of the standard solutions and reagents used in the analytical operations. Enseco continually monitors the quality of reagents and standard solutions through a series of well-documented procedures..

Primary reference standards and standard solutions used by the laboratories are obtained from an EPA Cooperator Supplier, the National Institute of Standards and Technology, or other reliable commercial sources to ensure the highest purity possible. All standards and standard solutions are tracked to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Standard solutions are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data (initials of preparer and date of preparation). Standards are stored separately from samples.

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used. In addition, bulk solvents are analyzed for undesirable contaminants prior to use in the laboratory. These analyses are documented.

## Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the working range established for the analytical method. The frequency of calibration and calibration verification and the concentration of calibration standards are determined by the manufacturer's guidelines, the analytical method, or the requirements of special contracts.

### Gas Chromatography/Mass Spectrometry (GC/MS)

Prior to analysis of samples, the instrument is tuned with bromofluorobenzene (BFB) for volatile compounds and decafluorotriphenylphosphine (DFTPP) for semivolatile compounds or other tune criteria as specified by the method used. No samples are analyzed until the instrument has met the tuning criteria of the method.

In general, the instrument is then calibrated for all target compounds. An initial calibration curve is produced to define the working range. This initial calibration is evaluated on a daily

basis (when samples are analyzed) to ensure that the system is within calibration. If the continuing calibration standard does not meet the established criteria, corrective action is taken, which may include recalibration.

Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to performance of analyses. Initial calibration consists of analyzing standards across the working range. The calibration is checked on a daily basis (when samples are analyzed) to ensure that the system remains within specifications. In addition, continuing calibration checks are performed at frequencies required by the method used. If the calibration checks do not meet established criteria, corrective action is taken which may include recalibration and reanalysis of samples. The corrective action procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted.

Metals

Metals analysis basically involves two types of analytical instrumentation: inductively coupled argon plasma emission spectroscopy (ICP), and atomic absorption spectroscopy (AA).

ICP

Each ICP is calibrated prior to any analyses being performed using criteria prescribed in the analytical method employed. The calibration is then verified using standards from an independent source. The working range of the instrument is established once every quarter using a linear range verification check standard. No values are reported out of the linear range without dilution.

A calibration curve is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration is monitored throughout the day by analyzing a Continuing Calibration Blank (CCB) and a Continuing Calibration Verification standard (CCV). If the verification standard and blank do not meet established criteria, corrective action must be performed. The corrective action procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted.

An interelement check standard is analyzed at the beginning and end of each analytical run on the ICP to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

AA

Each AA unit is calibrated prior to any analyses being conducted. A calibration curve is prepared with a minimum of a calibration blank and three standards and then verified with a standard that has been

prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then verified on an ongoing basis with a calibration blank (CCB) and a CCV. If the ongoing calibration standard and blank do not meet established acceptance criteria, corrective action must be performed. The corrective action procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted. For GFAA, all samples are spiked at the instrument to verify the absence of matrix effects or interferences. The method of standard additions or sample dilution is used when matrix interferences are present as determined by the results of the analytical spike.

Wet Chemistry

The field of conventional, non-metals analysis (wet chemistry) involves a variety of instrumental and wet chemical techniques. While calibration and standardization procedures vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply universally. Each system is calibrated prior to analyses being conducted. Calibration consists of defining the working range by use of a series of standard solutions and identifying potential interferences. The calibration is checked on an ongoing basis to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, corrective action must be performed. The corrective action procedures include examination of instrument performance and analysis information, consultation with the

Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted. Continuing calibrations are not performed for non-instrumental methods such as Total Dissolved Solids.

## 9. ANALYTICAL PROCEDURES

Most analyses performed by Enseco are driven by regulatory concerns. Therefore, methods used at Enseco predominantly originate from regulatory agencies. Generally the methods used are those specified by the U.S. EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including chlorinated dioxins and furans.
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," 40 CFR, Part 136.
- "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020 (revised March, 1983 or subsequent revision).
- "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA-600/4-82-057 (July, 1982).
- "Test Methods for Evaluating Solid Waste" (SW-846), 2nd Edition (revised), Update I (1984), Update II (1985), 3rd Edition (1986), Update I (1989), Office of Solid Waste and Emergency Response, U.S. EPA.
- "Standard Methods for the Examination of Water and Wastewater," 16th Edition (1985) and 17th Edition (1989) American Public Health Association, American Water Works Association, Water Pollution Control Federation, Washington, DC (1985).
- "Official Methods of Analysis," 14th Edition, Association of Official Analytical Chemists, Arlington, VA (1984).
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September, 1986 or subsequent revision).
- "Annual Book of ASTM Standards," Volumes 11.01, 11.02, 11.03, and 11.04, American Society for Testing and Materials (ASTM), Philadelphia, PA (1987).

- "Techniques of Water Resources Investigations of the United States Geological Survey (USGS), Book 5, Laboratory Analysis," USGS, Washington, DC (1979).

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, the type of matrix to be analyzed, and the regulatory program. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. Enseco may deviate from standard methodologies if necessary or appropriate due to the nature or composition of the sample, based on the reasonable judgment of Enseco.

## 10. DATA REDUCTION, VALIDATION, AND REPORTING

### Data Reduction and Validation

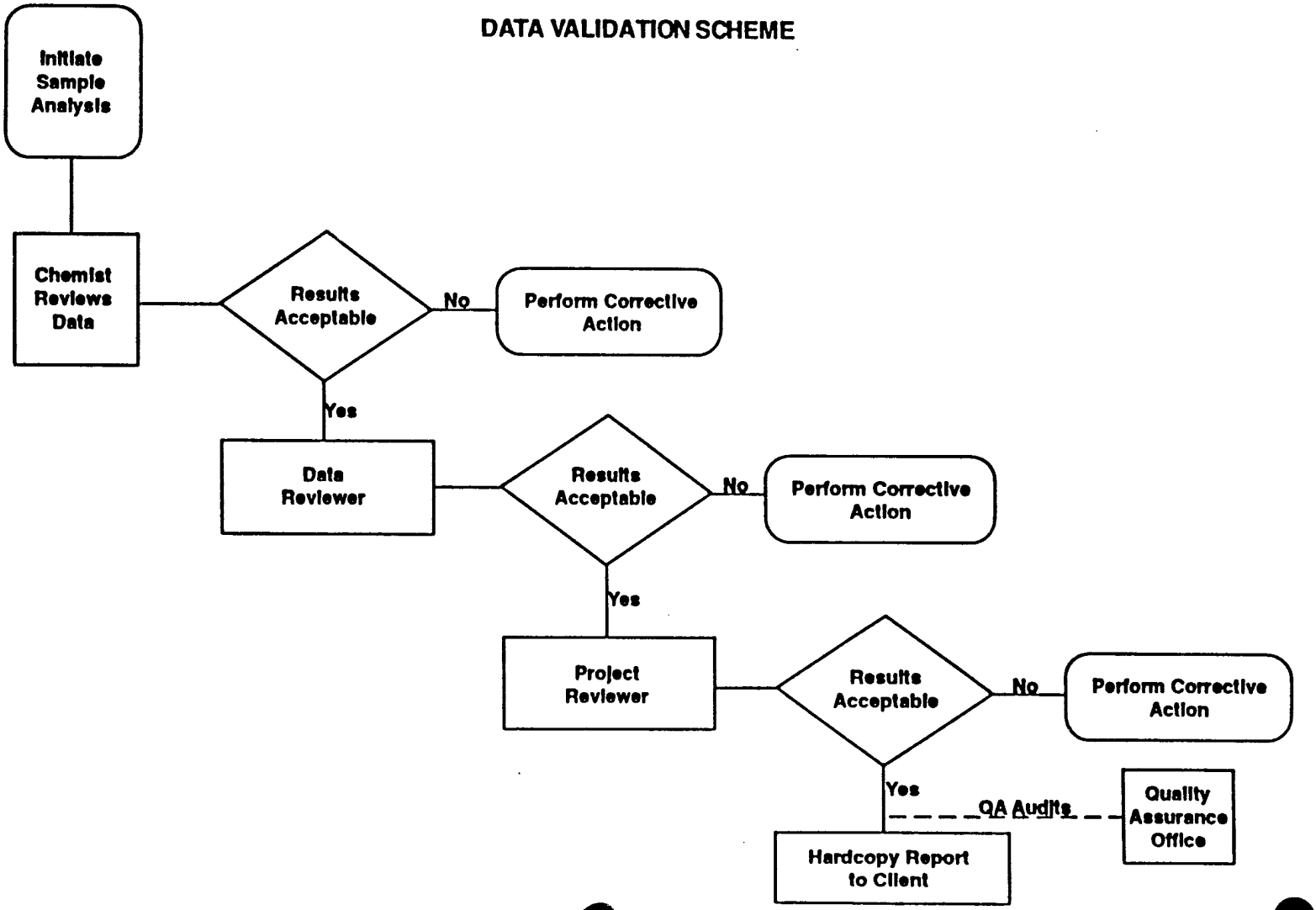
All analytical data generated within Enseco laboratories are extensively reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three documented reviews (see Figure 10-1). The first review is performed by the person generating the data. This review assures that the work is done correctly the first time. The second review is an independent technical review of the data to ensure the work is error-free and to provide a mechanism to correct errors that are missed during the first review. The third review serves to ensure that the completed project meets the client's specifications. In each stage, the review process is documented by the signature of the reviewer and the date reviewed. In addition to the three reviews, a periodic random data audit is performed by the QA Department. This is described in Section 12. This review process is described below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;

## FIGURE 10-1

### DATA VALIDATION SCHEME



- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Analytical and/or preparation holding times are met;
- Special sample preparation and analytical requirements have been met; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, holding times are documented, etc.).

The data reduction and validation steps are documented, signed and dated by the analyst. The analyst then passes the data package to an independent reviewer, who performs a second review.

The second review is performed by a supervisor or data reviewer whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are appropriate to the method and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; holding times are documented, etc.);

- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

The second review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

An important element of the second review is the documentation of any errors that have been identified and corrected during the review process. Enseco believes that the data package submitted by the analyst should be error-free. Errors that are found are documented. The cause of the errors is then addressed by the supervisor with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

The second data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the project is reviewed for completeness and to ensure that the data meet the overall objectives of the project. This review is labeled the third review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

General Discussion: Description of sample types, tests performed, any problems encountered and general comments are given.

Analytical Data: Data are reported by sample or by test with the appropriate significant figures and reporting limits, adjusted for dilution. Pertinent information including dates sampled, received, prepared, and extracted are provided.

Laboratory Performance QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic, metals, and pertinent wet chemistry parameters are given.

Matrix-Specific QC Information: Results of any sample duplicates, matrix spikes, matrix spike duplicates or other project-specific QC requested by the client are also reported. The results include

supporting information such as amount spiked, percent recovery or percent difference.

Methodology: Reference for analytical methodology used is cited.

Other Deliverables: Other deliverables available include disk deliverables, sample raw data packages, complete deliverable packages, and custom report formats.

## 11. INTERNAL QC CHECKS

The Enseco QA/QC program controls, monitors, and assesses data quality with internal QC checks. Internal QC checks are used to answer three questions:

1) Are laboratory operations "in control," (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

3) What effect do field conditions have on the analytical results?

The first question is answered by Laboratory Performance QC. Laboratory performance QC is based on the use of a standard, control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with Matrix-Specific QC. Matrix-Specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes, matrix duplicates, and matrix spike duplicates. This information is used to assess the effect of the matrix on analytical data.

The third question is addressed with Field QC samples. These samples, including field blanks, trip blanks, equipment blanks, field duplicates, and field splits monitor the collection, transport and storage of environmental samples.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. Matrix-Specific QC is available as an option to the client and should be specified based on the types of matrices to be analyzed and the Data Quality Objectives (DQOs) and regulatory requirements of the project. A complete discussion of these programs follows.

## Laboratory Performance QC Program

Laboratory Performance QC is performed for every routine Enseco analysis to demonstrate that laboratory operations are "in control". The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks, and
- The use of calibration standards to assure that both qualitative identification and quantitative measurements are within control limits.

The Laboratory Control Sample program is discussed below. Please refer to Section 8 of this manual for a discussion of calibration procedures.

### Laboratory Control Samples (LCS)

Laboratory Control Samples (LCS) are well-characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. Three types of LCS are routinely analyzed: Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks. Certain LCS (DCS, SCS) are used to monitor the precision and accuracy of the analytical process, independent of matrix

effects. Other LCS (method blanks) are used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data. Each of these LCS are described below.

The results of the LCS are compared to well-defined laboratory acceptance criteria to determine whether the laboratory system is "in control." Controlling lab operations with LCS (as opposed to matrix spike/matrix spike duplicate samples), offers the advantage of being able to differentiate quality problems due to laboratory procedural errors from those due to matrix effects. As a result, procedural errors can be identified and corrected by the analyst at the bench, without waiting for extensive senior level review or costly and time-consuming reanalysis of the sample.

Duplicate Control Samples (DCS)

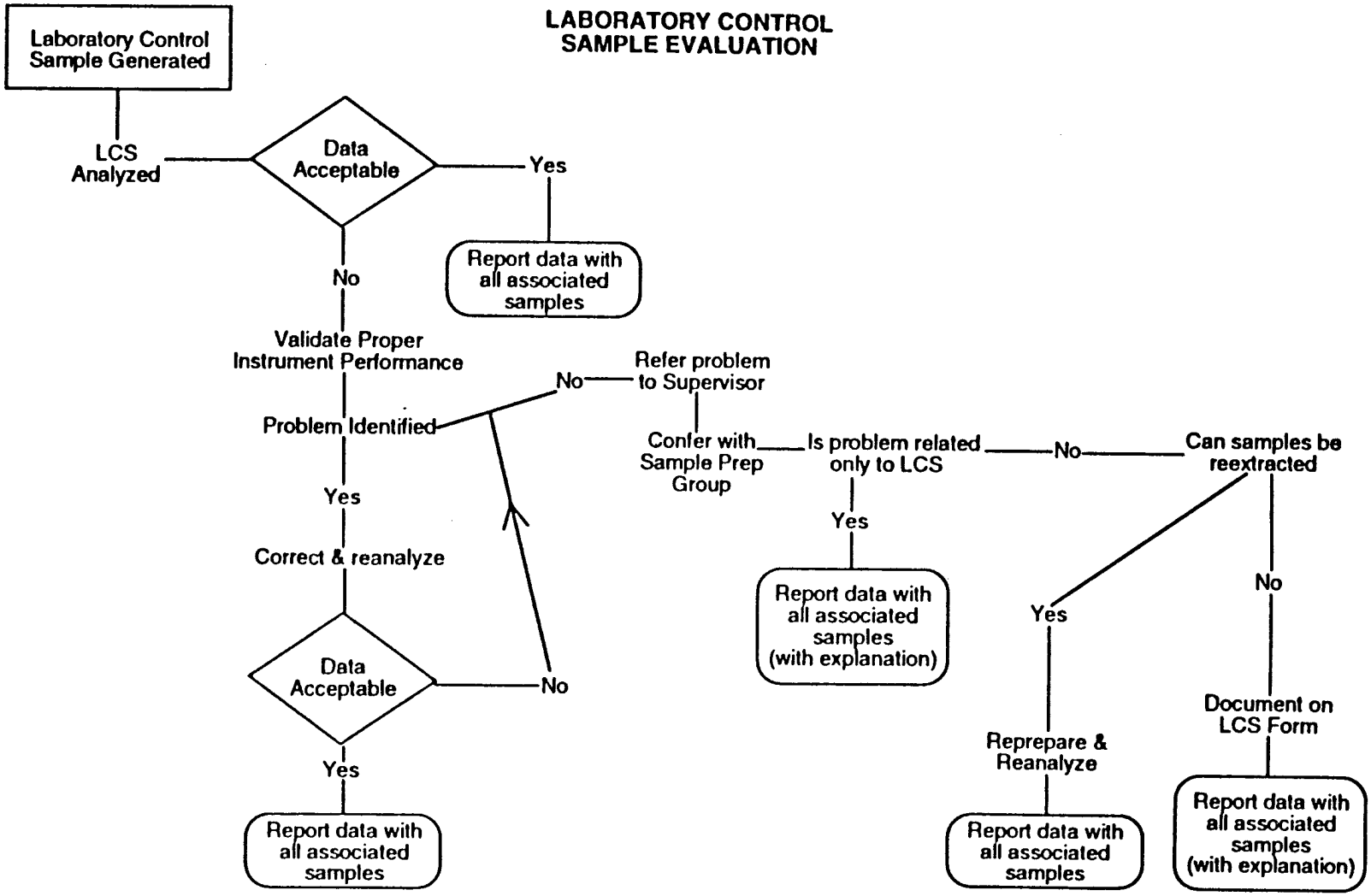
Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS set consists of a standard, control matrix that is spiked with a group of target compounds representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Accuracy data (average recovery of each analyte in the DCS pair) and precision data (Relative Percent Difference [RPD] between each analyte in the DCS pair) are compared to control limits that have been established for each of the analytes contained in the DCS. Initially, control limits

for analytes spiked into the DCS are taken directly from the CLP program or published methods. If published limits are not available, either Enseco historical data are used to set the control limits or limits are estimated from method validation data. The control limits are recalculated periodically, as sufficient laboratory data become available. Control limits for accuracy for each analyte are based on the historical average recovery (mean of the average recoveries of the DCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte are based on the historical RPD. Acceptable RPDs range from zero (no difference between DCS results) to the average RPD plus three standard deviation units. Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. Data generated with a DCS pair which falls outside of the control limits are considered suspect and corrective action must be performed. The procedure used to evaluate data from control samples is given in Figure 11-1. The corrective action procedures include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

DCS have been established for each routine analytical method. Reagent water is used as the control matrix for the analysis of aqueous samples and deionized water leachates of solids for wet chemistry parameters. The DCS compounds are spiked into reagent water and carried through the appropriate steps of the analysis. The control matrix for solids samples for organic analyses is typically standard Ottawa sand, an ASTM approved material for use in highway construction, due to its homogeneity. The DCS compounds are spiked into the Ottawa sand and carried through the appropriate steps of the analysis. For metal analyses, a spiked solid matrix from a commercial source is used. The DCS for some wet chemistry

## FIGURE 11-1
### LABORATORY CONTROL SAMPLE EVALUATION



Analytical data that are generated with an SCS which falls within the control limits are judged to be in control. Data that are generated with an SCS which falls outside of acceptance criteria are considered suspect and corrective action must be performed. The protocols for evaluating SCS are identical to those established for DCS (see Figure 11-1). SCS recovery (accuracy) data are archived in the LIMS. In addition, the associated SCS data are reported with each set of sample results to enable the client to make a quality assessment of the data.

Method Blanks

Method blanks, also known as analytical, process or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, clean-up and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

Ideally, the concentration of target analytes in the blank should be below the Reporting Limit for that analyte. In practice, however, some common laboratory solvents and metals are difficult to eliminate to the levels commonly reported in environmental analyses. Therefore, criteria for determining blank acceptability must be based on consideration of the analytical techniques used, analytes reported, and Reporting Limits required.

For organic analyses, the concentration of target analytes in the blank must be below the Reporting Limit for that analyte in order for the blank to be considered acceptable. An exception is made for common laboratory contaminants (methylene chloride, acetone, 2-butanone, and phthalate esters) which may be present in the blank at up to 5 times the Reporting Limit and still be considered acceptable. This policy has been established in recognition of the fact that these compounds are frequently found at low levels in method blanks due to the materials used in the collection, preparation, and analysis of samples for organic parameters.

For non-routine organic analyses, other components may be established as common contaminants for that particular analysis. For example, naphthalene is frequently found in PAH-SIM analyses. If, upon thorough review of the method during validation it is deemed impossible to eliminate trace amounts of analytes from the process, these analytes are likewise allowed at up to 5 times the reporting limit.

For metals and Wet Chemistry analyses, where the Reporting Limits are typically near the Instrument Detection Limit (IDL), the policy is that the concentration of the target analytes in the blank must be below two times the Reporting Limit. If the blank value for a target analyte lies below the Reporting Limit, the analyte is reported with no flag on the associated sample data. If the blank value lies between the Reporting Limit and two times the Reporting Limit, the analyte in the associated samples is flagged to indicate contamination was present in the blank. A blank containing an analyte(s) above two times the Reporting Limit is considered unacceptable unless the lowest concentration of the analyte in the associated samples is at least ten times the blank concentration or the concentration of the analyte in all samples associated with the blank is below the reporting limit.

In addition, for Wet Chemistry tests, the method SOP directs how the blank is treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a preparation blank is also analyzed to determine the extent of contamination or background interference. Some methods require that the concentration of analyte found in this preparation blank be subtracted from the concentration of the analyte found in any associated sample prior to calculating the final result. Blanks have no application or significance for some Wet Chemistry parameters (e.g. pH).

If the blank for any test does not meet acceptance criteria, the source of contamination must be investigated and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank, and/or repreparation and reanalysis of the blank and all associated samples. If a blank meets the criteria, but has analytes above the reporting limit, investigation should occur to determine whether any corrective action could eliminate an ongoing source of target analytes. Additional actions or explicit corrective action procedures detailed in protocols, methods or project-specific project plans must be followed when applicable.

For organic and metals analyses, and selected Wet Chemistry tests, method blank results are reported with each set of sample results. Sample results are <u>not</u> corrected for blank contamination unless required by the analytical method or requested by the client. Occasionally, due to limited sample volume or other constraints, the laboratory reports data associated with an unacceptable blank. In these cases, the actual

observed value (if any) is reported in the method blank. Sample results for any analyte(s) observed in the blank are flagged to indicate contamination was present in the associated method blank.

**Matrix-Specific QC**

Matrix-Specific QC is used to assess the effects of a sample matrix on the analytical data. The main elements of Matrix-Specific QC are:

- The analysis of matrix spikes, matrix duplicates, and matrix spike duplicates;
- Monitoring the recovery of surrogate compounds from environmental samples;
- Monitoring the results of standard additions in environmental samples; and
- The determination of method detection limits in a specific matrix.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. At a minimum, the laboratories analyze matrix spikes, matrix spike duplicates or matrix duplicates at the frequency specified by the method, in order to meet the regulatory requirements of the method. These data are only reported when requested. These data are not used to control the laboratory. In order to ensure that the data generated meet all Data Quality Objectives, Enseco recommends that its clients request and include Matrix-Specific QC for their samples that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Spikes, Matrix Duplicates, and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of representative target analytes have been added. The MS, in addition to an unspiked aliquot, is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Duplicate (MD) is an environmental sample that is divided into two separate aliquots. The aliquots are processed separately and the results compared to determine the effects of the matrix on the precision of the analysis. Results are expressed as RPD.

A Matrix Spike Duplicate (MSD) is an environmental sample that is divided into two separate aliquots, each of which is spiked with known concentrations of analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

Surrogate Recoveries

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis. Results are reported in terms of percent recovery.

Enseco routinely adds surrogates to samples requiring GC or GC/MS analysis and reports these surrogate recoveries to the client. The laboratory does not control its operations based on surrogate recoveries in environmental samples, however individual methods may dictate sample reanalyses based on surrogate criteria. When required by regulations, these method requirements supersede Enseco practices. As discussed earlier in this section, Enseco controls its analytical systems based on the results of Laboratory Control Samples. The surrogate recoveries are primarily used by the laboratory to assess matrix effects. However, obvious problems with sample preparation and analysis (e.g. evaporation to dryness, leaking septum, etc.) which can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

Field QC

Field QC are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. These include trip blanks, equipment blanks and field blanks. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample container, remains unopened in the field, and then is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. An equipment blank is blank water that is poured through the sample collection device to check the adequacy of the cleaning procedures for the sampling equipment. The blank water used to generate the equipment blank may be provided by the laboratory. The results from field QC samples are reported to the client as samples in the same concentration units as the samples. Field blanks are samples of the same or similar matrix exposed to the sampling environment at the time of sampling. No

correction of the analytical data is done in the laboratory based on the analysis of field QC samples.

Matrix-Specific Detection Limits

Method Detection Limits (MDL's) determined on a specific sample matrix are called Matrix-Specific Detection Limits. See Section 14 for a discussion of detection and reporting limits.

## 12. PERFORMANCE AND SYSTEM AUDITS

Enseco laboratories participate in a variety of federal and state programs that subject each of the laboratories to stringent system and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. The laboratories are approved or certified to perform environmental analyses under various programs (e.g., those administered by the U.S. Department of Energy, U.S. Air Force, U.S. Navy, and over 20 states). The most current list of certifications held by each laboratory is available upon request.

In addition to external audits conducted by certifying agencies or clients, Enseco regularly conducts the following internal audits:

- Data audits of randomly selected projects are reported. The frequency is determined by the error rate found. This is referred to as the QA data audit (see Figure 10-1). This audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. Any problems identified require corrective actions.

- The facility QA Director conducts a system audit periodically. These audits may be coordinated at the regional level. Individual laboratory groups conduct semiannual self-audits of their systems. These system audits monitor the conformance of the laboratory to the QA program and include assessment of facilities, staff, SOPs, sample management, and general documentation procedures.

- The facility QA Director is responsible for conducting compliance audits of each laboratory group to assess compliance with specific contracts, SOPs, and other requirements. Each laboratory group shall be audited at least once per year.

- Periodic (at least yearly) audits conducted by the Corporate QA Office. These audits provide an independent review of the system for compliance with the QA program and assess resolution of previously identified problems.

- Special audits by the facility or Regional QA Director or Corporate QA Office when a problem is suspected.

Enseco laboratories also routinely analyze check samples as described below:

- Laboratory Control Samples (DCS, SCS, and method blanks) are analyzed at a frequency equal to at least 5% of the total number of samples analyzed (see Section 11).

- Enseco laboratories participate in the analyses of EPA check samples provided under the Water Supply (WS) and Water Pollution (WP) Performance Evaluation Studies. The results of these PE samples are tabulated by the Corporate QA Office to identify performance trends within the Enseco laboratories.

- The laboratories participate in multiple state certification programs which require that PE samples be analyzed periodically.

- Blind check samples from an independent commercial firm are sent to the laboratories periodically by the Corporate QA Office.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required. Corrective action reports are prepared to document the investigation of these results and corrective actions implemented to correct any deficiencies revealed by these programs.

In both the system and performance audit processes, the proper implementation of corrective actions must be assured to effect permanent solutions to problems detected.

## 13. PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Laboratory personnel are trained in routine maintenance procedures for most instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has SOPs on file that describe preventive maintenance procedures and schedules. The laboratories also maintain detailed logbooks documenting the preventive maintenance and repairs performed on each analytical instrument.

## 14. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY AND DETERMINE REPORTING LIMITS

### Data Quality Assessment

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with the upper control limit established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation(s) of "n" measurements of "x" is commonly used to estimate precision and is calculated as follows:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation, which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$RSD = 100\ (s/\bar{x})$$

where: RSD = relative standard deviation

$s$ = standard deviation

$\bar{x}$ = mean

In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

$D_1$ = first sample value

$D_2$ = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using LCS, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing LCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where: x = the observed value of measurement

T = "true" value

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all analytical results and data specified for the project. In addition, all data are compared to project requirements to ensure that specifications were met. Any deviations are reported in the report narrative.

The percent completeness for each set of samples can be calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

where valid data are determined by the data acceptance criteria defined in the project plan.

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

## Reporting Limits

Assuring the validity of quantitative measurements at low concentrations is an extremely difficult technical problem. With regulatory action levels being pushed lower and lower, the validity of any given measurement becomes even more important. The consequences of false positive or false negative data can be significant.

A number of terms have been used, by the EPA and other technical groups, to express the lowest concentration of an analyte which can be measured. Some of these terms, their definitions, and sources are listed in Table 14-1. A graphical representation of these terms is given in Figure 14-1.

Enseco has established a Reporting Limit (RL) for each analyte in each method. These Reporting Limits were established by collecting Method Detection Limit (MDL) data for organic and wet chemistry analyses and Instrument Detection Limit (IDL) data for metals analyses from each Enseco laboratory. The MDL data were collected using the procedures described in 40 CFR 136 Appendix B. IDL data were calculated using the procedures outlined in the EPA Contract Laboratory Program (CLP) Statement of Work dated 7/88. The MDL/IDL data were then compared to various limits published in EPA methods and in the regulations. For example for Volatile Organics, the MDL data generated in Enseco

laboratories were compared to the Practical Quantitation Limits (PQLs) published in SW-846 method 8240; the PQLs contained in the July 9, 1987, Federal Register Final Rulemaking on Appendix IX; the Contract Required Quantitation Limits (CRQLs) in the CLP Method for Volatile Organics; and the MDLs in Method 624. Then a Reporting Limit for each analyte was established which considered all of this information. The Reporting Limit was set at a level above which we were confident that our laboratories could detect and quantify the analyte consistently. Using this procedure, the Reporting Limits established are generally between 2 to 5 times the laboratory MDL or IDL. This range is consistent with the American Chemical Society definition for the Limit of Quantitation (LOQ). (See Table 14-1)

Enseco routinely reports results below the reporting limit as Not Detected (ND) because, by definition, the reliability of the data at that level is questionable. As an option, Enseco can report data below the reporting limit and flag the data as estimated. Reporting limits are adjusted for sample dilution.

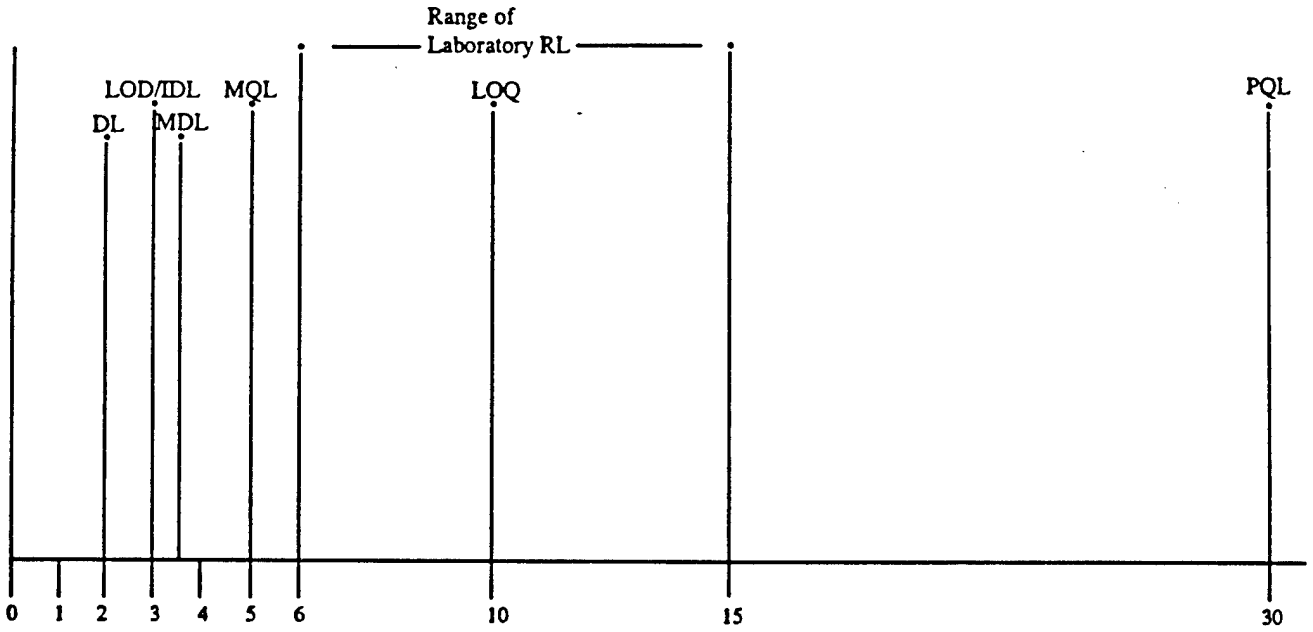
TABLE 14-1

DEFINITION OF DETECTION LIMIT TERMS

| | DEFINITION | DETERMINATION | CALCULATION | SOURCE |
|---|---|---|---|---|
| Detection Limit (DL) | The concentration which is distinctly detectable above, but close to a blank. | Analysis of replicate standards | Two times the standard deviation | Methods for Chemical Analysis of Water and Wastes |
| Limit of Detection (LOD) | The lowest concentration that can be determined to be statistically different from a blank | Analysis of replicate samples | Three times the standard deviation | ACS Definition |
| Method Detection Limit (MDL) | The minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. | Analysis of a minimum of seven replicates spiked at 1 to 5 times the expected detection limit. | The standard deviation times the Student t-value at the desired confidence level. (For seven replicates, the value is 3.14) | 40 CFR 136 Definition for EPA Water Programs |
| Instrument Detection Limit (IDL) | The smallest signal above background noise that an instrument can detect reliably. | Analysis of seven replicate standards on three non-consecutive days. | Three times the standard deviation | Contract Laboratory Program |
| Method Quantitation Limit (MQL) | The minimum concentration of a substance that can be measured and reported | Analysis of replicate samples | Five times the standard deviation | SW-846 |
| Limit of Quantitation (LOQ) | The level above which quantitative results may be obtained with a specified degree of confidence | Analysis of replicate samples | Ten times the standard deviation | ACS Definition |
| Practical Quantitation Limit (PQL) | The lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions | Interlaboratory analysis of check samples | 1) Ten times the MDL<br>2) Value where 80% of laboratories are within 20% of the true value | RCRA<br>SDWA Program |
| Contract Required Detection Limit (CRDL) | Reporting limit specified for laboratories under contract to the EPA for Superfund activities | Unknown | Unknown | Contract Laboratory Program |

## FIGURE 14-1

### Graphical Representation of Detection Limit Terms (See Table 14-1 for Definitions)




NOTE: The values along the horizontal "Standard Deviation (SD)" axis are approximate values and are meant to show the relative, not absolute, relationship between the terms.

## 15. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable limits for precision and accuracy;
- Blanks contain contaminants above acceptable levels;
- Undesirable trends are detected in DCS and SCS recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Every effort must be made to determine the cause of the problem so that a permanent solution can be implemented. Once resolved, full documentation of the corrective action procedure is filed with the project records.

Investigations made by laboratory or QA personnel that result in corrective actions affecting more than one project must be documented and reported in the monthly QA report to management. Documentation of investigations of negative performance on PE samples and corrective actions taken is forwarded to the appropriate certifying agencies when required. These reports are always included in the monthly reports to management.

## 16. QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Facility QA Directors submit extensive monthly reports to the Regional QA Director who is responsible for submitting the regional report to the Vice President/General Manager and the Corporate QA office. These reports include:

- Results of site visits and audits by regulatory agencies and clients including the laboratory's response to deficiencies or action items required by the auditors;
- Results of internal audits including facility audits, contract compliance audits and periodic data audits;
- Performance evaluation sample results and corrective action reports;
- Summary of certification activity including new certifications applied for, certifications renewed and any actions taken by certifying agencies (suspensions, decertifications, probations or reinstatements);
- Discussion of specific client inquiries including summary of the issue, resolution, and correspondence between the client and the laboratory;
- Holding Time Violations, by facility and by department including narrative discussion of problem areas and corrective actions implemented;
- Performance on major contracts;
- Narrative including comments and recommendations on any pertinent issues.

The Corporate QA Director regularly reports on the status of the QA Program to each Vice President/General Manager, to the Director of Technology/Quality Assurance, and to the President and CEO. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

## 17. LABORATORY DOCUMENTATION

Complete and accurate documentation of analytical and procedural information is an important part of the QA program. The following describes different types of documentation used in the Enseco laboratories.

### Standard Operating Procedures

Details of analytical and QC protocols are contained in Standard Operating Procedures (SOPs). SOPs are documents that contain detailed proprietary information on how to perform a laboratory procedure. Enseco has laboratory SOPs that describe:

- Performance of an Analytical Method;
- Preparation of Standards and Reagents;
- Equipment Operation, Calibration, and Maintenance; and
- General Laboratory Procedures.

Examples of the elements contained in these SOPs are given in Appendix II.

All SOPs are approved by the QA Department in concurrence with management, as documented by their signatures, before being implemented. The distribution of current SOPs and archiving of outdated ones is controlled through the QA Department.

Because of the detailed nature of SOPs, Enseco considers them to be proprietary documents. SOPs are available for review at each location.

## LIMS

Enseco laboratories rely on a customized Laboratory Information Management System (LIMS) as the primary database. Client information, sample results, and QC results are all stored in the LIMS. Reports are generated directly from the database to eliminate transcription errors.

## Laboratory Bench Sheets/Notebooks

Laboratory bench sheets or notebooks are used to document information from routine laboratory operations, including sample preparation and analysis. The information is recorded in a complete and organized manner such that the analysis can be reconstructed, if necessary. Portions of information from the bench sheet or notebook are also stored in the LIMS. Laboratory notebooks are also used to document information such as methods development information. Each bench sheet or notebook page is initialed and dated as information is entered.

## Control Charts

Enseco laboratories use control charts to visually track precision and accuracy data. These control charts are used to identify trends in the analyses which may indicate a problem with the analytical procedure. When an adverse trend is detected, corrective action is performed.

## Anomalies

Any situation which is outside of the normal scope of operations, as described in the laboratory SOPs, is documented. Examples of anomalous situations include: formation of a precipitate in an extract; formation of an emulsion during an extraction step; or missed holding times. These situations are documented to enable a thorough review of the data to occur. This documentation is maintained as part of the project record.

Out-of-Control situations are also documented. An Out-of-Control situation occurs when QC data fall outside of established control limits. At a minimum, the documentation associated with an Out-of-Control situation is reviewed by the supervisor. Out-of-Control situations trigger Corrective Action. Corrective Actions taken are also documented. The QA department must be notified when corrective actions affect more than an isolated occurrence of an event.

## Project Files

The project file consists of a project summary file and a raw data file. The project summary file includes correspondence from the client,

(letters, phone logs, contracts, project plans) copies of preliminary and final reports, chain of custody, air bills, level 3 review checklists, QA review checklist when applicable and the summary file inventory. The raw data file includes sample data, QC data, benchsheets, level 1 and level 2 review checklists, instrument logbook pages pertinent to the project and the raw data file inventory. Contracts, project plans, calibration data and QC data may be stored separately from the project record. All project records must contain cross-references to any information stored separately from the project record. When a project is complete, all records are passed to the Document Custodian who inventories the file, checks for completeness, and puts the file into document archive.

## Training Records

Employees participate in structured training which includes learning job skills; Environmental Health and Safety, First Aid and Hazard Communication training; quality training and other support skills (e.g. LIMS). Employee participation in and completion of company-sponsored or company-directed training programs must be documented.

# APPENDIX I

## MAXIMUM HOLDING TIMES AND SAMPLE COLLECTION/PRESERVATION INFORMATION

Sources:

Tables A-E
40 CFR Part 136
Methods for Chemical Analysis of Water and Wastes
SW-846, 3rd Edition, Update I
State of California Leaking Underground
Fuel Tank Field Manual, May 1988

Table F:
Contract Laboratory Program Statement of
Work for Organic Analysis dated 3/90 (as amended)
Contract Laboratory Program Statement of
Work for Inorganic Analysis dated 7/88

Table G:
Federal Register, June 29, 1990

## A. VOLATILE ORGANICS

| Matrix | Holding Time (From Date Sampled) | Container | Preservative | Minimum Sample Size |
|---|---|---|---|---|
| WATER SAMPLES | | | | |
| No Residual Chlorine Present | 14 days | 3 40 mL vials with Teflon lined septum caps | HCl to pH<2, 4°C | 40 mL |
| Residual Chlorine Present | 14 days | 3 40 mL vials with Teflon lined septum caps | 4 drops of 10% sodium thiosulfate, HCl to pH<2, 4°C | 40 mL |
| Acrolein and Acrylonitrile | 14 days | 3 40 mL vials with Teflon lined septum caps | Adjust to pH 4-5, 4°C | 40 mL |
| SOIL/SEDIMENTS AND SLUDGES | 14 days | Glass jar with Teflon liner or core tube | 4°C | 10 g |
| CONCENTRATED WASTE SAMPLES | 14 days | Glass jar with Teflon liner or core tube | None | 10 g |

The above information applies to the following parameters and methods:

| Parameter | Method |
|---|---|
| Volatile Halocarbons | 601/8010 (GC) |
| Volatile Aromatics | 602/8020 (GC) |
| Volatile Organics | 624/8240/8260 (GC/MS), 8015 (GC) |
| Acrolein/Acrylonitrile | 603/8030 (GC) |

## B. SEMIVOLATILE ORGANICS

| Matrix | Holding Time (From Date Sampled) | Container | Preservative | Minimum Sample Size |
|---|---|---|---|---|
| WATER SAMPLES | | | | |
| No Residual Chlorine Present | Samples must be extracted within 7 days and analyzed within 40 days of extraction. | 1 liter glass with Teflon liner | 4°C | 1 liter |
| Residual Chlorine Present | Samples must be extracted within 7 days and analyzed within 40 days of extraction. | 1 liter glass with Teflon liner | Add 3 mL 10% sodium thiosulfate per gallon, 4°C | 1 liter |
| SOIL/SEDIMENTS AND SLUDGES | Samples must be extracted within 14 days and analyzed within 40 days of extraction. | Glass jar with Teflon liner or core tube | 4°C | 50 g |
| CONCENTRATED WASTE SAMPLES | Samples must be extracted within 14 days and analyzed within 40 days of extraction. | Glass jar with Teflon liner or core tube | None | 50 g |

The above information applies to the following parameters and methods:

| Parameter | Method |
|---|---|
| Phenols | 604/8040 (GC) |
| Phthalate Esters | 606/8060 (GC) |
| Organochlorine Pesticides/PCBs | 608/8080 (GC) |
| Polyaromatic Hydrocarbons | 610/8310 (HPLC) |
| Organophosphate Pesticides | 614/8140 (GC) |
| Phenoxy Acid Herbicides | 615/8150 (GC) |
| Semivolatile Organics | 625/8270 (GC/MS) |
| Carbamate & Urea Pesticides | 632 (HPLC) |

## C. OTHER ORGANICS

| Parameter | Method No. | Matrix | Holding Time[a] (from Date Sampled) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Dioxins/Furans | 8280 | Water | 30 days extn.<br>45 days anal.[b] | One liter glass | 4°C | 1000 ml |
| | | Soil/Waste | 30 days extn.<br>45 days anal.[b] | core tube or glass jar | 4°C | 50 g |
| Petroleum Hydrocarbons as Gasoline | TPH-Gasoline Purge & Trap (LUFT manual) | Water | 14 days | 3 40 mL vials with Teflon liners | 4°C, HCl to pH < 2 | 40 mL |
| | | Soil/Waste | 14 days | Core tube or glass jar | 4°C | 50 g |
| Petroleum Hydrocarbons as Gasoline | TPH-Gasoline Extractable (LUFT manual) | Water | 14 days extn.<br>40 days anal. | One liter glass | 4°C, HCl to pH < 2 | 500 mL |
| | | Soil/Waste | 14 days extn.<br>40 days anal. | Core tube or glass jar | 4°C | 50 g |
| Petroleum Hydrocarbons as Diesel | TPH-Diesel Extractable (LUFT manual) | Water | 14 days extn.<br>40 days anal. | One liter glass | 4°C | 500 mL |
| | | Soil/Waste | 14 days extn.<br>40 days anal. | Core tube or glass jar | 4°C | 50 g |
| Petroleum Hydrocarbons (TPH) | TPH-IR (418.1) | Water | 28 days | One liter glass | 4°C, $H_2SO_4$ to pH < 2 | 1000 mL |

(a) extn: extraction anal: analysis
(b) from date of collection

## D. METALS

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled to Analysis) | Container | Preservative(a) | Min. Sample Size |
|---|---|---|---|---|---|---|
| Metals (ICP) | 200.7/6010 | Water | 6 months | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 6 months | core tube/glass jar | 4°C | 10 g |
| Arsenic (GF-AA) | 206.2/7060 | Water | 6 months | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 6 months | core tube/glass jar | 4°C | 10 g |
| Mercury (CV-AA) | 245.1/7470 | Water | 28 days | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 28 days | core tube/glass jar | 4°C | 10 g |
| Selenium (GF-AA) | 270.2/7740 | Water | 6 months | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 6 months | core tube/glass jar | 4°C | 10 g |
| Thallium (GF-AA) | 279.2/7841 | Water | 6 months | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 6 months | core tube/glass jar | 4°C | 10 g |
| Lead (GF-AA) | 239.2/7421 | Water | 6 months | Poly | $HNO_3$ to pH < 2.0 | 100 ml |
| | | Soil/Waste | 6 months | core tube/glass jar | 4°C | 10 g |
| Chromium (III/VI) | 220.7/218.4/ 312B/7196 | Water | 24 hours | Poly | 4°C | 100 ml |
| | | Soil/Waste | 24 hours extn. (b) | core tube/glass jar | 4°C | 10 g |

(a) Listed preservative is for total metals. Dissolved or suspended metals require filtration prior to pH adjustment.
(b) Holding time applies to extract obtained from leached sample.

## E. WET CHEMISTRY

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled to Analysis) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Acidity | 305.1 | Water | 14 days | Poly | 4°C | 50 ml |
| Alkalinity | 310.1 | Water | 14 days | Poly | 4°C | 50 ml |
| Ammonia | 350.1 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 50 ml |
| Biochemical Oxygen Demand | 405.1 | Water | 48 hours | Poly | 4°C | 200 ml |
| Bromide | 300.0 | Water | 28 days | Poly | 4°C | 50 ml |
| Chemical Oxygen Demand | 410.4 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 100 ml |
| Chloride | 300.0 | Water | 28 days | Poly | 4°C | 50 ml |
| Chlorine, residual | 330.1 | Water | ASAP | Poly | 4°C | 100 ml |
| Coliform, Total & Fecal | 909A/ 909C | Water | 6 hours | Sterile poly | 4°C, $Na_2S_2O_3$ | 100 ml |

E. WET CHEMISTRY (Cont.)

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Color | 110.2 | Water | 48 hours | Poly | 4°C | 100 ml |
| Cyanide | 335.1/ 335.2/335.3 | Water | 14 days | Poly | 4°C, NaOH to pH > 12 (a) | 250 ml |
| Fluoride | 340.2 | Water | 28 days | Poly | 4°C | 50 ml |
| Gross Alpha, Beta and Radium | 9310/ 9315 | Water | 6 months | Poly | $HNO_3$ to pH < 2 | 2000 ml |
| Hardness | 200.7/ 314A/314B | Water | 6 months | Poly | $HNO_3$ to pH < 2 | 50 ml |
| Iodide | Dionex | Water | 28 days | Poly | 4°C | 50 ml |
| Nitrate | 353.2/300.0 | Water | 48 hours | Poly | 4°C | 50 ml |
| Nitrite | 354.1 | Water | 48 hours | Poly | 4°C | 50 ml |
| Nitrite plus Nitrate | 353.2 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 50 ml |

E. WET CHEMISTRY (Cont.)

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Odor | 140.1 | Water | ASAP | Glass | 4°C | 1000 mL |
| Oil and Grease | 413.1/ 413.2 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 1000 ml |
| Orthophosphate | 365.3 | Water | 48 hours | Poly | 4°C | 100 ml |
| pH | 150.1 | Water | ASAP | Poly | 4°C | 50 ml |
| Phenolics | 420.1/ 420.2 | Water | 28 days (b) | Glass | 4°C, $H_2SO_4$ to pH < 2 (c) | 100 ml |
| Specific Conductance | 120.1 | Water | 28 days | Poly | 4°C | 50 ml |
| Sulfate | 300.0 | Water | 28 days | Poly | 4°C | 50 ml |
| Sulfide | 376.2 | Water | 7 days | Poly | 4°C, NaOH to pH > 9 $Zn(C_2H_3O_3)2$ | 100 ml |
| Sulfite | 377.1 | Water | ASAP | Poly | 4°C | 100 ml |

E. WET CHEMISTRY (Cont.)

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Surfactants (MBAS) | 425.1 | Water | 48 hours | Poly | 4°C | 100 ml |
| Total Dissolved Solids | 160.1 | Water | 7 days | Poly | 4°C | 100 ml |
| Total Kjeldahl Nitrogen | 351.2 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 100 ml |
| Total Organic Carbon (TOC) | 415.1 | Water | 28 days | Glass | 4°C, $H_2SO_4$ to pH < 2 | 100 ml |
| Total Organic Halogen (TOX) | 9020 | Water | 28 days (d) | Glass | 4°C, $H_2SO_4$ to pH < 2 | 200 ml |
| Total Phosphorus | 365.3 | Water | 28 days | Glass | $H_2SO_4$ to pH < 2 | 100 ml |
| Total Solids | 160.3 | Water | 7 days | Poly | 4°C | 100 ml |
| Total Suspended Solids | 160.2 | Water | 7 days | Poly | 4°C | 100 ml |

E. WET CHEMISTRY (Cont.)

| Parameter | Method No. | Matrix | Holding Time (from Date Sampled) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|---|
| Total Volatile Solids | 160.4 | Water | 7 days | Poly | 4°C | 100 ml |
| Turbidity | 180.1 | Water | 48 hours | Poly | 4°C | 50 ml |

a) Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.

b) The 28 day holding time is specified in Table 1 of Methods for Chemical Analysis of Water and Wastes, issued March 1983. This information supercedes that contained in Method 420.1/420.2 published in 1979.

c) Samples to be analyzed for phenolics should be field-tested for residual chlorine. If residual chlorine is detected, ferrous ammonium sulfate should be added.

d) The 28 day holding time is specified in Table 2-20 of SW-846 3rd edition, Update I, 1987.

## F. CLP HOLDING TIMES

| Parameter | Matrix | Holding Time[a] (from Date Received) | Container | Preservative | Min. Sample Size |
|---|---|---|---|---|---|
| Volatile Organics | Water | 10 days | 3 40 mL vials with Teflon lined caps | 4°C | 40 mL |
| | Soil | 10 days | Glass jar with Teflon liner or core tube | 4°C | 10 g |
| Extractable Organics | Water | 5 days extn.<br>40 days anal. | 1 liter glass with Teflon liner | 4°C | 1000 mL |
| | Soil | 10 days extn.<br>40 days anal. | Glass jar with Teflon liner or core tube | 4°C | 50 g |
| Metals (other than Mercury) | Water | 180 days | P,G [b] | $HNO_3$ to pH < 2 | 100 mL |
| | Soil | 180 days | P,G | 4°C | 10 g |
| Mercury | Water | 26 days | P,G | $HNO_3$ to pH < 2 | 100 mL |
| | Soil | 26 days | P,G | 4°C | 10 g |
| Cyanide | Water | 12 days | P,G | 0.6 g ascorbic acid,[c] NaOH to pH >12, 4°C | 100 mL |
| | Soil | 12 days | P,G | 4°C | 10 g |

(a) Holding times calculated from verified time of sample receipt (VTSR) at laboratory
(b) Polyethylene (P) or glass (G)
(c) Only used in the presence of residual chlorine

## G. TCLP HOLDING TIMES

| Parameter | Matrix | From: Field Collection To: TCLP Extraction | From: TCLP Extraction To: Analysis | Container | Preservative (1) | Min. Sample Size |
|---|---|---|---|---|---|---|
| Volatiles | Waste | 14 | 14 | Glass | 4 degrees C | 4 oz |
| Semivolatiles | Waste | 14 | (2) | Glass | 4 degrees C | 32 oz (3) |
| Mercury | Waste | 28 | 28 | Glass | 4 degrees C | 32 oz (3) |
| Metals (Except Mercury) | Waste | 180 | 180 | Glass | 4 degrees C | 32 oz (3) |

(1) Preservative of incoming sample from field, unless refrigeration results in irreversible physical change to the sample. Refrigeration required for volatiles fraction.

(2) Two-tiered holding time: Must be prepared within 7 days of TCLP Extraction and must be analyzed within 40 days of analytical prep extraction.

(3) Smaller sample size is adequate for solid samples or individual fractions. A combined volume of 32 oz is recommended for semivolatiles and metals. A separate 4 oz container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.

# APPENDIX II

## STANDARD OPERATING PROCEDURES (SOP) ELEMENTS

## ELEMENTS FOR SOP

## LABORATORY, ANALYTICAL METHOD

Title (includes method number)

1. Scope and Application
   - 1.1 Analytes
   - 1.2 Detection limit (instrument and method)
   - 1.3 Applicable matrices
   - 1.4 Dynamic range
   - 1.5 Approximate analytical time (i.e., 5 minutes, 2 days)

2. Method Summary
   - 2.1 Generic description of method and chemistry behind it (i.e., extract with solvent, convert to methyl ester, analyze by electron-capture gas chromatography)

3. Comments
   - 3.1 Interferences
   - 3.2 Helpful hints

4. Safety Issues

5. Sample Collection, Preservation, Containers, and Holding Times

6. Apparatus

7. Reagents and Standards

8. Procedure (detailed step-by-step)
   - 8.1 Sample preparation
   - 8.2 Calibration
   - 8.3 Analysis

## ELEMENTS FOR SOP

## LABORATORY, ANALYTICAL METHOD

(continued)

9. QA/QC Requirements

   9.1 QC samples

   9.2 Acceptance criteria (precision and accuracy, % of multi-component QC analytes which must be within windows)

   9.3 Corrective action required (reference current QC manual)

10. Calculations

11. Reporting

   11.1 Reporting units

   11.2 Reporting limits

   11.3 Significant figures and reporting values below detection limit

   11.4 LIMS data entry

12. References

   12.1 Method source

   12.2 Deviations from source method and rationale

13. Appendices (optional)

   Additional information may be placed in appendices. This may include supporting data (e.g. method validation information), tables, flow charts, etc.

## ELEMENTS FOR SOP

## LABORATORY, STANDARDS AND REAGENTS

Title

1. Reagent/Standard Name

2. Type (reagent, calibration standard, DCS, SCS, stock solution, etc.)

3. Constituents/concentration/solvent

4. Safety Issues

5. Shelf Life

6. Procedure
   - 6.1 Preparation
   - 6.2 Documentation (purchase date, open date, labeling, etc.)
   - 6.3 Verification

7. Responsibilities

8. Appendices (optional) Any additional information.

## ELEMENTS FOR SOP

## LABORATORY, EQUIPMENT OPERATION, CALIBRATION, AND MAINTENANCE

Title

1. Purpose

2. Safety Issues (applicable to the specific equipment)

3. Procedure

   3.1 Initial start-up
   3.2 Calibration and performance documentation
   3.3 Example output
   3.4 Shut-down sequence
   3.5 Maintenance and maintenance records

4. Responsibilities

5. Comments

6. Definitions

7. Appendices (optional) Any additional information.

## ELEMENTS FOR SOP

## LABORATORY, PROCEDURAL

Title

1. Purpose

2. Policies

3. Safety Issues

4. Procedure

5. Responsibilities

6. Comments

7. Definitions

8. Appendices (optional) Any additional information.

# ANALYTICAL SERVICES
# QUALITY ASSURANCE PROJECT PLAN

FOR

## ANALYSIS OF POLYCHLORINATED DIOXINS/FURANS BY LOW RESOLUTION GC/MS

Prepared by:

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Michael J. Miille, Ph.D., General Manager — Date: 8/22/91

Gail C. Celaschi, QA Manager — Date: 8/29/91

Shelly Eyraud, Manager/Low Resolution Dioxin — Date: 8/29/91

# Enseco-California Analytical Laboratory
# Analytical Quality Assurance Project Plan
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## 3.0 PROJECT DESCRIPTION

Enseco is committed to providing quality environmental analytical services to both the public and private sectors. To ensure the production of scientifically sound, legally defensible data of known, documentable and verifiable quality, an extensive Quality Assurance (QA) program has been implemented within Enseco. This program relies on clearly defined objectives, well-documented procedures, a comprehensive audit system, and management support, both Corporate and Regional, for its effectiveness.

This analytical quality assurance plan addresses analyzing project samples for tetra through octa dibenzo-p-dioxins and dibenzofurans. It describes the methods and procedures that are used by Enseco-California Analytical Laboratory (CAL) to ensure that definable quality, precision, accuracy, and completeness objectives are met. Procedures and policies outlined in this document serves to supplement the quality assurance objectives delineated in "Enseco Incorporated-Quality Assurance Program Plan for Environmental Chemical Monitoring", Revision 3.4, April 1991 and is based on the USEPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

## 4.0 RESPONSIBILITIES AND AUTHORITIES

The advanced technologies services group hierarchy is shown in Figure 4-1 and a schematic of the project work flow is shown in Figure 4-2. Responsibilities are outlined as follows:

### DIVISIONAL QUALITY ASSURANCE DEPARTMENTS

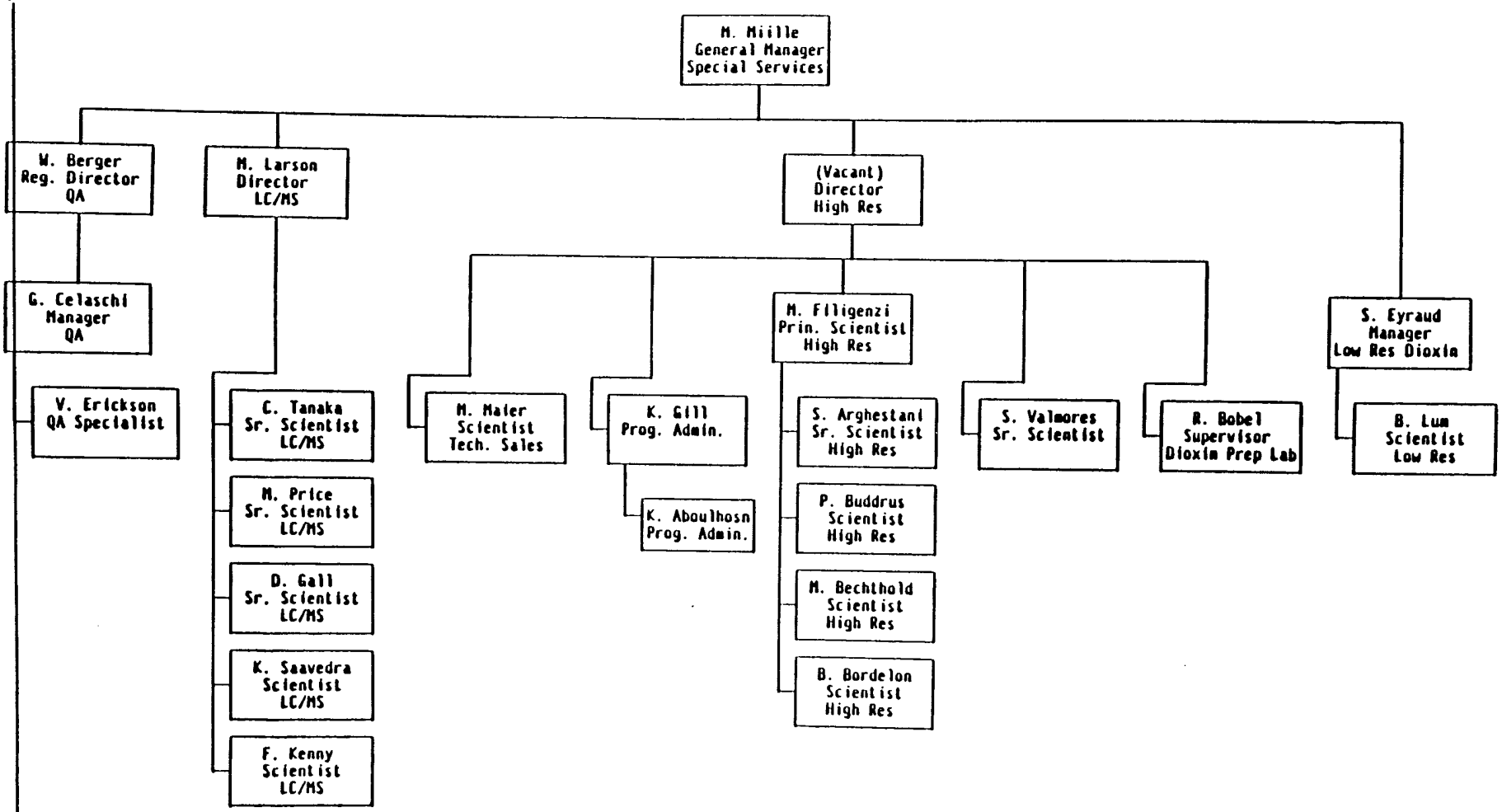
#### Members

Each Regional QA Department is managed by a QA Manager with oversite from a Regional Quality Assurance Director. The QA Manager reports directly to the Regional General Manager and the Regional QA Director. The QA Manager is supported by a QA staff within the laboratory. The QA Manager is the final authority within each laboratory on all issues dealing with data quality. He/she has the authority to require that procedures be amended to discontinued or analyses suspended or repeated. He/she can make recommendations to the Regional General Manager and the Corporate QA Director regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the Division QA Manager comes directly from the Corporate QA Director.

### DIVISIONAL MANAGEMENT

#### Members

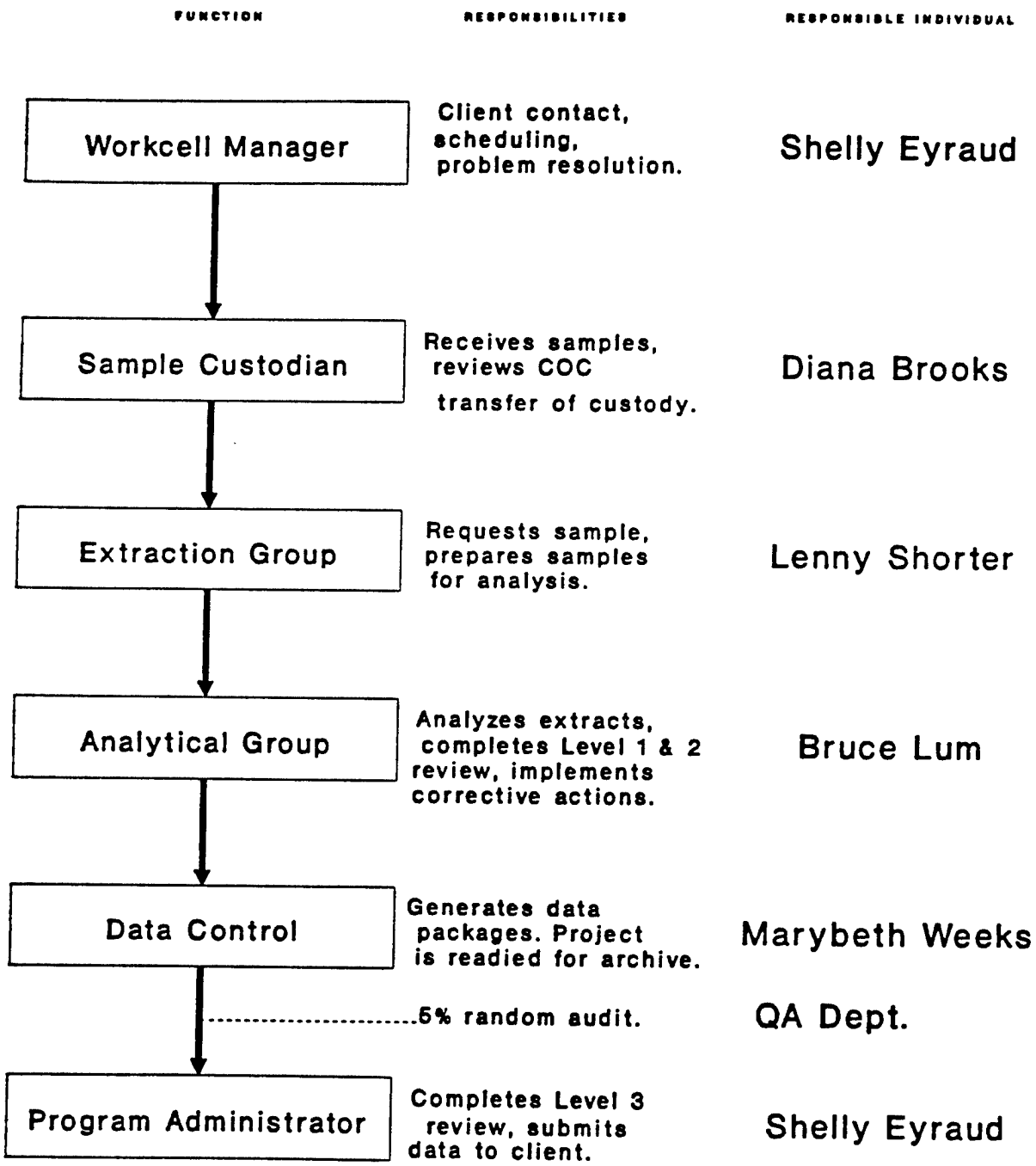
The managers, supervisors, department directors, and program administrators who direct the analytical work at each laboratory are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.




*nseco-California Analytical Laboratory*
West Sacramento, California

# Project Flow
## Figure 4.2
## Dioxin/Furans Workcell





The managers and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgement, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the laboratory management comes directly from the President and the Regional General Manager.

DIVISIONAL PERSONNEL

Members

All laboratory personnel (including chemists, managers, etc.) involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan.

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance or rejection of data that fall outside of established QC guidelines must be approved by laboratory management and the QA department. The authority of the laboratory personnel flows from the Regional General Manager.

# 5.0 QUALITY ASSURANCE OBJECTIVES

## FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

DCS = Duplicate control samples, a pair of standard, control matrix that is spiked with a group of target compounds.

RPD = Relative percent difference.

RSD = Relative standard deviation.

CV = Coefficient of variation.

S = Standard deviation.

X = A measured value.

$\bar{X}$ = Average. Calculated as the sum of all measured values in a population divided by the number of values in the population.

n = Number of measurements or values in a population.

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with control limits established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.

Standard deviation (S) is calculated as follows:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{N} (Xi - \bar{X})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$RSD = 100 (s/\bar{X})$$

or

$$CV = 100 (s/\bar{X})$$

where: RSD = relative standard deviation

CV = coefficient of variation

s = Standard deviation

$\bar{X}$ = mean

In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

$D_1$ = first sample value

$D_2$ = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using DCS, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing DCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} - \frac{X}{T} \times 100$$

where: X = the observed value of measurement

T = "true" value

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

The completeness objective is 100%. Reanalysis will be performed in accordance with the procedures stated in Section 9.0, Analytical Procedures, and in Appendix I in order to meet this completeness goal.

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

Modified Method 8280 (Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, November 1986) will be used for the analysis of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans by low resolution GC/MS. See Appendix I for the list of enhancements to Method 8280 that are part of Enseco-CAL's procedure.

Method specific accuracy and precision objectives are listed in Table 5.1.

TABLE 5.1

ACCURACY AND PRECISION OBJECTIVES

| COMPOUND | SPIKE CONCENTRATION (ng) | % RECOVERY | RPD |
|---|---|---|---|
| 2,3,7,8-TCDF | 10 | 60-140 | 50 |
| 1,2,3,7,8-PeCDF | 10 | 60-140 | 50 |
| 1,2,3,4,7,8-HxCDF | 10 | 60-140 | 50 |
| 1,2,3,4,6,7,8-HpCDF | 10 | 60-140 | 50 |
| OCDF | 50 | 60-140 | 50 |
| 2,3,7,8-TCDD | 10 | 60-140 | 50 |
| 1,2,3,7,8-PeCDD | 10 | 60-140 | 50 |
| 1,2,3,4,7,8-HxCDD | 10 | 60-140 | 50 |
| 1,2,3,4,6,7,8-HpCDD | 10 | 60-140 | 50 |
| OCDD | 50 | 60-140 | 50 |

Units = ng/sample

# 6.0 SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters;
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization;
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed;
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met.

## Sample Containers and Preservatives

Enseco can assist in the sample collection process by providing consultation and assistance to client designing sampling programs. Also, Enseco can make available to the client the Enseco "Sample Safe™", a set of sample containers that are properly cleaned and preserved for use in sample collection.

EPA has established holding time requirements for some analyses. However, holding times for dioxins/furans can vary depending upon the method requested. EPA Method 1613A states that up to one year before extraction of aqueous and solid samples is acceptable due to the stability of dioxins and furans in the environment. This is consistent with the scientific literature which contains several references to the half-life of dioxins in soil - all of which measure the time in years. Samples will be extracted and analyzed according to a client's contract specifications.

On occasion, a sample must be reanalyzed to comply with this QA Program Plan. If this reanalysis is conducted outside of the holding time, the laboratory will be considered to have fulfilled its obligation to meet holding times if the first preparation and/or analysis was initiated within the prescribed holding time.

# 7.0 SAMPLE CUSTODY

A sample is considered in custody if:

- It is in the sampler's or the transferee's actual possession;
- It is in the sampler's or the transferee's view, after being in his/her physical possession;
- It was in the sampler's or the transferee's physical possession and then he/she secured it to prevent tampering; and
- It is placed in a designated secure area.

Upon transfer of custody to Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

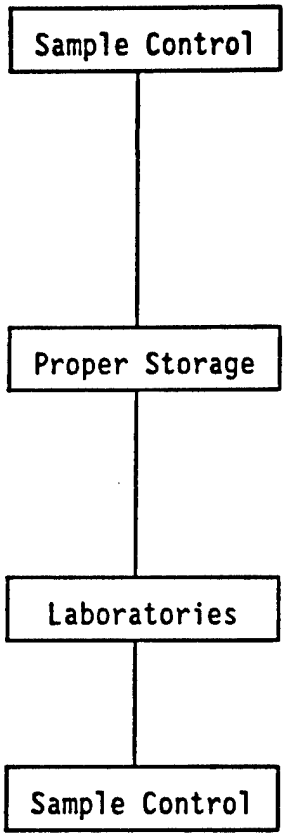
All samples are received by Enseco's Sample Control Group's designated sample custodian and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

In the event that the laboratory sample custodian judges the sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), the Program Administrator (PA) will be advised immediately and the samples will not be analyzed until the PA so authorizes. The PA or designated representative will immediately contact the client. The PA and the client will make a decision as to the fate of the sample(s) in question on a case-by-case basis. The sample(s) will either be processed "as is" with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. Any problem with a sample will be noted on the chain-of-custody form.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2.

FIGURE 7-1

ENSECO SAMPLE PROCESSING FLOW CHART




* Check and document physical condition of sample
* Verify documentation and parameter assignment
* Log into LIMS
* Send acknowledgement letter to client

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

* Document analytical work
* Return unused samples to Sample Control

* Return sample to client or arrange for sample disposal

## CHAIN-OF-CUSTODY RECORD

SAMPLER: (*Signature*)

Date Shipped _____ Carrier _____

Phone _____ Airbill No. _____ Cooler No. _____

**SHIP TO:**

Enseco-Cal Lab
2544 Industrial Blvd.
West Sacramento, CA 95691
(916) 372-1393

ATTENTION: _____

**SEND RESULTS TO:**

Client Name _____

Company _____

Address _____

Phone _____

**PROJECT NAME** _____ **PROJECT NO.** _____ **P.O. NO.** _____

| | | | |
|---|---|---|---|
| Relinquished by: (*Signature*) | Received by: (*Signature*) | Date | Time |
| Relinquished by: (*Signature*) | Received by: (*Signature*) | Date | Time |
| Relinquished by: (*Signature*) | Received at lab by: (*Signature*) | Date | Time |
| Relinquished from lab by: (*Signature*) | Received by: (*Signature*) | Date | Time |

## ANALYSIS REQUEST

| Sample ID Number | Sample Description | Date Time Sampled | Analysis Requested | Sample Condition Upon Receipt |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Special Instructions Comments:

**NOTE: UNUSED PORTIONS OF NON-AQUEOUS SAMPLES WILL BE RETURNED TO CLIENT**

Expected Analytical T.A.T's:

_____ Immediate Attention (200% surcharge) _____ RUSH (50-100% surcharge) _____ Standard

**Cal Lab ID Number:** *(for lab use only)*

## 8.0 ANALYTICAL CALIBRATION PROCEDURES AND FREQUENCY

### Standard/Reagent Preparation

To ensure the highest purity possible, all primary reference standards and standard solutions used by Enseco are obtained from the National Institute of Standards and Technology, the EPA Repository or other reliable commercial sources. Dioxins/furans standards will be purchased from Cambridge Isotope Laboratories, Woburn, Massachusetts or from the US EPA, EMSL-Las Vegas (when available). All standards and standard solutions are logged into a data base that identifies the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

### Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Specific procedures are described as follows:

Tuning

Mass calibration of the mass spectrometer is tuned prior to the analysis of standards or samples each analysis day. The compound FC43 is used to tune the instrument for greater sensitivity in the high mass range to achieve better response in the later eluting compounds.

Window Defining Mix (WDM)

The window defining mix is analyzed to verify that the switching times between the descriptors have been appropriately set and is analyzed for the following conditions:

- Before initial calibration on each instrument and on each gas chromatography column used for analysis.
- Each time a new initial calibration is performed, regardless of reason.
- Each time that adjustments or instrument maintenance activities are performed that may affect retention times.

Initial Calibration

Five calibration solutions (CC1-CC5) containing 10 unlabelled and 6 carbon labeled PCDDs/PCDFs at known concentrations are used to calibrate the instrument before sample analysis can commence. Analytes and concentrations are listed on Table 8.1. The relative ion abundance ratios (using areas to calculate the ratios) must be within the limits outlined in Table 8.2 In addition, all analytes must fall within the retention time windows as determined by the WDM and meet the mass spectrometer sensitivity criteria, i.e., the signal-to-noise ratio must be greater than 2.5 for the unlabelled PCDDs/PCDFs ions and greater than 10 the the internal standard ions. The %RSD of the RRF's for the unlabelled PCDDs/PCDFs, the surrogate, and the internal standards must not exceed 15%. Formulas used to calculate %RSD and RRF are listed in Section 14.0.

Continuing Calibration Standard (Daily Standard)

The daily standard is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance. The RRF of the compounds in the continuing calibration standard must be within 30% of the average RRF determined from the Initial Calibration. If this criteria cannot be met, analysis is suspended, the problem investigated, corrective actions implemented, and a new 5 point calibration is performed, if needed.

Optimum Range

The optimum concentration range of this method is 0.5 - 10 ppb.

TABLE 8.1

Concentration Calibration Solutions*
Used for Initial Calibration

| | Low | Mid-Low | Med. | Mid-high | High |
|---|---|---|---|---|---|
| 2,3,7,8-TCDF | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,7,8-PnCDF | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,6,7,8-HxCDF | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,4,6,7,8-HpCDF | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| OCDF | 0.40 | 0.50 | 2.0 | 4.0 | 5.0 |
| 2,3,7,8-TCDD | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,7,8-PnCDD | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,6,7,8-HxCDD | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| 1,2,3,4,6,7,8-HpCDD | 0.2 | 0.5 | 1.0 | 2.0 | 5.0 |
| OCDD | 0.40 | 0.50 | 2.0 | 4.0 | 5.0 |
| 13C-2,3,7,8-TCDF | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| 13C-2,3,7,8-TCDD | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| 13C-1,2,3,7,8-PnCDD | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 13C-1,2,3,6,7,8-HxCDD | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 13C-1,2,3,4,6,7,8-HpCDD | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 13C-OCDD | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| 37Cl-2,3,7,8-TCDD | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |

* Concentrations are in ng/ul.

TABLE 8.2

Criteria for Isotopic Ratio Measurements
for PCDDs and PCDFs

| Analyte | Selected ions | Relative Intensity |
|---|---|---|
| PCDFs | | |
| Tetra | 304/306 | 0.65-0.89 |
| Penta | 340/342 | 1.31-1.77 |
| Hexa | 374/376 | 1.05-1.41 |
| Hepta | 408/410 | 0.88-1.18 |
| Octa | 442/444 | 0.75-1.01 |
| PCDDs | | |
| Tetra | 320/322 | 0.65-0.89 |
| Penta | 356/358 | 1.31-1.77 |
| Hexa | 390/392 | 1.05-1.41 |
| Hepta | 424/426 | 0.88-1.18 |
| Octa | 458/460 | 0.75-1.01 |
| Internal Standards | | |
| 13C-TCDF | 316/318 | 0.65-0.89 |
| 13C-2,3,7,8-TCDD | 332/334 | 0.65-0.89 |
| 13C-PnCDD | 368/370 | 1.31-1.77 |
| 13C-HxCDD | 402/404 | 1.05-1.41 |
| 13C-HpCDD | 436/438 | 0.88-1.18 |
| 13C-OCDD | 470/472 | 0.75-1.01 |
| Recovery Standard | | |
| 13C-1,2,3,4-TCDD | 332/334 | 0.65-0.89 |

## 9.0 ANALYTICAL PROCEDURES

The methods used are those specified by the US EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including chlorinated dioxins and furans.
- "Test Methods for Evaluating Solid Waste" (SW-846), 2nd Edition (revised), Update I (1984), Update II (1985), 3rd Edition (1986), Update I (1989), Office of Solid Waste and Emergency Response, US EPA.

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. The method selected will detect and quantify 2,3,7,8-tetrachlorinated dibenzo-p-dioxin (2,3,7,8-TCDD), 2,3,7,8-tetrachlorinated dibenzofuran (2,3,7,8-TCDF), and the 2,3,7,8-substituted penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs). Any deviations from published methodology are documented and explained in Appendix I. Samples that initially do not meet quality assurance objectives will be reanalyzed once to verify observed anomalies. Additional reanalysis can be performed at the request of the client for a minimal fee. Specific analytical procedures are as follows:

Internal Standards

To quantitate and measure recovery of the analytes, labeled internal standards will be added to all pre-extraction samples, QC samples, blanks, extraction, and to calibration solutions. Calculated internal standard recoveries must be >=40% or the signal-to-noise ratio must be at least 10:1 before data is considered acceptable. Otherwise, samples will be reextracted and reanalyzed at a smaller sample volume. Associated analytes and internal standards are listed in Appendix I.

Surrogate

The surrogate 37Cl-2,3,7,8-TCDD will be added to all pre-cleanup blanks, samples, QC samples, and to calibration solutions. The calculated % recovery is used to verify the recovery of unlabelled PCDDs/PCDFs and to monitor the efficiency of the cleanup procedures.

Recovery Standard

To measure the % recovery of the labeled internal standards, the recovery standard 13C-1,2,3,4-TCDD will be added to all blank, sample, and quality control sample extracts just prior to GC/MS analysis.

Additional Quality Control

In addition, the following quality control samples will be analyzed:

- Method blanks
- Matrix spike/matrix spike duplicate sample
- Duplicate control sample (DCS)

Definitions, frequency, and corrective action are discussed in Section 11.0, Internal QC Checks and Frequency. Acceptability criteria are listed in Table 5.1, Accuracy and Precision Objectives.

Calculations

A summary of calculations used to determine PCDF/PCDD recoveries and concentrations are listed in Section 14.0, Specific Procedures Used to Assess Data Precision, Accuracy, and Completeness.

Identification

For a gas chromatographic peak to be unambiguously identified as a PCDD/PCDF, it must met the following criteria:

- The absolute retention times must be within method acceptance limits.
- All of the specified ions listed in Appendix I for each PCDD/PCDF must be present in the ion current profile. The ion current response for the two quantitation ions and the M-[COCl]+ ions for the analytes must maximize simultaneously.
- The integrated ion current for each analyte ion listed in Appendix I must be at least 2.5 times background noise with no detector saturation.
- Ion abundance ratios must meet the criteria listed in Table 8.2.

Confirmation

Because specificity for all of the isomers cannot be achieved on a 60 M DB-5 chromatographic column, a second column (SP-2331) will be used to confirm the presence of any 2,3,7,8-substituted PCDDs/PCDFs detected. Samples are first analyzed on a GC/MS fitted with a 60 M DB-5 chromatographic column. If any 2,3,7,8-substituted tetra-, penta-, or hexa- PCDDs/PCDFs are detected, the sample extracts will be reanalyzed using a 60 M SP-2331 chromatographic column. If data resulting from SP-2331 column does not confirm results from the DB-5 column, only data calculated from the SP-2331 column will be reported. All data will be corrected following proper error correction protocol.

## 10.0 DATA REDUCTION, VALIDATION, AND REPORTING

All analytical data generated within Enseco laboratories are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review, as described below (also see Figure 10-1).

### Level 1 Review

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs.

Data will be reduced by an analyst in one of the following ways:

- Manually computing results directly on the data sheet, chromatogram, or on calculation pages attached to the raw data;
- Inputting raw data for computer processing.

If data are manually reduced by an analyst, all steps in the computation will be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and RFs, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than).

Finally, each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample identification is correct;
- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met;
- Congener identification is correct; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, Out-of-Control forms [if required] are complete; holding times are documented, etc.).

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

Level 2 Review

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; Out-of-Control forms [if required] are complete; holding times are documented, etc.).
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Level 3 Review

Before the report is released to the client, the Program Administrator who is responsible for interfacing directly with the client reviews the report to ensure that the date meet the overall objectives of the client, as understood by the Program Administrator. This review is labeled Level 3 review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgement of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. Errors that are found are documented and transmitted to the appropriate supervisors and managers. The cause of the errors is then addressed with additional training or clarification of procedures to that quality data is generated from the lab. The process continues until no errors are found or until the data package has been reviewed in its entirety.

Detection Limits

The detection limit will be calculated for the 10 congeners when no unlabelled PCDFs/PCDDs are detected in the samples. The process is discussed in Appendix I. Calculations are listed in Section 14.0.

Reporting

Results will be reported to the second decimal place in units of ng/l (ppt) for aqueous samples, and ng/g (ppb) for solid samples on a wet weight basis.

An example of a dioxin/furan data validation scheme is shown as Figure 10-1.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

- General Discussion: Description of samples types, tests performed, any problems encountered and general comments are given.
- Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.
- QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

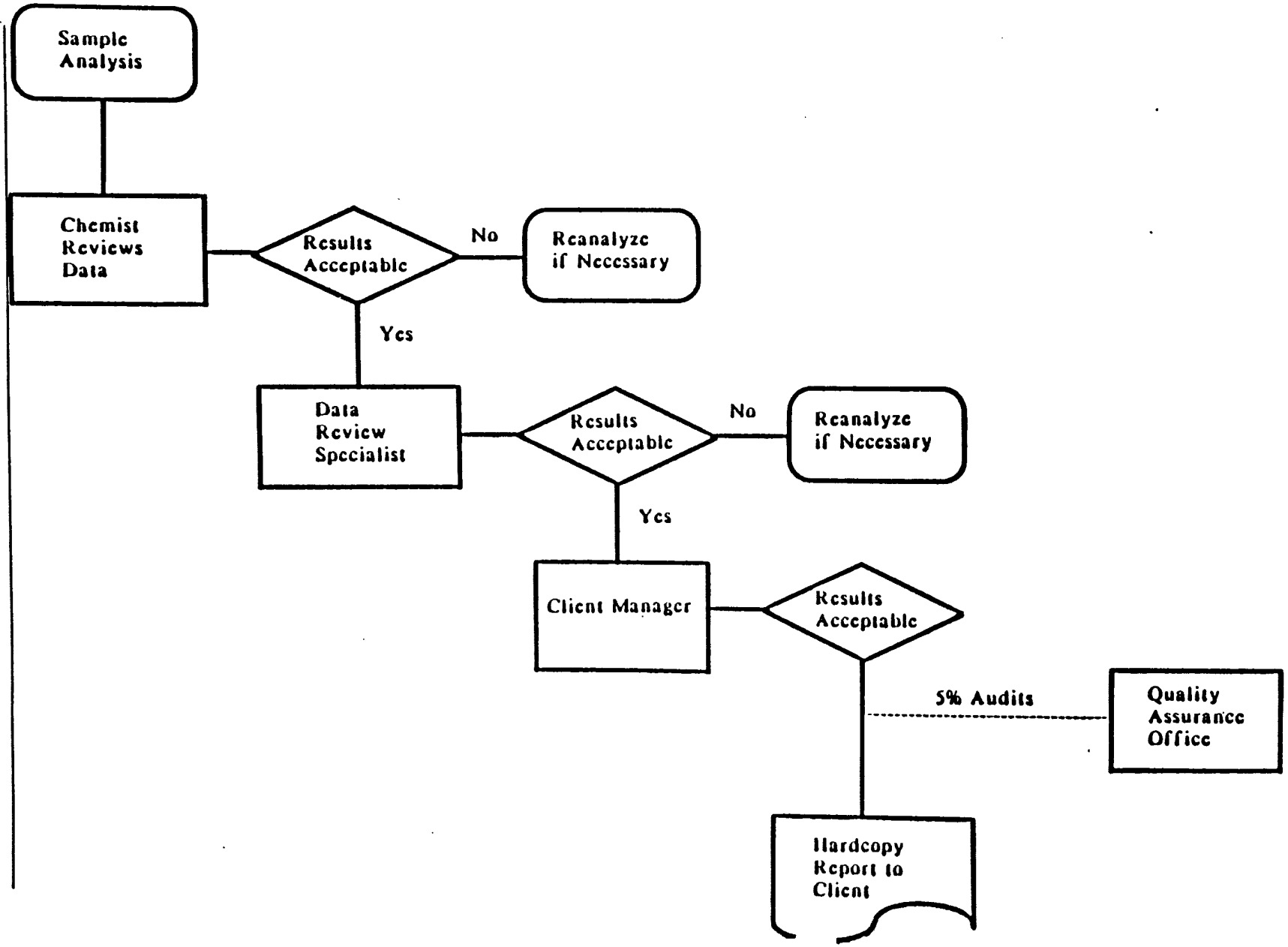
  Results of any matrix spikes, duplicates, matrix spike duplicates or other project-specific QC are also reported.
- Methodology: Reference for analytical methodology used is cited.
- Raw Data: Including calibration data, window defining mix data are included in CLP-type deliverables.

Data Validation

Data validation involves reviewing sample results, quality control, and procedures for compliance with the data quality objectives outlined in this document or with client specified objectives. The scheme used for validation of data is shown in Figure 10.1.

Figure 10-1

# Data Validation Scheme



## 11.0 INTERNAL QC CHECKS

Laboratory Performance QC samples will be added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration so that accuracy and precision can be determined and control charts can be prepared. Measures taken to control analytical data quality include use of specific acceptance criteria for instrument calibration, laboratory control samples, duplicate analyses, blank samples, and spiked samples.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks; and
- The generation of daily calibration data.

Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS consists of a standard, control matrix that is spiked with the internal standards and the surrogate representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference [RPD] between each analyte in the DCS pair) data are compared to control limits that have been established for each of the analytes contained in the DCS. Initially, control limits for analytes spiked into the DCS are taken directly from the CLP program. If CLP limits are not available, Enseco historical data are used to set the control limits. As sufficient laboratory data become available, the control limits are redefined based upon the most recent nine months of DCS data. Control limits for accuracy for each analyte are based on the historical average recovery (mean of the average recoveries of the DCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte are based on the historical RPD and range from zero (no difference between DCS results) to the average RPD plus three standard deviation units.

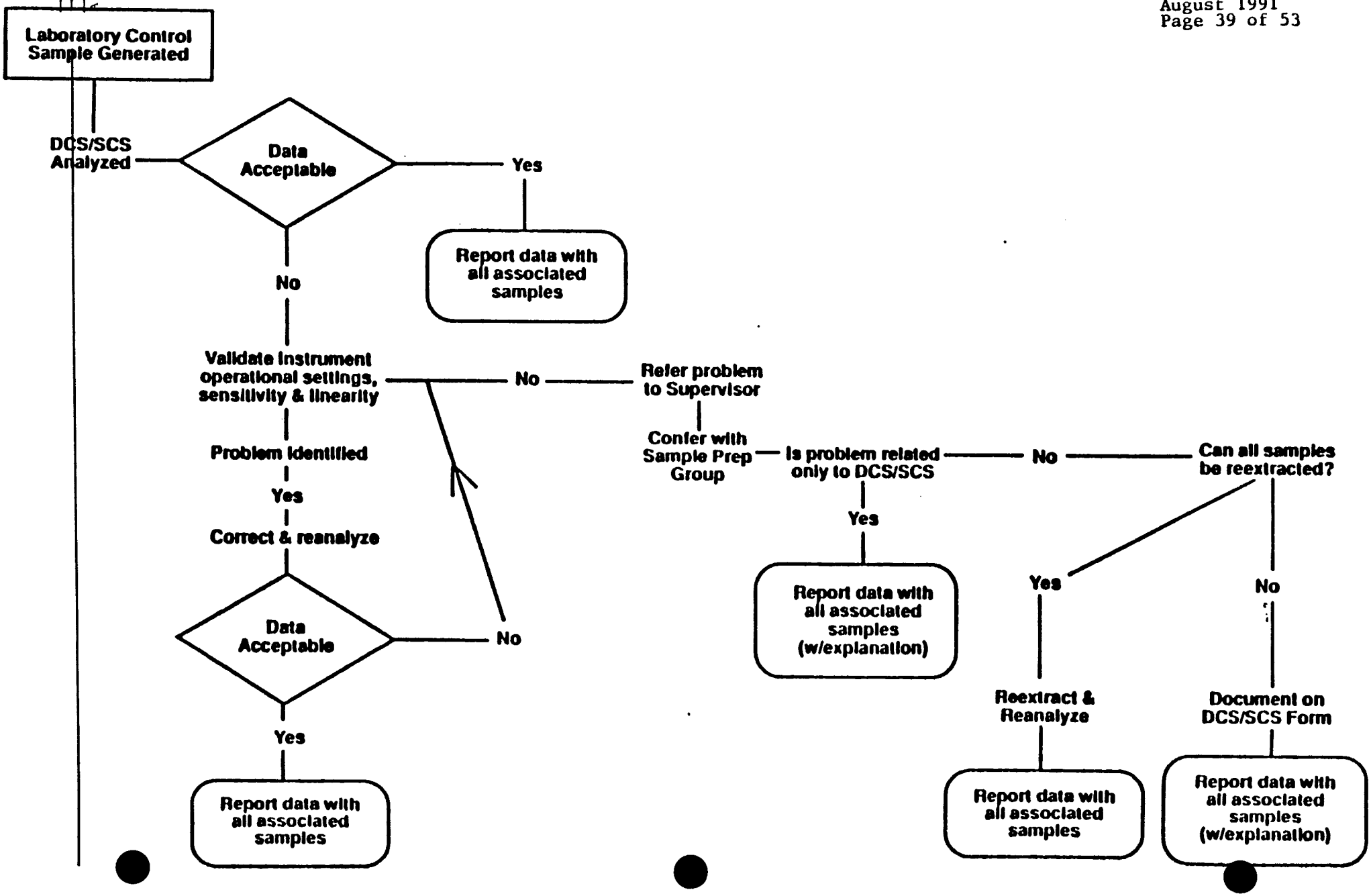
Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. The procedure used to evaluate data from control samples is given in Figure 11-1. The protocols include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

Method Blank

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

# Laboratory Performance QC Control Sample Evaluation





As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

A method blank is prepared and analyzed with every analytical lot or for every 20 samples, whichever is more frequent. Any analyte detected in the blank must be below the reporting limit, otherwise samples are reextracted and reanalyze.

Matrix-Specific QC

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, Enseco encourages its clients to include Matrix-Specific QC that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Spike Duplicate (MSD) is an environmental sample that is divided into two separate aliquots, each of which is spiked with known concentrations of the analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

Surrogate Compounds

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. A labeled surrogate will be added to all extracted, pre-cleanup blanks, samples, QC samples, and to calibration solutions to monitor the efficiency of the cleanup procedure.

Field Blank

Field blanks are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. Solvents such as trichloroethylene are the medium of choice for field blanks when sampling for dioxins and furans; however, if solvents are also parameters of interest, no field blank will be collected. One example of a field blank is an equipment blank. Another type of field blank is a trip blank. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample container, remains unopened in the field, and then is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks. The purpose of the trip blank is to assess the impact of field and shipping condition on the samples.

## 12.0 PERFORMANCE AND SYSTEMS AUDIT

Enseco laboratories participate in a variety of federal and state certification programs, (including the US EPA CLP), that subject each of the laboratories to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the US EPA, US Army, US Navy, and over 4 states. The most current list of Enseco certification is available upon request.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

Ciba-Geigy, Inc. performed both a system and a performance audit for this project. Two 2,3,7,8-TCDD performance evaluation samples were supplied by EPA Region I and submitted to the laboratory for concurrent analysis with samples. Diana Baldi and Frank Saksa of Ciba-Geigy conducted a systems audit while samples were being processed by the laboratory.

A summary of the types and frequency of systems and performance audits is summarized in Table 12.1.

## TABLE 12.1

### SUMMARY OF PERFORMANCE AND SYSTEM AUDITS

| PROGRAM | NAME | TYPE (P OR S) | FREQUENCY |
|---|---|---|---|
| Drinking water | WS | Performance | Semi-Annual |
| Waste water | WP | Performance | Semi-Annual |
| EPA-CLP | QB | Performance | Quarterly* |
| U.S. Navy | PE | Performance | Every 18 mos.* |
| U.S. Army | PE | Performance | Every 18 mos.* |
| Calif. Dept. Food and Agriculture | PE | Performance | Quarterly |
| NPDES | DMR-QA | Performance | Annually |
| EPA-CLP | | System | Annually* |
| Calif. ELAP | | System | Biannually |
| U.S. Navy | | System | Annually |
| Utah | | System | Annually |
| U.S. Army | | System | Every 18 mos.* |
| Divisional QA | | System | Quarterly (approx.) |
| ENSECO Corporate | | System | Annually |
| Ciba-Geigy, Inc. | | System | Random |

Clients may request performance of specific performance and systems audits as a requirement of contract award.

* Contract award required.

## 13.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and schedules.

All aspects of routine and non-routine instrument maintenance are recorded in logbooks, and a log book is dedicated to each instrument.

Balances are calibrated daily or as used with Class S or Class S traceable weights at specific weights of use with the results entered in a logbook kept near the balance.

Ovens and refrigerators are fitted with uniquely marked thermometers and monitored daily. Limits for refrigerators are 2°C to 6°C. If a temperature falls outside these limits, the appropriate laboratory manager is alerted and corrective action is taken. The readings are entered in a logbook kept near the thermometer. Annually the thermometer is calibrated vs. an NIST traceable thermometer.

# 14.0 SPECIFIC PROCEDURES USED TO ASSESS DATA PRECISION

## ACCURACY, AND COMPLETENESS

Calculations for accuracy (% R) and precision (RPD, RSD) were discussed and defined in Section 5.0 Data Quality Objectives. Formulas used to calculate relative response factors (RRF), concentration, and detection limits are as follows:

Definitions

Cn = concentration of native PCDD/PCDF found in the sample

Cis = concentration of internal standards

W = weight (g) of sample extracted

V = volume (mL) of sample extracted

Qis = quantity (ng) of internal standard added to sample before extraction

Qrs = quantity (ng) of recovery standard added to sample extract

Qs = quantity (ng) of surrogate added to sample before extraction

An = integrated ion abundance of the quantitation ion of the isomer of interest (Table 1).

Ais = integrated ion abundance of the quantitation ion of the appropriate internal standard (Table 1 and 2).

RRFn is the response factor of the quantitation ion of the isomer of interest relative to that of the appropriate internal standard.

RRFs is the response factor of the quantitation ion of the surrogate relative to that of the appropriate internal standard.

RRFis is the response factor of the internal standard relative to that of the appropriate recovery standard.

Relative Response Factors

$$RRFn = \frac{An \times Cis}{Ais \times Cn}$$

$$RRFis = \frac{Ais \times Crs}{Ars \times Cis}$$

$$RRFs = \frac{As \times Cis}{Ais \times Cs}$$

Concentration

The concentrations of the various isomers of each congener shall be calculated using the RRF determined for that particular isomer from the initial calibration.

= SOIL =

$$Cn\ (ng/g) = \frac{Qis \times An}{W \times Ais \times RRFn}$$

= WATER =

$$Cn\ (ng/L) = \frac{Qis \times An}{V \times Ais \times RRFn}$$

Detection Limits

For samples in which no unlabelled PCDFs/PCDDs are detected, calculate the detection limit for each of the 10 congeners. The DL is the concentration of a given PCDF/PCDD congener that would produce a signal with peak height of 2.5 times the background signal level, or when no interference is present, the equivalent concentration that the peak would represent if it were a PCDF/PCDD congener. The data must be carefully examined to determine which DL calculation will be used. Follow the rules below.

When only electronic noise is present, or if only one chemical peak is present at either the primary or secondary ion, use the following calculation:

$$DL = \frac{\text{ht of the line (i.e., 2.5 x noise)} \times Qis}{His \times RRFn \times W\ (\text{or } V)}$$

When chemical peaks are present at both the primary and secondary ions that do not meet ratio criteria use formula 2 below:

$$MPC = \frac{Hx \times Qis}{His \times RRFn \times (W \text{ or } V)}$$

## 15.0 CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department. Corrective action documentation is routinely reviewed by the of QA.

## 16.0 QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Regional QA Directors submit extensive monthly reports to the Corporate QA Director and the Regional General Manager. These reports include:

- The results of internal systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the 5% QA data audits conducted.

The Corporate Director of QA submits regularly reports on the status of the QA program to the President and Regional General Manager. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

# APPENDIX I PCDDS/PCDFS

Enseco-Cal Lab performs United States Environmental Protection Agency (US EPA) Method 8280 for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) with some minor modifications to the published method. Since the method was first published in the Federal Register in September 1986, this method has undergone several updates and changes by EPA to keep pace with technology and method improvements during that time period.

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 M DB-5 column. In order to determine the concentrations of the individual 2,3,7,8-substituted isomers, the sample extract shall be reanalyzed on a 60 M SP-2331 GC column. The chromatographic resolution is evaluated using a commercially available column performance mixture containing the TCDD isomers that elute most closely with 2,3,7,8-TCDD.

## GENERAL DIFFERENCES

- Calculation of Detection Limits

  The sensitivity of this method is dependent upon the level of interferences within the sample matrix. All PCDD and PCDF analyses performed for EPA since 1982 has used a technique for calculating the detection limit for each of the chlorination levels and each congener by using the noise level present in the elution window and the height of the chromatographic peak of the internal standard. Both the signal to noise and peak height are determined by the data system of the GC/MS. The result of the calculation is a detection limit that is specific to the homologous series and sample. We are not aware of any laboratories in the dioxin field that use or have used the MDL study referenced in the original 8280 method.

- Internal Standards

Due to the increased availability of the $^{13}C$-labeled PCDD and PCDF internal standards, Enseco-Cal Lab currently uses six instead of only the two internal standards specified and one recommended in the method. This clearly improves the quality of the data.

Internal Standards and Corresponding Analytes

| $^{13}C$-TCDD | $^{13}C$-TCDF | $^{13}C$-PnCDD | $^{13}C$-HxCDD | $^{13}C$-HpCDD | $^{13}C$-OCDD |
|---|---|---|---|---|---|
| TCDD | TCDF | PnCDF | HxCDF | HpCDF | OCDF |
| 37Cl-TCDD | ---- | PnCDD | HxCDD | HpCDD | OCDD |

- Reporting Format

The reporting forms at the end of Method 8280 have been revised a number of times to improve the initial version. Enseco-Cal Lab results, while not reported on the forms in Method 8280, are in a "CLP-like" format.

- Initial Calibration

The initial calibration is performed with single injections of a five point curve.

- Acceptance of Internal Standard Recoveries

Method 8280 specifies internal standard recoveries must be 40% or greater. If recoveries do fall below 40%, then the signal-to-noise ratio is calculated. The recoveries of the internal standards are judged acceptable if the signal-to-noise ratio is greater than 10:1.

- Key Ions Used in the Analysis

Below are the ions used in the determination of PCDDs and PCDFs, they will differ from those listed in Method 8280. The quality of data will not be effected as the ions are consistently used in both the analytical standards and samples.

## Ions Specified for Selected Ion Monitoring for PCDDs and PCDFs

| Analyte | Quantitation ion | Confirmation ion | M-[COCl] + |
|---|---|---|---|
| **PCDFs** | | | |
| Tetra | 306 | 304 | 243 |
| Penta | 340 | 342 | 277 |
| Hexa | 376 | 374 | 311 |
| Hepta | 410 | 408 | 345 |
| Octa | 444 | 442 | 379 |
| **PCDDs** | | | |
| Tetra | 322 | 320 | 259/257 |
| Penta | 358 | 356 | 293 |
| Hexa | 392 | 390 | 327 |
| Hepta | 426 | 424 | 361 |
| Octa | 460 | 458 | 395 |
| **Internal Standards** | | | |
| 13C-TCDF | 318 | 316 | |
| 13C-2378-TCDD | 334 | 332 | |
| 13C-PnCDD | 370 | 368 | |
| 13C-HxCDD | 404 | 402 | |
| 13C-HpCDD | 438 | 436 | |
| 13C-OCDD | 472 | 470 | |

- PCDD/PCDF isomers in the window defining mix for a 60 M DB-5 (or equivalent) column.

| Homologue | First Eluted | Last Eluted |
|---|---|---|
| TCDD | 1368- | 1238- |
| TCDF | 1368- | 1289- |
| PeCDD | 12478 | 12389- |
| PeCDF | 13468- | 12389- |
| HxCDD | 124679- | 123467- |
| HxCDF | 123468- | 123489- |
| HpCDD | 1234679- | 1234678- |
| HPCDF | 1234678- | 1234789- |

SPECIFIC DIFFERENCES

- Analytical

| | | |
|---|---|---|
| Section 4.3.2 - | 8280: | 30 M DB-5 |
| | Cal: | 60 M DB-5 |
| Section 10.3 - | 8280: | Recommended that the GC/MS run be divided into five selected ion monitoring sections. |
| | Cal: | GC/MS run is divided into three selected ion monitoring sections. |
| Section 11.1 - | 8280: | Response factor of the quantitation ion (m/z 334) of the internal standard, $^{13}C$-2,3,7,8-TCDD. |
| | Cal: | Response factor of the quantitation ion of the compound of interest relative to the appropriate $^{13}C_{12}$-labelled internal standard. |

- Sample Preparation

| | | |
|---|---|---|
| Section 9.2.5 - | 8280: | Soil extraction with 20 mL methanol in and 80 mL petroleum ether. |
| | Cal: | 20 mL methanol and 150 mL hexane. Acceptable internal standard recoveries have been demonstrated using hexane as an extraction solvent. |
| Section 9.2.5.1 - | 8280: | Kuderna-Danish concentration. |
| | Cal: | Concentration performed by rotary evaporation. |

Section 9.3 - 9.6 8280: Regents used 20% KOH, 5% NaCl

Cal: Regents used 10N NaOH, DI $H_2O$

The acid/base wash is performed on an optional basis. A silica/alumina column cleanup is the first column cleanup performed and follows (Section 11.3) from SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. The second column is the carbon column cleanup. This cleanup is also from SOW 9/86, Rev. 8/87, Section 11.4.3 - 11.4.5 with omitting 5% benzene as the only modification.

Section 9.7 8280: Gravity column with 4 g of Woelm super 1 neutral alumina.

Cal: Two columns in series. The first consisting of 1 cm $Na_2SO_4$, silica gel, 4 g 44% $H_2SO_4$ (silica gel, 1 g silica gel, 2 g 33% 1M NaOH/silica gel, 1 g silica gel and glasswool. The second consisting of 1 cm $Na_2SO_4$, 6g acid alumina and glasswool. The above column packing materials can be found in Section 11.3, SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. This cleanup is mandatory.