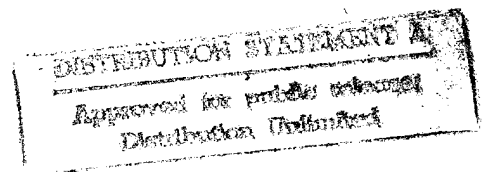
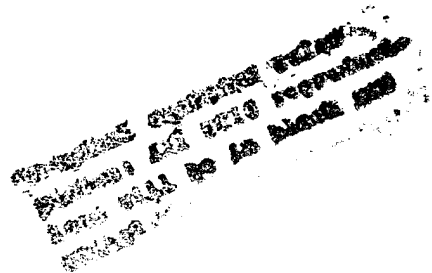


**United States Air Force  
611th Civil Engineer Squadron**

**Elmendorf AFB, Alaska**



**Final  
Addendum to the Work Plan  
Galena Airport, Alaska**

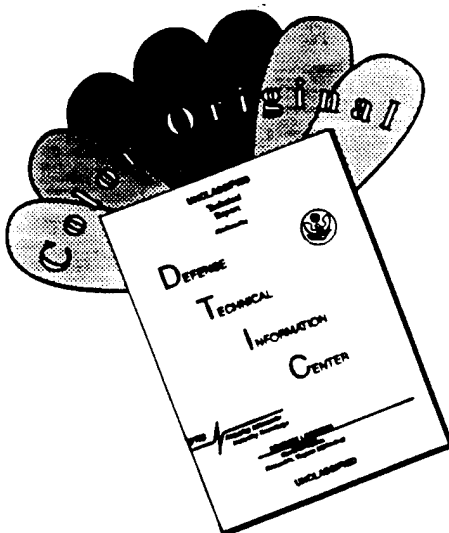


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**United States Air Force  
611th Civil Engineer Squadron**

**Elmendorf AFB, Alaska**

**Final**

**Addendum to the Work Plan  
Galena Airport, Alaska**

**July 1995**

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# WORK PLAN DISCLAIMER

## NOTICE

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**LIST OF ACRONYMS**

BFB	bromofluorbenzene
bgl	below ground level
BTEX	benzene, toluene, ethylbenzene, xylene
CCV	continuing calibration
CTDSA	Control Tower Drum Storage Area
DFTPP	decafluorotriphenylphosphine
DPT	direct-push technology
DRO	diesel range organics
EE/CA	Engineering Evaluation/Cost Analysis
EPA	Environmental Protection Agency
FAA	Federal Aviation Administration
FSP	field sampling plan
GC	gas chromatography
GRO	gasoline range organics
H&SP	health and safety plan
ICP	inductively-coupled plasma spectroscopy
IDW	investigation-derived waste
IRP	Installation Restoration Program
LCS	laboratory control sample
LSE	liquid-solid extraction
MDL	method detection limit
MS	mass spectrometry
MS/MSD	matrix spike/matrix spike duplicate
POL	petroleum, oils, and lubricants
PVC	polyvinyl chloride
QAPP	Quality assurance project plan
RI	Remedial Investigation
RSD	relative standard deviation
SAP	sampling and analysis plan
SVE	soil vapor extraction
TCE	trichloroethene
TPH	total petroleum hydrocarbons
USAF	U.S. Air Force
USTs	underground storage tanks
VOCs	volatile organic compounds
WIMS-ES	Work Information Management System-Environmental Subsystem

## Section 1 INTRODUCTION

This document is the Addendum to the Work Plan for the 1995 work at Galena Airport (shown in Figure 1-1). The *IRP Stage 3 Work Plan, Galena and Campion Air Force Stations, Alaska* (USAF, 1992b) contains information regarding the Installation Restoration Program (IRP), a description of Galena Airport, and the history of past IRP work at the site (USAF, 1992b). This information will not be repeated in this document.

The purpose of this work plan is to describe the work to be performed at Galena Airport during 1995. The proposed work falls into two categories:

1. Completion of investigative work at sites where field screening results indicated that further investigation is warranted to support a baseline risk assessment and to determine whether remedial action or no further action is necessary; and
2. Implementation of removal actions at sites where action has been deemed necessary on the basis of the remedial investigation (RI) results.

In addition, sections of this work plan will serve as the addenda to the sampling and analysis plan (SAP) and the health and safety plan (H&SP), as necessary. Previous addenda to the SAP and the H&SP will be cited for details for all tasks already performed at Galena over the course of the investigation.

### 1.1 Description of Current Study

The current study at Galena Airport involves the continuation of the RI and treatability study started in September 1991. The methodology used to achieve current project objectives is provided in the *IRP Stage 3 Sampling and Analy-*

*sis Plan, Galena and Campion Air Force Stations, Alaska* (USAF, 1992a), the *Addendum to the Sampling and Analysis Plan, Galena and Campion Air Force Stations, Alaska* (USAF, 1993a), or the *Addendum to the Sampling and Analysis Plan, Galena Airport and Kalakaket Creek Radio Relay Station, Alaska* (USAF, 1994), hereafter referred to as the 1992 SAP, the 1993 Addendum to the SAP, and the 1994 Addendum to the SAP, respectively. Except where new, the methodology will not be repeated. Any new techniques that will be implemented as part of the 1995 work are detailed in Section 3 of this document.

#### 1.1.1 Project Objectives

The overall objective of the project is to remediate and/or close sites through investigation. The results of the investigation will be used to support a baseline risk assessment and, if necessary, remedial design and implementation. Figure 1-2 shows the locations of all sites where work is proposed for 1995.

Field investigations will be conducted at the Southeast Runway Fuel Spill site, the Control Tower Drum Storage Area (CTDSA), and the northwest portion of the POL Tank Farm. The objective of the investigation task is to define the extent of contamination quickly and efficiently by conducting soil gas surveys and collecting field samples for rapid turn-around analysis. These efforts will enable the baseline risk assessment and potential removal actions to be conducted as soon as possible. The results of these activities will direct additional sampling, monitor well installation, or remediation activities as appropriate.

Removal actions will be conducted at the POL Tank Farm Area, the Million Gallon Hill Area, and the airport water treatment plant. The objective of the removal actions at these sites is to

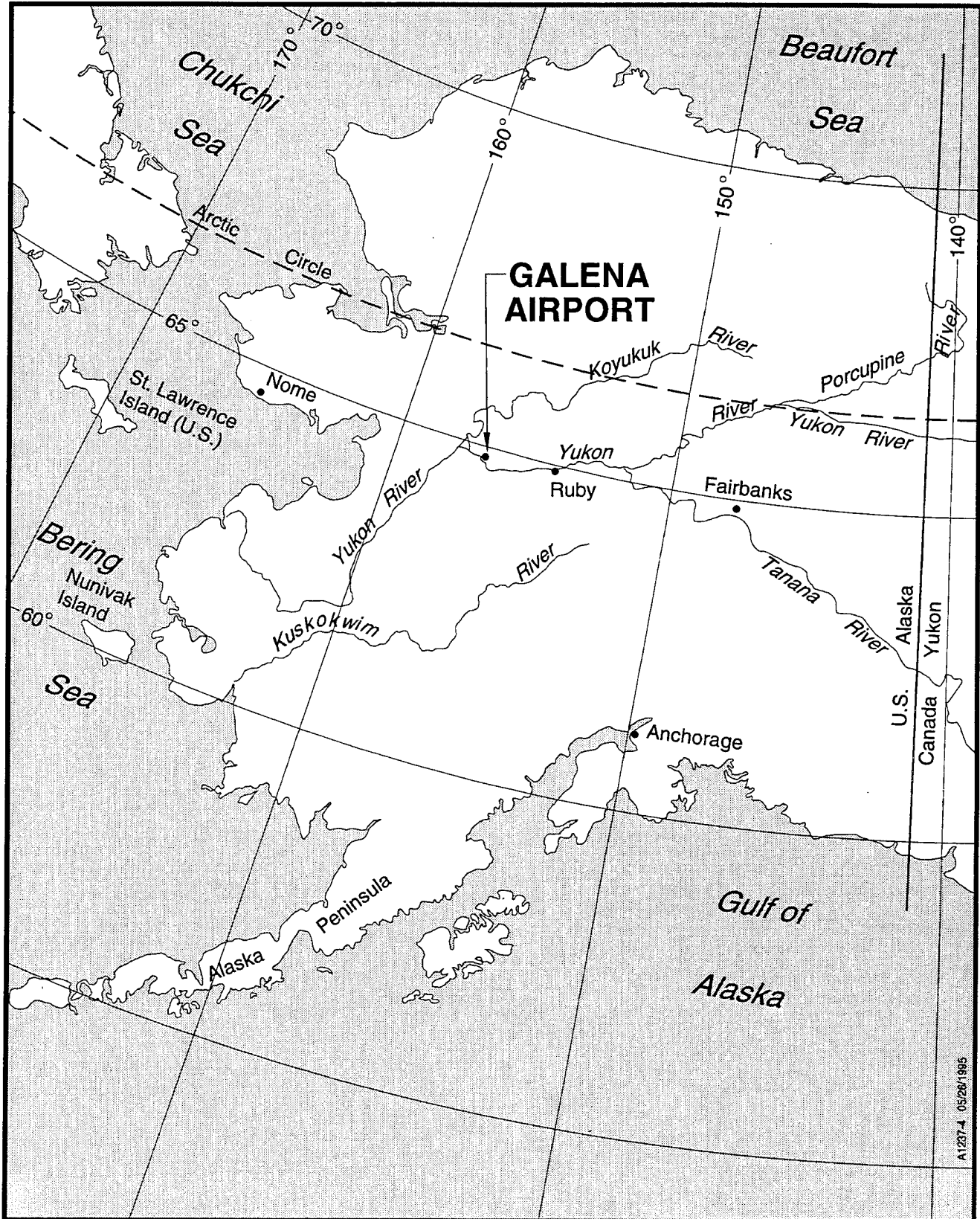
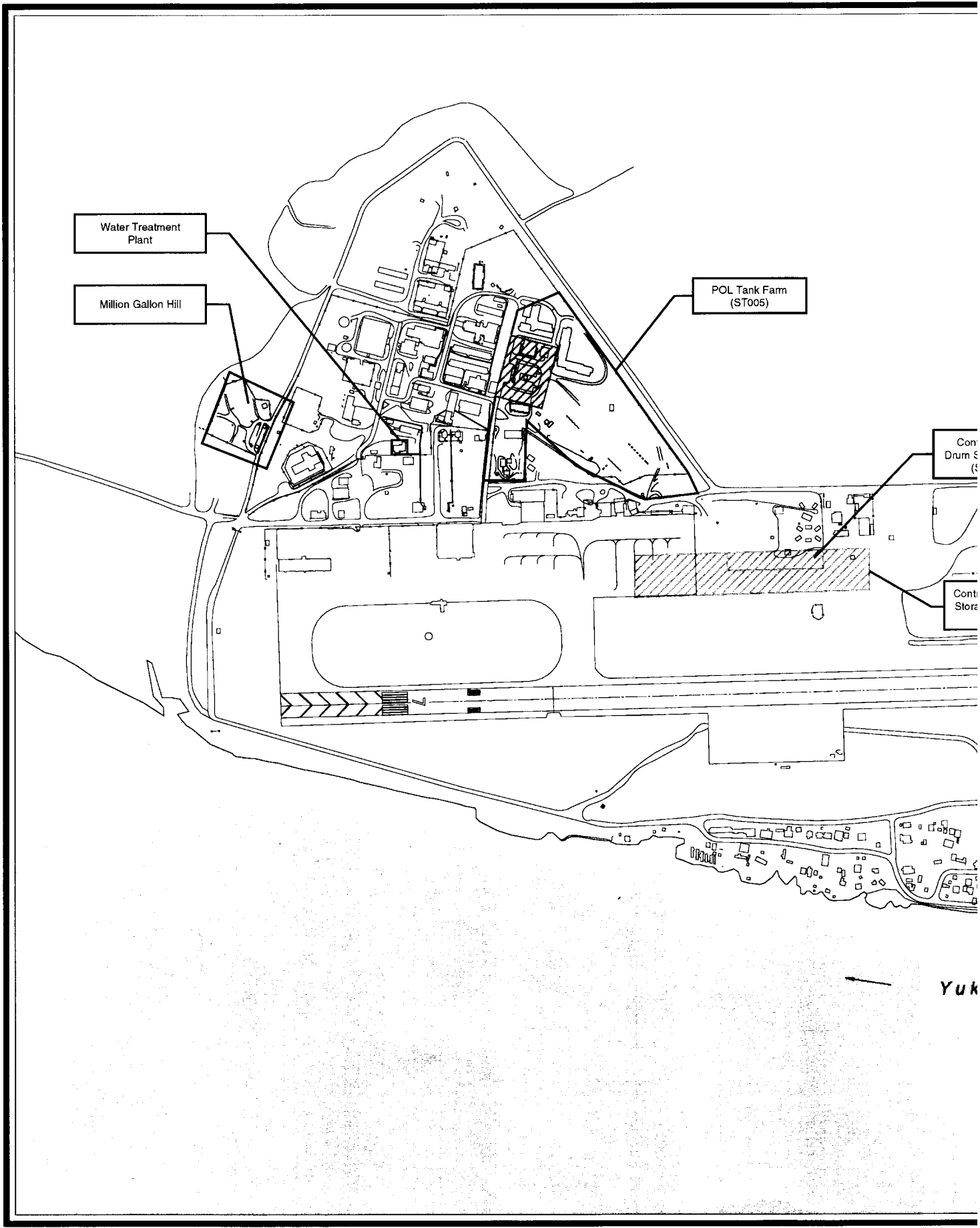
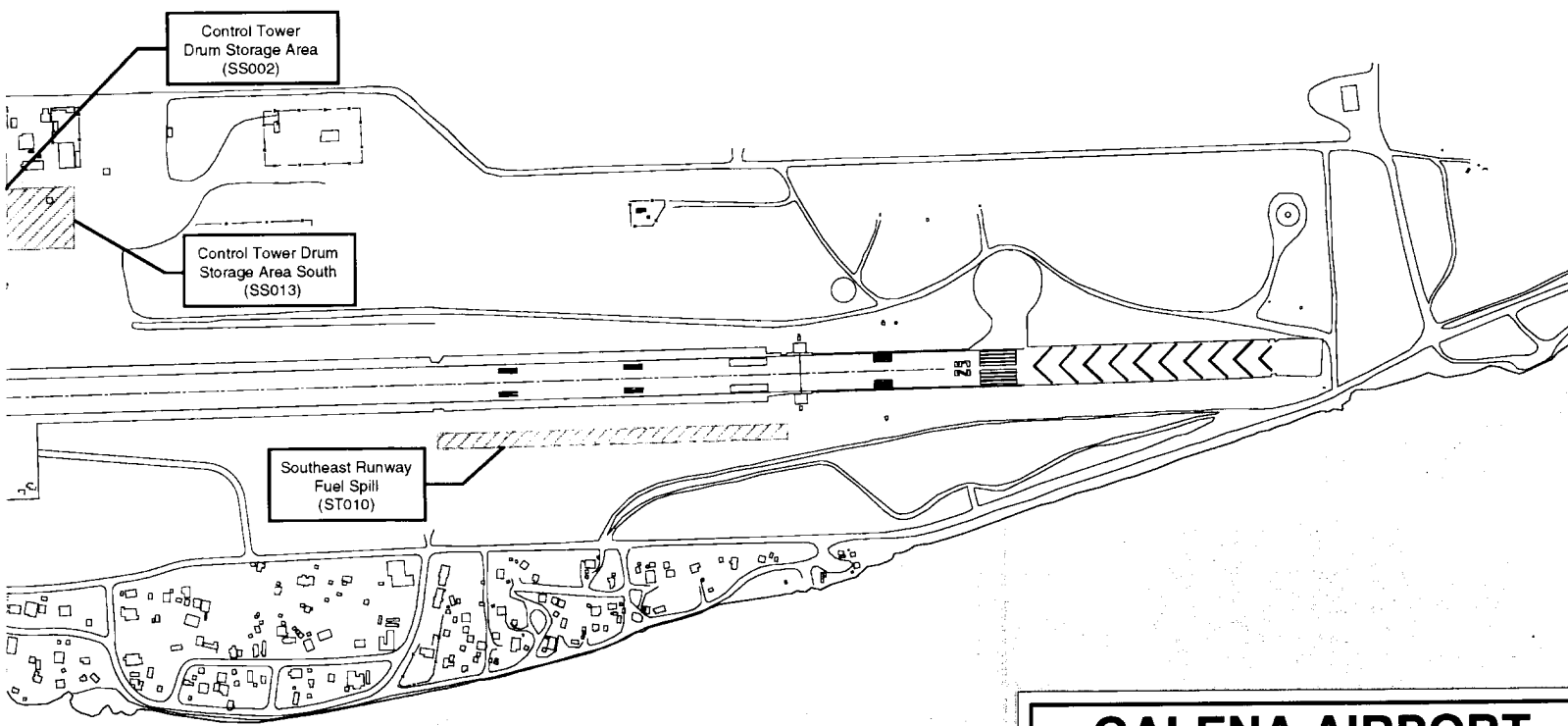
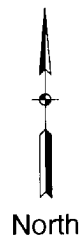



Figure 1-1. Location of Galena Airport, Alaska


Galena Airport





**GALENA AIRPORT  
IRP INVESTIGATION**

 IRP Sites Included in 1995 RI

 Removal Action Sites

0 200 400 600 800 1000 1200  
APPROXIMATE SCALE IN FEET

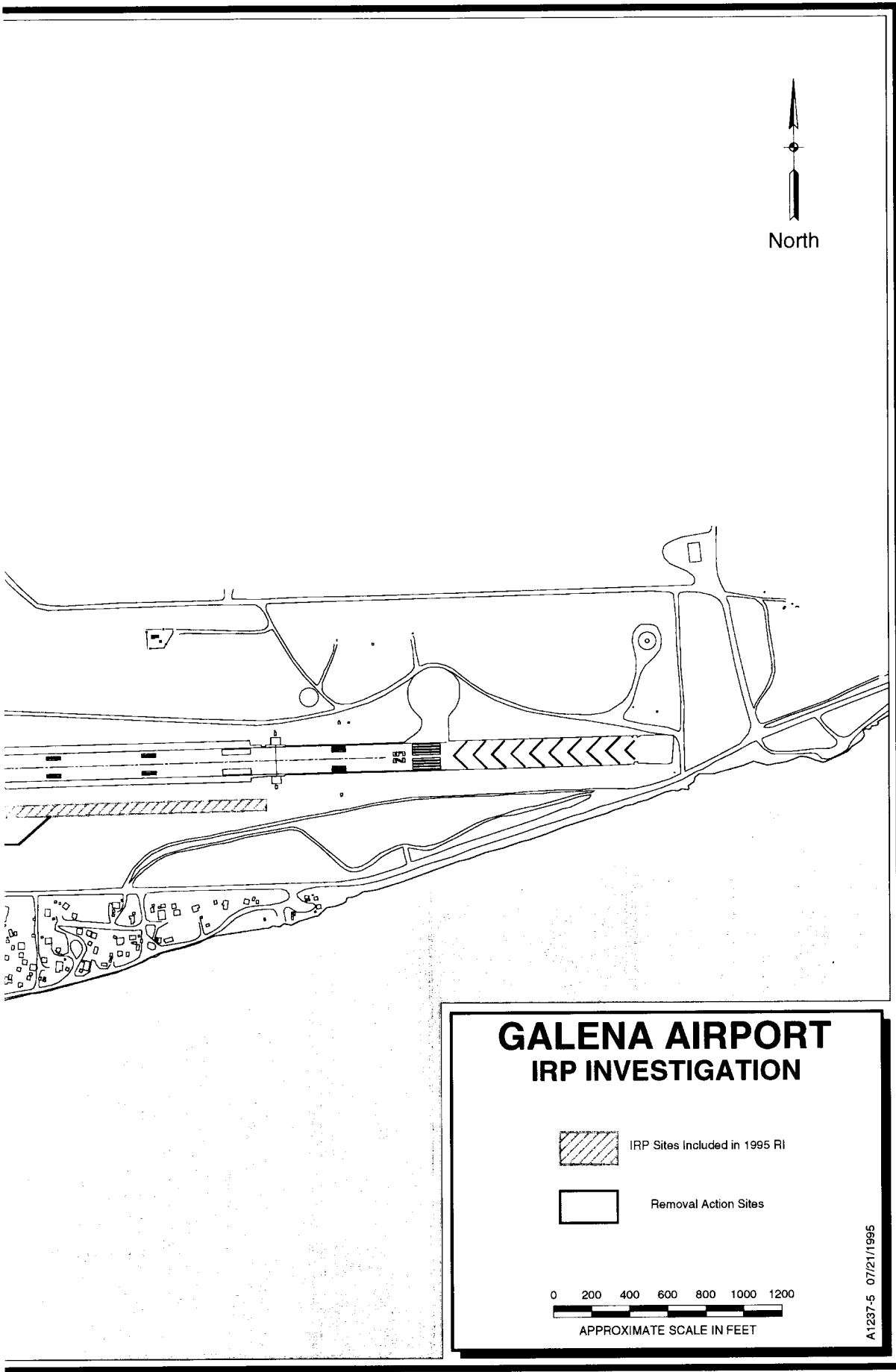


Figure 1-2. Location of RI and Removal Action Sites, Galena Airport, Alaska

prevent the possibility of risk to human health or the environment and/or to remove continuing sources of contamination.

## 1.2 Organization of this Document

Section 1 of this document serves as a brief introduction to the work to be performed at Galena Airport during 1995. For more background information regarding Galena Airport, refer to the *Draft IRP Remedial Investigation Report, Galena Airport and Campion Air Force Station, Alaska* (USAF, 1995). The work plan, which details the work to be performed at Galena Airport, is in-

cluded as Section 2 of this document. Sections 3 and 4, respectively, make up the addenda to the quality assurance project plan (QAPP) and the field sampling plan (FSP); these two sections together are the equivalent of an addendum to the SAP. Section 5 is the addendum to the H&SP, which also refers to the original H&SP. Only information that has not been previously presented in Galena Airport scoping documents will be presented in this document. The references used to prepare this document are given in Section 6.

## Section 2

### WORK PLAN

The purpose of this work plan is to present the work to be done at each of the four sites, plus the water treatment plant, at Galena Airport during 1995.

#### 2.1 Summary of Existing Information

This section briefly summarizes the setting and current information for the locations where work will be performed during 1995. The *Draft RI Report* (USAF, 1995) gives more detail regarding each of these sites. Figure 1-2 shows the locations of these sites at Galena Airport.

##### 2.1.1 Southeast Runway Fuel Spill

The Southeast Runway Fuel Spill (ST010) is the reported location of a fuel-line leak that gathered in the surface water runoff ditch south of the runway and north of the dike. The site was identified through an interview with a local resident (Danny Patrick, personal communication, October 4, 1992), who reported that the release occurred during the winter of 1984. The source of the spill appears to be the petroleum, oil, and lubricant (POL) pipeline that leads from the barge loading area to the POL Tank Farm Area. A soil gas survey and field screening for total petroleum hydrocarbons (TPH) were conducted at the site to determine the lateral extent of the contamination (USAF, 1995). The results indicated that the fuel had infiltrated the soil to some extent, and that further investigation was warranted to determine the potential effect on groundwater. Validated data are needed to support a baseline risk assessment and, if necessary, a removal action.

##### 2.1.2 Control Tower Drum Storage Area

The Control Tower Drum Storage Area (CTDSA—SS002) is an area where drums were formerly stored near the present day control tower. As described in the *Installation Restoration Program, Phase I: Records Search, AAC-Northern Region* (USAF, 1985), the site (Spill/Leak No. 1) is an unpaved area located between the runway and

apron where a large number of drums were stored (stacked horizontally about 3 high and 10 wide). The drums contained unused AVGAS, JP-4, JP-1, diesel fuel, solvents, thinners, cooking oil, and possibly some waste products. Low levels of TPH contamination in the soils were detected at or near the water table during the Stage 1 RI (USAF, 1989). In addition, benzene, toluene, ethylbenzene, and xylenes (BTEX) were detected in subsurface soil and groundwater samples. Traces of trichloroethene (TCE) and lead also were detected in the groundwater samples. Examination of the available aerial photographs indicated that the storage area covered an area extending farther east toward the present-day control tower than the area studied under the Stage 1 RI. Another site, the CTDSA South (SS013), was added to the WIMSES database to address this area. Groundwater sampling and field screening were conducted during 1993 and 1994 to delineate contamination at these sites. The soil gas survey conducted in 1993 showed sporadic, elevated concentrations of volatile organic compounds (VOCs) in the soils. Groundwater sampling conducted in 1994 confirmed the presence of low levels of TCE (approximately 9 µg/L) in one of the monitor wells at the site, but the presence of lead was not confirmed.

The Federal Aviation Administration (FAA) is currently proposing to extend the tarmac in the area of the CTDSA. The proposed paving will decrease risk associated with the site by eliminating infiltration of surface water through potentially contaminated soils and into groundwater, as well as hazards associated with fugitive dust or volatile emissions from surface soils. However, additional surface soil data are needed from unpaved areas to support the baseline risk assessment.

##### 2.1.3 POL Tank Farm

Investigation of the POL Tank Farm (ST005) began in 1986, following identification in

the Phase I Records Search (USAF, 1985). With the exception of a small area in the extreme northwest portion of the site, the POL Tank Farm has been fully characterized with respect to soil and groundwater contamination.

Multiple spills and leaks at the POL Tank Farm have resulted in two main areas of fuel contamination in the soil and groundwater. In the southeastern portion of the tank farm, free product (primarily jet fuel and gasoline) is present on the groundwater. Water table fluctuations, due to seasonal flooding, have produced a "smear zone" of soil contamination. The results of a soil gas survey conducted in the northwestern portion of the POL Tank Farm Area indicate a separate area of fuels contamination. The northern extent of this area of contamination has not been completely defined.

#### **2.1.4 Million Gallon Hill**

Million Gallon Hill is one of the source areas in the West Unit (ST009). Like the POL Tank Farm, Million Gallon Hill has been investigated since its inclusion in the Phase I Records Search (USAF, 1985) and has been characterized with respect to contamination.

Leaks from two large underground storage tanks (USTs) have created an area of free product contamination of the groundwater. Contamination at Million Gallon Hill appears to consist largely of weathered diesel. The results of a soil gas survey conducted over this area showed that the fuels on the groundwater have very low volatility.

#### **2.1.5 Water Treatment Plant**

TCE, suspected to have leaked from the original vehicle maintenance facility (Building 1845), has been detected in several monitor wells upgradient of the airport potable water supply wells. At present, the TCE contamination appears to be confined to the groundwater above the screened interval of the water supply wells, which is approximately 200 ft below ground level (bgl). No TCE has been detected in samples from the airport supply wells to date. Samples from a 200-

ft-deep nonpotable supply well, located downgradient of the TCE plume but upgradient of the potable water supply wells, were found not to contain TCE. Measures need to be taken to ensure that TCE or other contaminants will not affect the drinking water supply in the future.

### **2.2 RI and Removal Action Field Tasks**

This section describes the field activities that will be conducted at Galena Airport during the 1995 field season. These activities include both investigative work and remedial design tasks.

#### **2.2.1 Site Objectives**

The site objectives for the 1995 activities are twofold: 1) to define the extent of contamination quickly and efficiently so that the baseline risk assessment and potential removal actions may be conducted as soon as possible at sites where additional information is required, and 2) to safeguard against the possibility of risk to human health or the environment and/or to remove continuing sources of contamination at sites where the nature and extent of contamination have been determined. To achieve the first objective, soil gas surveys and rapid turnaround analysis results will be used to optimize placement of soil borings, monitoring wells, or remediation wells. To achieve the second objective, information from the RI will be used to conduct removal actions. The main field tasks associated with the removal actions are installation of product recovery, soil vapor extraction (SVE), and bioventing wells, and modification of the existing airport water treatment system. Although installation will take place in 1995, the removal actions may not begin until 1996.

#### **2.2.2 Field Tasks/Sampling Activities**

The objectives given above for the field activities will be applied to two groups of sites. The investigation activities will be conducted at the Southeast Runway Fuel Spill Area, the CTDSA, and the northwest portion of the POL Tank Farm Area. Removal actions will be conducted at the entire POL Tank Farm Area, the

Million Gallon Hill source area, and the airport water treatment plant. The following subsections describe the specific activities to be conducted on a site-by-site basis. These activities are given in Table 2-1. For all sites, the following general activities will take place: location of underground utilities, site clearance, and the surveying of all sampling points. For more information on the sampling and testing procedures, refer to Section 3 of this document, the 1992 SAP (USAF, 1992a), and the 1993 Addendum to the SAP (USAF, 1993a).

### **Southeast Runway Fuel Spill**

The proximity of the Southeast Runway Fuel Spill site to the community of Old Galena makes it a priority site for the 1995. Field activities at this site are designed to define the extent of contamination, especially in groundwater beneath the site, and to support a risk assessment and removal action, if necessary. Work to be conducted at this site consists of the following:

- Conducting a soil gas survey to quickly delineate the soil contamination that has already been confirmed at this site;
- Collecting direct-push technology (DPT) water samples and submitting for rapid turnaround analysis to direct the installation of monitoring wells, if necessary;
- Collecting surface and subsurface soil samples to support the baseline risk assessment; and
- Installing, developing, and sampling up to three flush-mount monitoring wells.

The specific locations for monitoring wells and soil samples will be determined by the results of the soil gas and DPT water sampling at this site. Figure 2-1 shows a conceptual sampling approach for the Southeast Runway Fuel Spill Site. The actual locations and numbers of samples will change on the basis of field information. If it is determined that monitoring wells are necessary, three will be installed so that groundwater flow direction can be determined. Two will be installed

downgradient of groundwater contamination to track movement of the plume. One will be installed within the plume to confirm the nature of the contamination.

The analytical plan for the samples to be collected at the Southeast Runway Fuel Spill site is detailed in Tables 2-2 and 2-3. The numbers of samples given are an estimated maximum; if it is determined that the area of contamination is limited, fewer samples will be required to characterize the site.

### **Control Tower Drum Storage Area**

The presence of surface contamination as a result of drum storage has been investigated over a portion of the CTDSA. The results indicate that "hotspots" of contamination are present over limited areas. However, no laboratory data have been gathered for other portions of the CTDSA. The proposed paving project will eliminate the risk from some of these areas by eliminating fugitive dust and volatile emissions, as well as prohibiting contaminants from leaching to the groundwater. Other areas require additional sampling to support the risk assessment and subsequent site closure or removal actions.

Six surface samples, from the approximate locations shown in Figure 2-2, will be collected at the CTDSA during 1995 and submitted for a full suite of analytes. None of the proposed sample locations are slated to be paved during the extension of the tarmac. Tables 2-2 and 2-3 summarize the analytical plan for CTDSA soil samples.

### **POL Tank Farm**

Both investigation and remedial design tasks will take place at the POL Tank Farm during the 1995 field season. One of the USAF buildings in the northwest portion of the POL area is being proposed as a boarding school dormitory for high school students. The area around this dormitory is the subject of further investigation to better direct the removal action, which is planned for the remainder of the contaminated areas at the site.

**Table 2-1  
 Site Specific Field Activities**

Site Name	History	Proposed Activities
<b>Galena Site Activities</b>		
POL Tank Farm (ST005)	Leaks and spills associated with POL tanks, piping, valves, and drum storage.	<ul style="list-style-type: none"> <li>• Conduct soil gas survey to delineate northwestern plume extent.</li> <li>• Sample soil based on screening results.</li> <li>• Install product recovery and soil vapor extraction wells and vapor monitoring probes.</li> </ul>
Million Gallon Hill	Leaks and spills from USTs Nos. 37 and 38.	<ul style="list-style-type: none"> <li>• Install product recovery and bioventing wells and vapor monitoring probes.</li> </ul>
Southeast Runway Fuel Spill (ST010)	Reported fuel spill gathered in ditch; unknown quantity spilled or recovered.	<ul style="list-style-type: none"> <li>• Conduct soil gas survey and groundwater screening.</li> <li>• Sample soil based on screening results.</li> <li>• Install monitor wells and sample groundwater.</li> </ul>
Control Tower Drum Storage Area (SS002 and SS013)	Spills of various fuels and solvents from drum storage area active from 1940s to 1971.	<ul style="list-style-type: none"> <li>• Collect six surface soil samples from areas where no pavement exists or is proposed.</li> </ul>
Water Treatment Plant	TCE in groundwater above water supply well screens presents potential for exposure.	<ul style="list-style-type: none"> <li>• Conduct triannual sampling of all deep supply wells.</li> <li>• Modify the current water treatment system to safeguard against future potential contamination.</li> </ul>

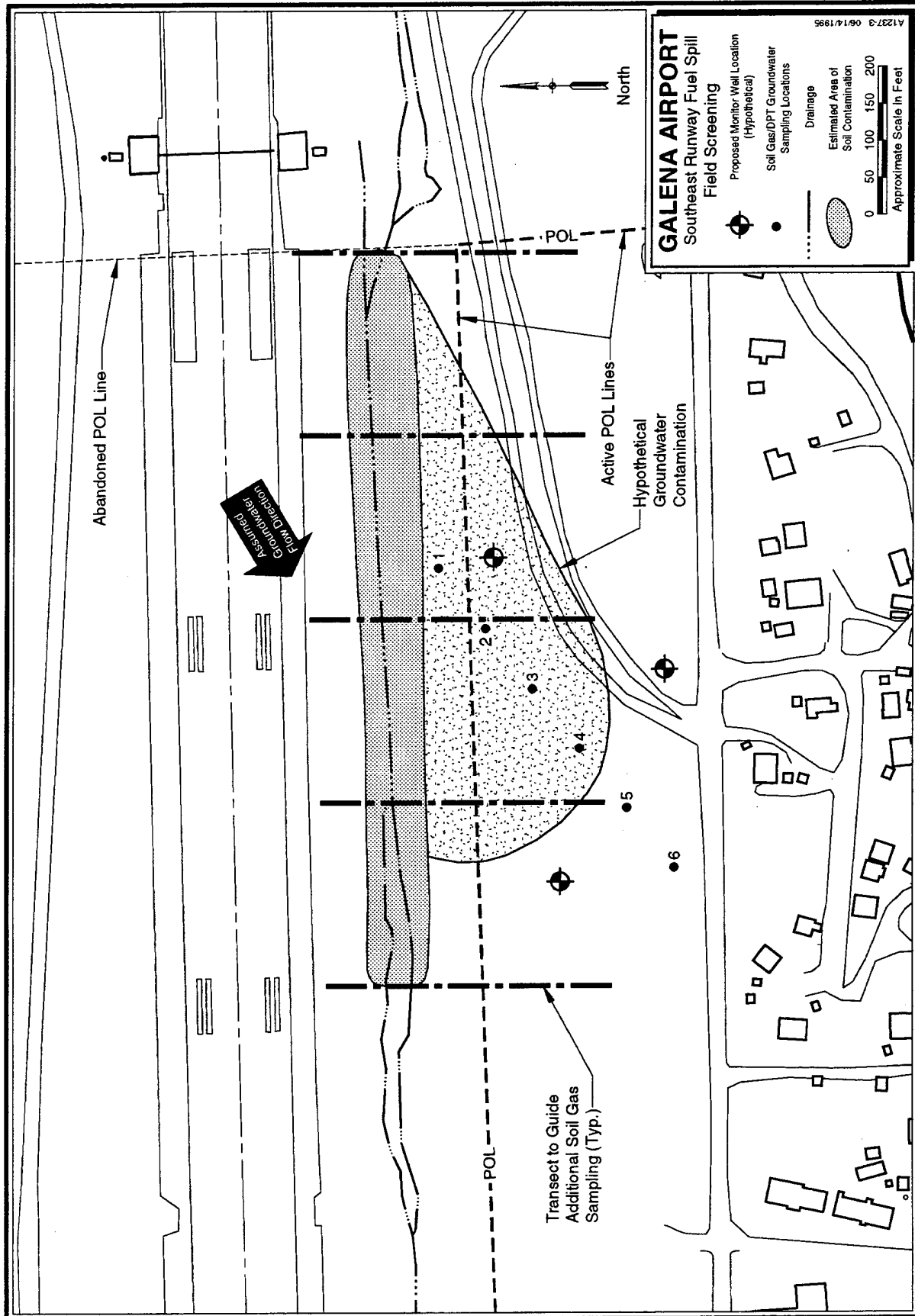


Figure 2-1. Proposed Field Screening Approach for the Southeast Runway Fuel Spill Site (ST010)

**Table 2-2**  
**1995 Sampling Plan for Galena Airport**

Parameter	Method	Number of Field Samples per Area					Total Field Samples	Total QC <sup>a</sup> Samples	Total Samples
		Southeast Runway	Control Tower Drum Storage Area	POL Tank Farm	Airport Water Supply Wells				
<b>SOILS</b>									
Moisture Content	ASTM-D 2216 Mod	10	6	4	--	20	0	20	
Gasoline Range Organics	AK101	10	6	4	--	20	5	25	
Diesel Range Organics	AK102	10	6	4	--	20	5	25	
Volatile Organic Compounds	SW8240	10	6	4	--	20	5	25	
Semivolatile Organic Compounds	SW8270	10	6	--	--	16	4	20	
Pesticides and PCBs <sup>b</sup>	SW8080	--	6	--	--	6	3	9	
ICP <sup>c</sup> Metals	SW6010	--	6	--	--	6	3	9	
Arsenic	SW7060	--	6	--	--	6	3	9	
Lead	SW7421	10	6	--	--	16	4	20	
Selenium	SW740	--	6	--	--	6	3	9	
<b>WATERS</b>									
Gasoline Range Organics	AK101	5	--	--	--	5	6	11	
Diesel Range Organics	AK102	18	--	--	--	18	6	24	
Volatile Organic Compounds	SW8260	5	--	--	--	5	6	11	
Semivolatile Organic Compounds	SW8270	5	--	--	--	5	4	9	
Drinking Water—VOCs <sup>d</sup>	E524	--	--	--	5	5	3	8	
Drinking Water—SVOCs <sup>e</sup>	E525	--	--	--	5	5	1	6	
Lead	SW7421	5	--	--	--	5	4	9	

<sup>a</sup> QC = Quality control. Note: for a detailed breakdown of QC samples, see Table 2-3.

<sup>b</sup> PCBs = Polychlorinated biphenyls.

<sup>c</sup> ICP = Inductively coupled plasma spectroscopy.

<sup>d</sup> VOCs = Volatile organic compounds.

<sup>e</sup> SVOCs = Semivolatile organic compounds.

Table 2-3  
Laboratory and QC Samples for Galena Airport

Parameter	Method	Normal Samples	Field Duplicates	Equipment Blanks	Trip Blanks	Ambient Blanks <sup>b</sup>	MS/MSD <sup>a</sup>
<b>Southeast Runway Soil Samples</b>							
Moisture Content	ASTM D2216M	10	0	0	0	0	0
Gasoline Range Organics	AK101	10	0	1	0	0	2
Diesel Range Organics	AK102	10	0	1	0	0	2
Volatile Organic Compounds	SW5030/SW8240	10	0	1	0	0	2
Semivolatle Organic Compounds	SW3540/SW8270	10	0	1	0	0	2
Lead	SW3050/SW7421	10	0	1	0	0	2
<b>Southeast Runway Groundwater (quick turnaround DPT samples)</b>							
Diesel Range Organics	AK102	15	1	1	0	0	0
<b>Southeast Runway Groundwater (well and DPT samples)</b>							
Gasoline Range Organics	AK101	5	1	1	1	1	2
Diesel Range Organics	AK 102	3	1	1	0	0	2
Volatile Organic Compounds	SW5030/SW8260	5	1	1	1	1	2
Semivolatle Organic Compounds	SW3510/SW8270	5	1	1	0	0	2
Lead	SW3020/SW7421	5	1	1	0	0	2
<b>Control Tower Drum Storage Area Surface Soil Samples</b>							
Moisture Content	ASTM D2216M	6	0	0	0	0	0
Gasoline Range Organics	AK101	6	0	1	0	0	0
Diesel Range Organics	AK102	6	0	1	0	0	0
Volatile Organic Compounds	SW5030/SW8240	6	0	1	0	0	0
Semivolatle Organic Compounds	SW3540/SW8270	6	0	1	0	0	0
Pesticides/PCBs	SW3540/SW8080	6	0	1	0	0	2
ICP Metals	SW3050/SW6010	6	0	1	0	0	2

Table 2-3  
(Continued)

Parameter	Method	Normal Samples	Field Duplicates	Equipment Blanks	Trip Blanks	Ambient Blanks <sup>b</sup>	MS/MSD <sup>a</sup>
Arsenic	SW3050/SW7060	6	0	1	0	0	2
Lead	SW3050/SW7421	6	0	1	0	0	0
Selenium	SW3050/SW7740	6	0	1	0	0	2
<b>POL Tank Farm Soil Samples</b>							
Moisture Content	ASTM D2216M	4	0	0	0	0	0
Gasoline Range Organics	AK101	4	0	1	0	0	0
Diesel Range Organics	AK102	4	0	1	0	0	0
Volatile Organic Compounds	SW5030/SW8240	4	0	1	0	0	0
<b>Airport Water Supply Well Samples</b>							
Drinking Water—VOCs	E524.2	5	1	0	1	1	0
Drinking Water—SVOCs	E525	5	1	0	0	0	0

Note: One MS/MSD pair counts as two analytical samples.

<sup>a</sup> MS/MSD = Matrix spike/matrix spike duplicate.

<sup>b</sup> Ambient blanks will be collected only if an upgradient source of VOCs exists.

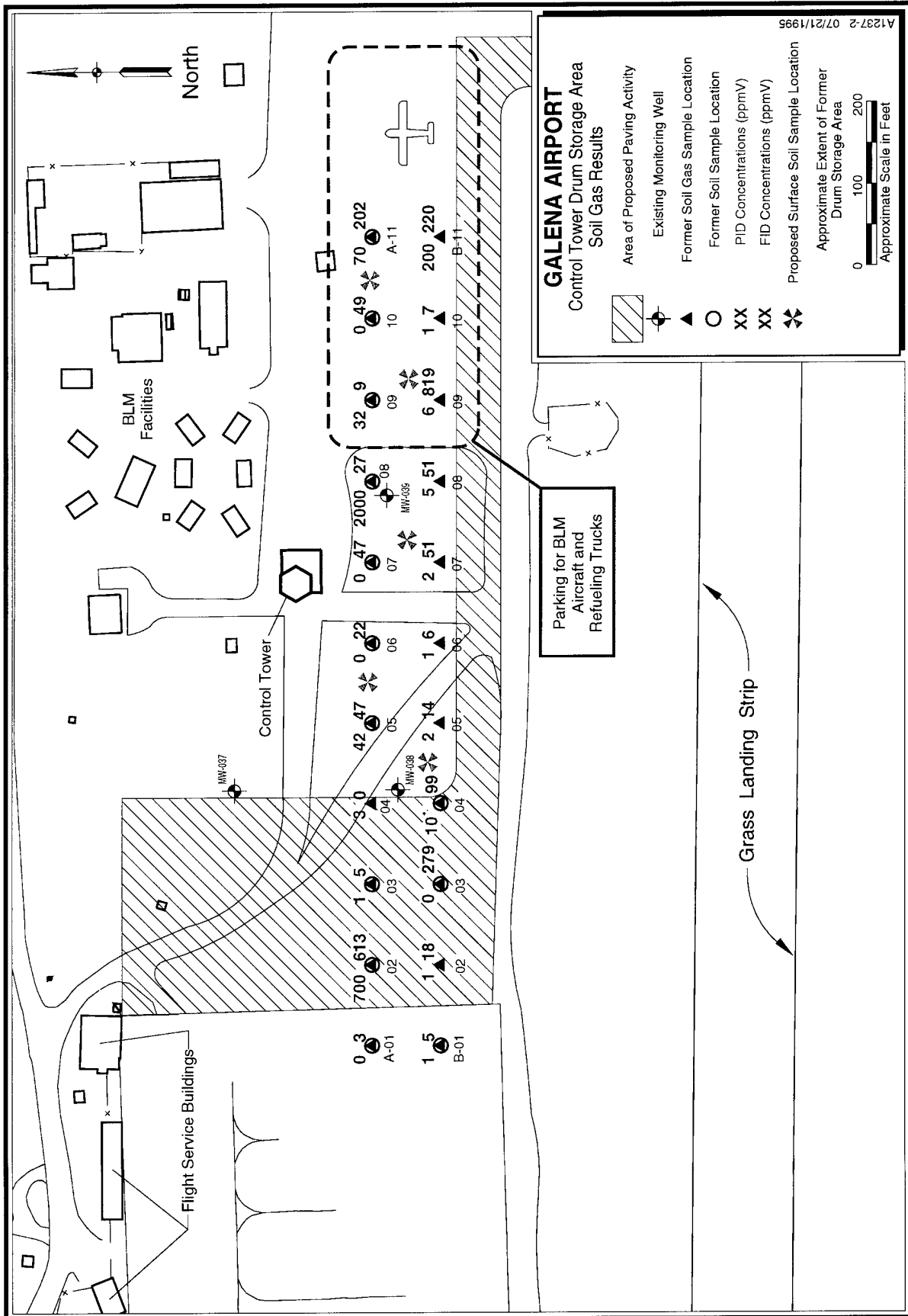


Figure 2-2. Soil Gas Survey Results and Proposed Surface Soil Sample Locations for the Control Tower Drum Storage Area (SS002 & SS013)

The northwest portion of the site will be investigated using soil gas techniques to refine some of the information gathered during a 1993 soil gas survey. Figure 2-3 shows the existing and planned soil gas sample locations; the planned locations are subject to change on the basis of field observations. On the basis of the results of this soil gas survey, a soil boring will be placed with a hollow-stem auger drilling rig. Samples will be collected from four intervals within the boring, from the surface down to groundwater. These samples will be submitted for rapid turnaround analysis of diesel range organics (DRO) and gasoline range organics (GRO) using the State of Alaska Methods AK102 and AK101, respectively (see Tables 2-2 and 2-3). The results of these analyses will be used to decide the optimum location of an SVE well. One SVE well is already planned for near the southwestern corner of the proposed boarding school dormitory.

The southeast portion of the POL Tank Farm has been thoroughly characterized with respect to soil and groundwater contamination. Therefore, no further investigation is necessary prior to the installation of SVE and product recovery wells.

The design for the removal action at the POL Tank Farm consists of:

- A network of 9 product recovery wells (including 2 existing wells) in the southeast portion of the site, centered near the valve pit just north of the flight services access road.
- A network of 7 SVE wells, 5 in the southeast portion of the POL and 1 to 2 in the northwest portion of the POL, near the proposed high school dormitory.
- A network of 42 vapor probes (21 pairs) and thermocouples installed around the 7 SVE well locations. Typically, 3 sets of vapor probes will be associated with each of the shallow and deep screened intervals in each SVE well location.

The layout of the remediation system at the POL Tank Farm is shown in Figure 2-4. Well construction specifications are shown in Table 2-4. Product recovery wells will be installed in areas where free product has been measured in monitoring wells. SVE wells will be installed to remove residual hydrocarbons from areas of vadose zone contamination. Soil vapor probes will be installed around each SVE well to monitor system performance during operations. Final design specifications for the remedial system layout will be provided as full-size drawings before field work begins on 18 July 1995. The final design drawings should be used as the primary guide to all field construction activities rather than the information shown here.

#### **Million Gallon Hill**

The design for the removal action at the Million Gallon Hill area consists of:

- A network of 10 product recovery wells (including 3 existing wells) covering the area south and east of the USTs where free product is present on top of the groundwater.
- A network of 7 bioventing wells (including 3 existing wells), 5 of which will also be used as product recovery wells. Each bioventing well location will have two separate 4-in. wells screened in the upper and lower portions of the vadose zone contamination.
- A network of 42 vapor probes (21 pairs) and thermocouples installed around the 7 bioventing well locations. Typically, 3 sets of vapor probes will be associated with each of the shallow and deep screened intervals in each bioventing well location.

Well construction specifications are shown in Table 2-5, and the layout of the planned remediation system is shown in Figure 2-5. Product recovery wells will be installed in areas where free product has been detected in the subsurface.

Bioventing wells will be installed rather than SVE wells because of the lower volatility of the hydrocarbon contamination. Soil vapor probes will be installed in conjunction with the bioventing wells to monitor system performance during operations. Final design specifications for the remedial system layout will be provided as full-size drawings before field work begins on 18 July 1995. The final design drawings should be used as the primary guide to all field construction activities rather than the information shown here.

### **Water Treatment Plant**

An addition to the water treatment plant is being designed and will be constructed during the fall of 1995. This additional treatment step will be designed to remove potential contaminants from the airport drinking water supply, should they ever reach the screened interval of the supply wells. The design of the water treatment plant has not been finalized, but will be detailed under separate cover in the *Design Analysis Report* (USAF, in press).

An engineering evaluation/cost analysis (EE/CA) is being developed to determine the most effective interim removal technology to address the threat of groundwater contamination that may affect the public water supply. In general, the EE/CA is a streamlined study that evaluates several removal action alternatives on the basis of effectiveness, implementability, and cost. This evaluation studies each alternative's effectiveness in terms of its protectiveness (of human health and the environment), its compliance with applicable regulations, its long- and short-term effectiveness in removing and containing contaminants, and its ability to reduce the toxicity, mobility, or volume of contamination. The implementability criterion is evaluated in light of the availability of services

and materials, technical and administrative feasibility, and the acceptance by the public and regulatory communities. The cost criterion is evaluated in terms of both the capital cost and the costs to operate and maintain any remedial systems, if applicable.

Triannual monitoring of the airport water supply wells, including one upgradient nonpotable supply well, will be implemented in conjunction with the treatment system. The first round of sampling will be conducted during the 1995 field season. Table 2-3 details the analytical plan. Section 4 of this document serves as an addendum to the QAPP to describe the analytical methods that will be used for this monitoring task.

## **2.3 Reporting Requirements**

This section describes the supplemental reporting requirements specified in the modification to the Statement of Work (Appendix A), and not contained in the 1992 work plan (USAF, 1992b).

### **2.3.1 Daily Field Activities Report**

Each day field activities are conducted, a daily field activities report will be completed by the field manager. An example of the daily field activities report that will be used for this effort is shown in Figure 2-6.

### **2.3.2 Technical Report**

The results of the additional field activities will be summarized and integrated into the final remedial investigation report as inserts or replacement pages to the current draft.

## **2.4 Project Schedule**

The schedule for the Galena Airport field work planned for 1995 is shown in Figure 2-7.



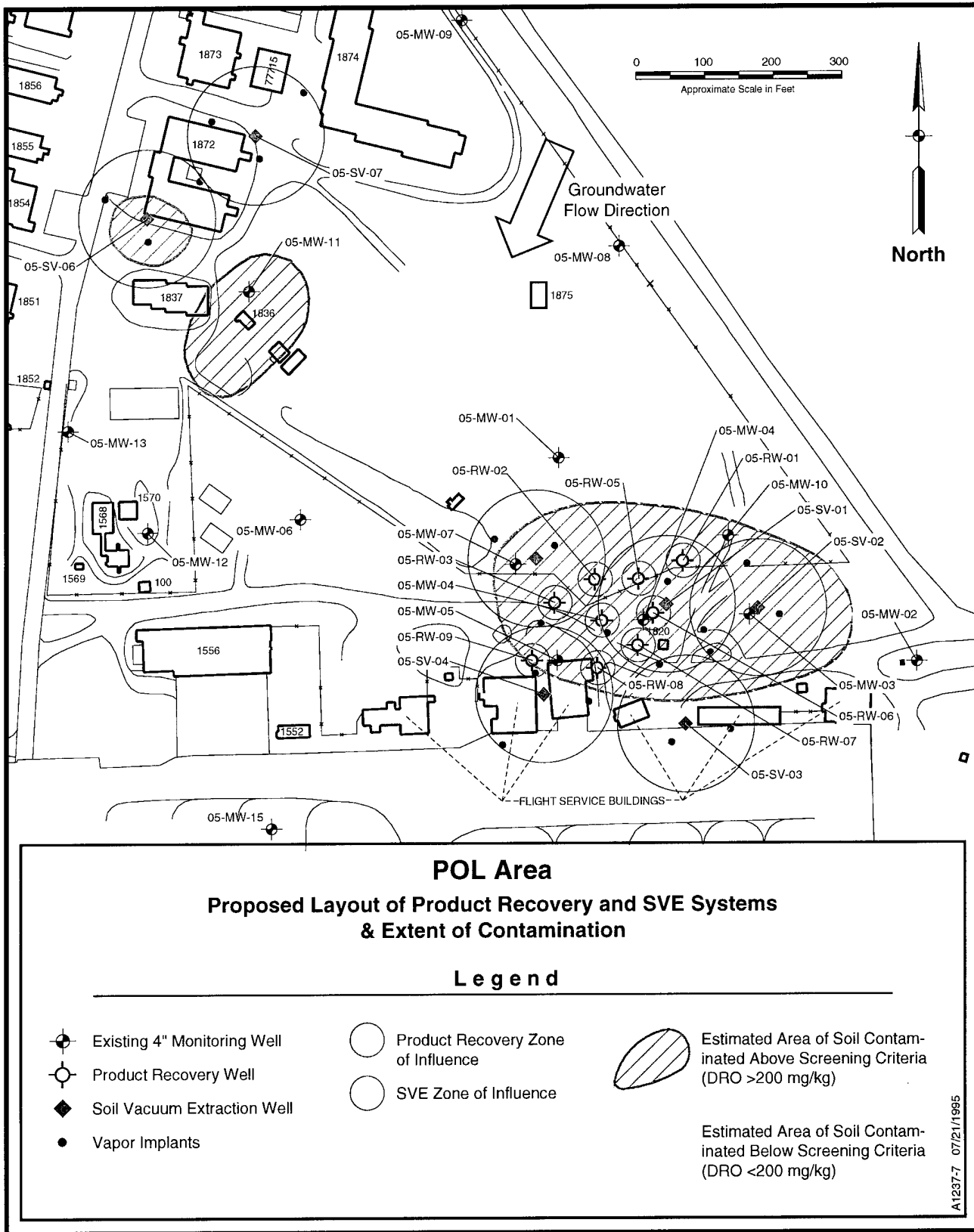


Figure 2-4. Preliminary Layout of Product Recovery and SVE Systems at the POL Tank Farm

Table 2-4  
Remediation Well Specifications—POL Tank Farm

Well ID	Surface Elev. (ft MSL)	Total Depth Elev. (ft MSL)	Screen Interval		Riser Interval		Bentonite Seal Interval		Implant Depths (ft)	No. Implants	Use
			(ft)	(ft)	(ft)	(ft)	(ft)	(ft)			
05-RW-03	145	115.5	17	27	+2	17	13	15	-	0	Product recovery only
05-RW-04	145	115.5	17	27	+2	17	13	15	-	0	Product recovery only
05-RW-05	150	115.5	22	32	+2	22	18	20	-	0	Product recovery only
05-RW-06	146	115.5	18	28	+2	18	14	16	-	0	Product recovery only
05-RW-07	145	115.5	17	27	+2	17	13	15	-	0	Product recovery only
05-RW-08	145	115.5	17	27	+2	17	13	15	-	0	Product recovery only
05-RW-09	145	115.5	17	27	+2	17	13	15	-	0	Product recovery only
05-SV-01	-	-	-	-	-	-	-	-	15, 25	3	SVE only - existing well
05-SV-02	-	-	-	-	-	-	-	-	15, 25	3	SVE only - existing well
05-SV-03	146	115.5	18	28	+2	18	14	16	20.5, 25.5	3	SVE only
05-SV-04	145	115.5	17	27	+2	17	13	15	19.5, 24.5	3	SVE only
05-SV-05	150	115.5	22	32	+2	22	18	20	24.5, 29.5	3	SVE only
05-SV-06	150	115.5	22	32	+2	22	18	20	24.5, 29.5	3	SVE only
05-SV-07	150	115.5	22	32	+2	22	18	20	24.5, 29.5	3	SVE only

**Table 2-5  
Remediation Well Specifications—Million Gallon Hill**

Well ID	Surface Elev. (ft MSL)	Total Depth Elev. (ft MSL)	Screen Interval		Riser Interval		Bentonite Seal Interval		Implant Depths (ft)	No. Implants	Use
			(ft)	(ft)	(ft)	(ft)	(ft)	(ft)			
09-MW-10A	166	133.5	20	30	+2	20	16	18	25, 38	3	Bioventing only, lower screen exists
09-MW-11A	163	130.5	20	30	+2	20	16	18	25, 37	3	Bioventing only, lower screen exists
09-RW-01	155	114.5	28	38	+2	28	24	26	--		Product recovery only
09-RW-02A	166	130.5	18	33	+2	18	14	16	25.5	3	Bioventing only
09-RW-02B	166	114.5	34	49	+2	34	30	32	41.5	3	Product recovery and Bioventing
09-RW-03	171	114.5	44	54	+2	44	40	42	-	0	Product recovery only
09-RW-04	148	114.5	21	31	+2	21	17	19	-	0	Product recovery only
09-RW-05	141	114.5	14	24	+2	14	10	12	-	0	Product recovery only
09-RW-06A	175	130.5	27	42	+2	27	23	25	34.5, 50.5	3	Bioventing only
09-RW-06B	175	114.5	43	58	+2	43	39	41	50.5	3	Product recovery and Bioventing
09-RW-07	150	114.5	18	33	+2	18	14	16	21.75, 29.3	3	Product recovery and Bioventing
09-RW-08	144	114.5	12	27	+2	12	8	10	15.75, 23.3	3	Product recovery and Bioventing

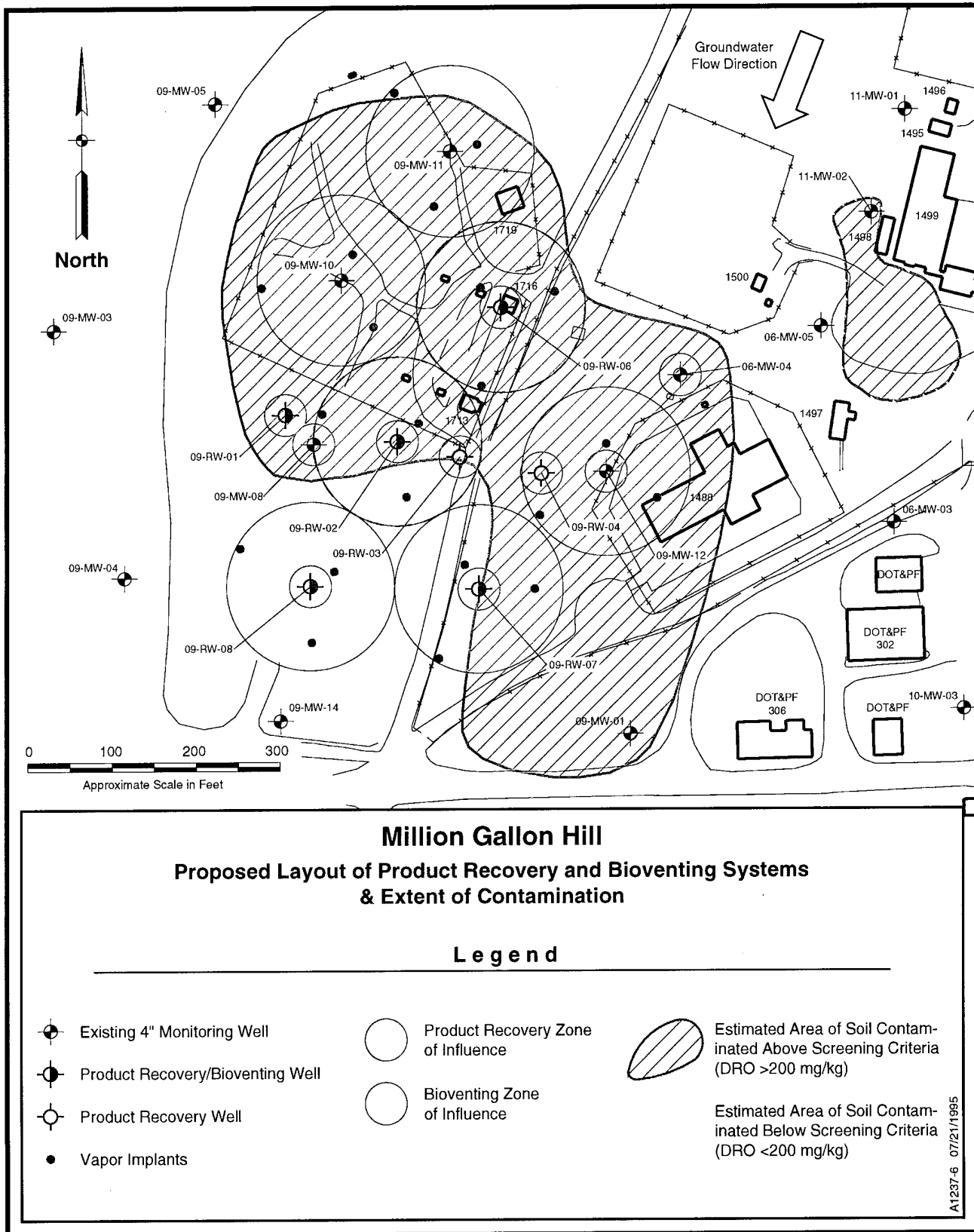


Figure 2-5. Proposed Layout of Product Recovery and Bioventing Systems at Million Gallon Hill



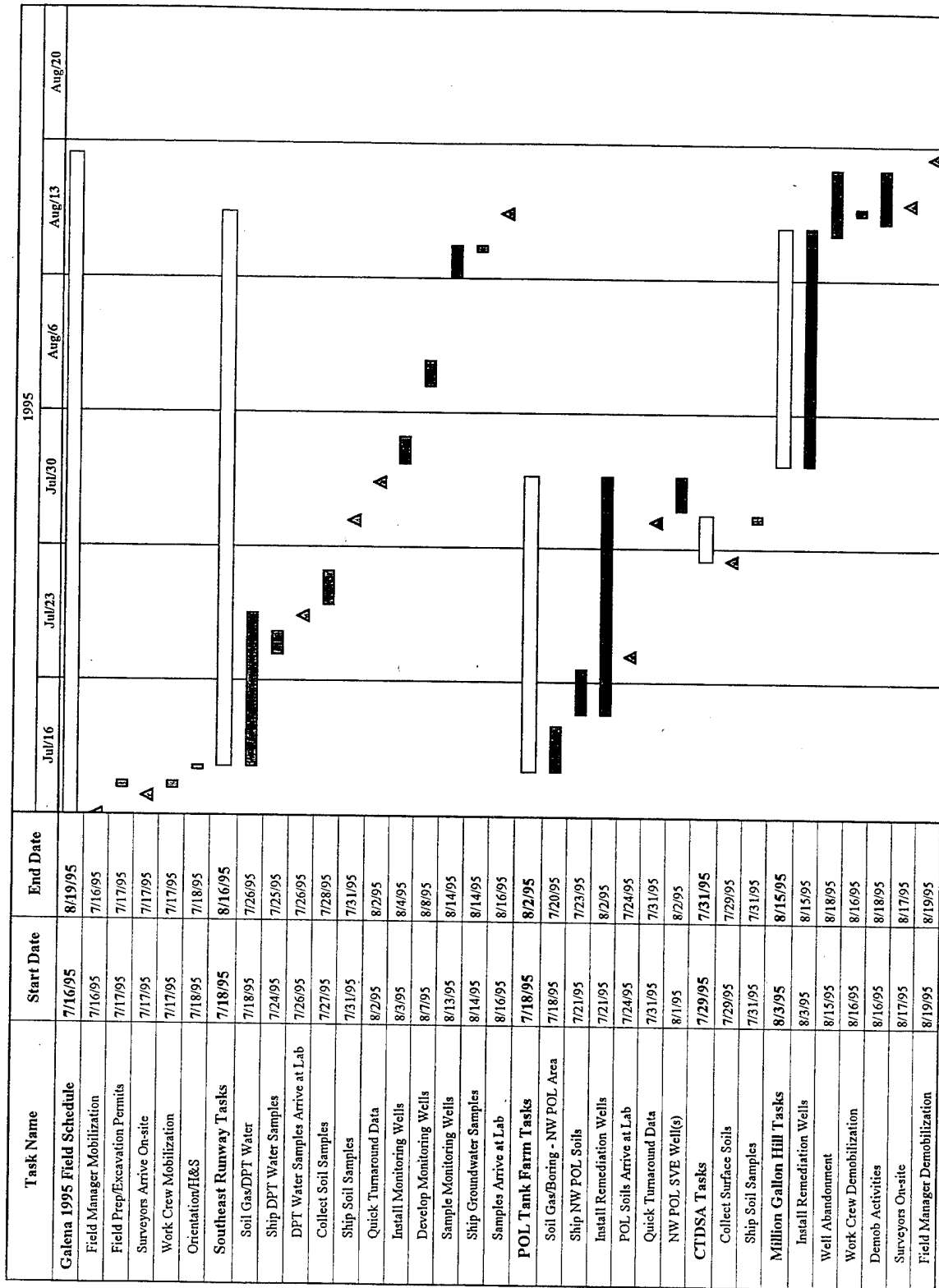


Figure 2-7. Galena Airport 1995 Field Schedule

## Section 3

### FIELD SAMPLING PLAN

The FSP provides details on the methodology used to accomplish the 1995 field tasks. This FSP updates Section 2 of the 1992 SAP (USAF, 1992a) and the 1993 Addendum to the SAP (USAF, 1993a). This section is not intended to be a stand-alone document and must be implemented in conjunction with the 1992 FSP and the 1993 Addendum to the FSP.

#### 3.1 Field Operations

The 1995 field activities in support of the Galena RI include soil gas, soil, and groundwater sampling at the Southeast Runway Fuel Spill site; surface soil sampling at the CTDSA, and soil gas and soil sampling at the northwest portion of the POL Tank Farm, as described in Section 2. Procedures for sampling surface soils and for installing and sampling soil borings and monitoring wells using a hollow-stem auger drill rig are described in the 1992 SAP (USAF, 1992a) and are not repeated here. The sections below update field sampling and screening methods that will be used in 1995 to assess the extent of contamination at the RI sites. Procedures for managing investigation-derived wastes are also updated from the 1993 Addendum to the SAP (USAF, 1993a).

##### 3.1.1 Management of Investigation-Derived Wastes

The term "investigation-derived waste" (IDW) refers to solid or liquid wastes resulting from investigation activities and includes purged groundwater, soil cuttings, and wastewater generated by decontamination activities. This section discusses a methodology for proper disposal or treatment of IDW according to federal regulation guidance and past experience at Galena Airport.

IDW generated by the 1995 field program in Galena will consist primarily of soil cuttings

from installation of remedial wells in portions of the POL Tank Farm and the Million Gallon Hill area where removal actions are planned to extract free-phase hydrocarbon product and conduct SVE and bioventing remediation. Smaller amounts of liquid IDW will be generated during the removal of silt from these same wells. A much smaller amount of IDW may be generated from RI activities at the Southeast Runway Fuel Spill site if monitoring wells are required in that location. Finally, IDW will be generated by decontamination of drilling and sampling equipment.

All IDW will be drummed, labeled, and dated as it is produced, following the procedures outlined in the 1993 Addendum to the SAP (USAF, 1993a). All drums should be clearly labeled on both the top and sides to enable stacked or palleted drums to be identified. Provided information should include date generated, location ID, type of IDW, depth interval (if soil), and the drum headspace organic vapor concentration. Drums will be moved from the well site to a central waste accumulation area inside the fenced compound of Building 1488 by the 611 CES crew.

IDW generated from areas previously characterized during the RI will not require further testing for treatment and disposal. The 1995 IDW generated during removal actions at these areas will be managed in the same fashion as IDW from previous RI activities at these sites. The management practice for liquid (aqueous) IDW from the POL and Million Gallon Hill areas of fuels contamination has been to treat the drummed wastewaters using an activated charcoal filtration unit and discharge the treated water to the base wastewater treatment system. A burn analysis will be conducted on the free-phase hydrocarbon product to determine whether it can be burned in the waste oil burner at Building 1850. If it is suitable, the product will be used to supplement

the current fuel supply. Management plans for IDW soils previously generated from these areas call for treatment in a biopile.

The 1995 IDW generated from RI activities at the Southeast Runway Fuel Spill site is expected to contain contamination from fuels only and thus can be treated in the same fashion as IDW from the POL Tank Farm and Million Gallon Hill. However, because soil and groundwater contamination at this site has not been completely characterized, the IDW from this area will not be treated until laboratory results are reported. If analytical results confirm the expected nature of contamination, then IDW from the Southeast Runway Fuel Spill site will be managed with IDW from the other sites. Analytical results will be related verbally to the manager as soon as they are available so that IDW may be managed in a timely manner following the procedures outlined in the 1993 Addendum to the SAP (USAF, 1993a). These verbal results will be documented in the field manager's log book.

IDW wastewater generated from sampling equipment decontamination will be collected and drummed for treatment on site using the activated charcoal filtration unit. Wastewater generated by drilling equipment decontamination activities will also be collected in drums and treated on site.

### 3.1.2 Field Screening Methods

Field screening methods will be employed at the Southeast Runway Fuel Spill site and the northwest portion of the POL Tank Farm in order to direct additional activities during the 1995 field season. Section 2 of this document outlines the field screening that will be done at each of these sites. The following subsections describe how the field screening will be conducted. For additional information on field screening methods, refer to the 1993 Addendum to the SAP (USAF, 1993a).

### Soil Gas Surveys

Sampling points for the soil gas screening task will be established by locating a grid over the site. The total number of sampling points at each area will depend on the extent of contamination. A step-out approach will be followed in sampling the grid to more efficiently characterize the area. If no VOCs are detected in a soil gas sample from one point, no further sampling needs to be conducted at points downgradient.

Soil gas samples will be collected with a Geoprobe<sup>®</sup> gas vapor probe equipped with disposable, single-use vapor probes or reusable, retractable tips (see Appendix B). Dedicated tubing is attached to a decontaminated stainless steel fitting that screws into the probe tip after the probe rod is hammered into the soil with an electric rotary hammer drill. The vapor probe tips allow the collection of soil gas from discrete intervals. The exact sample depth will depend on the depth to the water table at the time of sampling. The expected sample depth is approximately 5 ft bgl at the Southeast Runway Fuel Spill site and 10 to 15 ft bgl at the POL Tank Farm.

After the probe is advanced to the desired sampling depth, the rotary hammer drill is removed and a retrieval jack is attached to the probe rod. The probe rod is lifted approximately 2 in. to expose the inlet holes in the retractable tip. Small disposable screens surrounding the inlet holes prevent sand from entering the sampling probe. Soil vapor samples are collected by first pushing the vapor tubing and attached fitting down inside the probe rod and screwing it onto the left-hand threads on the probe tip, then attaching a hand-held vacuum pump to the dedicated tubing and purging approximately 1 L of air from the system. A GasTech catalytic hydrocarbon vapor meter is then attached to the tubing and used to measure the hydrocarbon content in the soil vapor. If the internal pump in the hydrocarbon meter is unable to withdraw sufficient sample volume, the hand pump may be used to fill a 3-L Tedlar<sup>®</sup> bag, which

will be subsequently analyzed with the hydrocarbon meter. The vapor probe may also be redriven in an adjacent location.

### **DPT Water Sampling**

The collection of DPT water samples is very similar to that of soil gas samples. A screen-point sampling probe is advanced to below the water table. The extension tubes are lifted 18 in. to expose the fine mesh stainless steel screen. The dedicated sample collection tubing is attached to the screen-point probe tip and the other end is attached to a reservoir jar equipped with a transfer lid. This jar is then attached to the hand-held vacuum pump, which is used to draw sample into the reservoir jar. The groundwater sample is then transferred to the appropriate sample container(s) for transportation to the field laboratory for analysis. An aliquot of sample is also collected for field determination of pH, conductivity, temperature, and alkalinity. After the sample has been collected, the extension tubing and screen-point sampler are extracted using the retrieval jack. The tubing is discarded and the screen-point sampler is decontaminated prior to reuse. Appendix B contains the manufacturer's standard operating procedure for use of the screen-point sampler.

### **3.1.3 Well Installation and Development**

The following sections outline typical construction practices for monitoring, product recovery, SVE, and bioventing wells, and for installation of vapor probes and thermocouples.

#### **Monitoring Wells**

Refer to the 1992 SAP (USAF, 1992a) for details regarding the installation of monitoring wells. These procedures will be followed for the installation of the monitoring wells during the 1995 field season, with the exception that all 1995 wells will be 4-in. diameter wells.

New monitoring wells are only planned for the Southeast Runway Fuel Spill Area. Three

wells will be installed in this area if DPT groundwater samples indicate that groundwater contamination is present. The three wells will be located so as to provide information on the groundwater flow direction and on groundwater contamination in the source area and downgradient. Because of this area's proximity to the Galena Airport runway, no upgradient wells are planned.

#### **Product Recovery Well Construction**

Product recovery wells are 4-inch diameter wells completed just below the seasonal low water table elevation (approximately 118 ft. above mean sea level). Product recovery pumps, which skim hydrocarbons floating above the water surface, will be installed in these wells. Typical construction of the product recovery wells is shown in Figure 3-1. Field forms for borehole logging and well construction will be completed, as outlined in the 1992 SAP (USAF, 1992a).

A 6 5/8-in. inner diameter hollow-stem auger will be advanced to the total depth of the well. A knock-out plug will be placed over the opening in the auger bit, since heaving sands have historically presented problems during well installation at Galena Airport. This plug will serve to minimize heave and accelerate well construction. A limited amount of clean, raw (unchlorinated) water may be used to equalize hydrostatic pressures in the augers if heaving sands continue to be a problem. In this case the water source and total volume of water added will be recorded on the well construction log. Once the total depth of the hole is reached, the drop hammer and drill rod can be used to knock the plug out of the bit, allowing placement of the well screen and casing. Four inch screen and Schedule 40 PVC (polyvinyl chloride) casing will be used for all product recovery wells.

Placement of the sand pack, bentonite seal, and bentonite grout around the screen and casing will follow standard procedures for monitoring

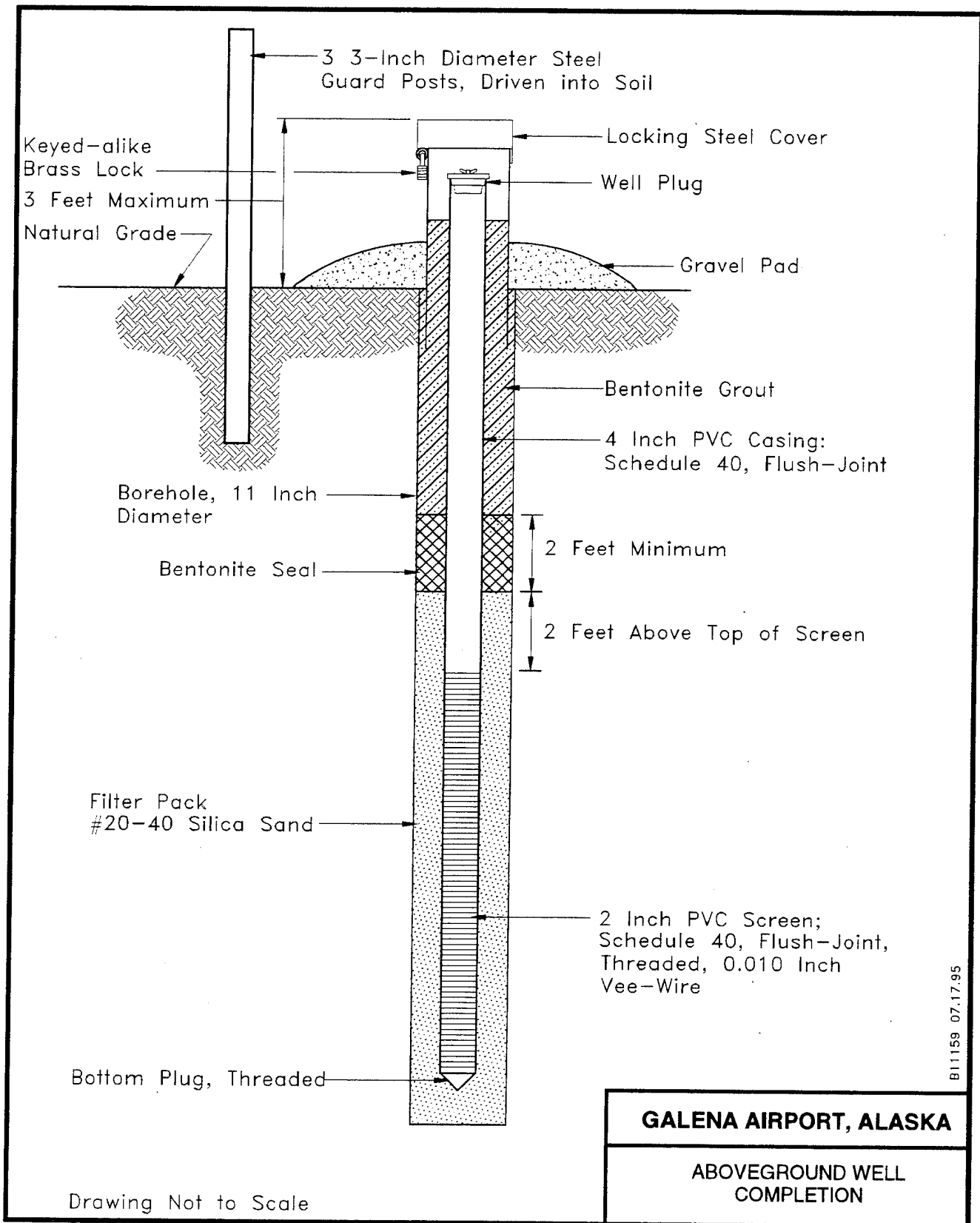


Figure 3-1. Construction Diagram of a Remediation Well

well construction. Screened intervals in all wells will consist of 4-in. Schedule 40, 0.010-in. slot PVC. Sand pack will consist of 20-40 grade silica sand. The sand pack should be completed to a level two feet above the top of the screen and then surged with a vented surge block for ten minutes or until no additional settling is observed. Additional sand is then added, if necessary, and the well surged again. Two ft of bentonite chips are then added over the top of the filter pack and hydrated to provide a seal. If the top of the filter pack is below the water table, no additional water will be required for hydrating the bentonite chips. When bentonite is placed below the water table it is essential to keep the bottom of the augers just above the level of bentonite in the hole. Care should be taken to avoid placing bentonite in the augers below water table because it will hydrate and swell, forming a plug inside the auger which will complicate successful completion of the well.

Surface completion of the product recovery wells will consist of a gravel pad around the well riser and a lockable protective casing. Three steel bollards will be installed around each product recovery well. The bollards will consist of 7-foot long steel pipes driven three to four feet into the ground at a distance three to four feet away from the protective casing to prevent damage to the well.

Product recovery wells will be developed at least 48 hours after installation is completed. Development will be accomplished by pumping approximately 50 gal. of water from the well. A 5-horsepower sump pump with a 2-in. inlet hose will be used for development. The pump inlet hose should be placed near the bottom of the screened interval so that all sediment which entered the well during surging is removed.

#### **SVE and Bioventing Well Construction**

SVE and bioventing well construction will be accomplished using the same methods as for product recovery wells. SVE and bioventing wells

differ from product recovery wells in that they are designed to operate in the vadose zone. However, because of the fluctuating water table elevation at Galena Airport, many of the SVE and bioventing wells will be installed below the seasonal high water table elevation. SVE wells are designed to operate by removing air and volatile hydrocarbons from the soil under moderate vacuum. Bioventing wells are designed to introduce oxygen into the formation and enhance microbial activity by blowing air into the subsurface at low pressure.

Typical construction of a SVE or bioventing well is the same as for product recovery wells (refer to Figure 3-1). SVE or bioventing wells completed below the water table will be developed as for the product recovery wells described above.

#### **Vapor Probe/Thermocouple Installation**

Vapor probes consist of 1-ft by 3/8-in. diameter stainless steel screens attached to 1/4-in. polyethylene tubing. The probes are compatible with the Geoprobe DPT sampling system and can be driven into the soil to the desired depth and left in place for periodic sampling after the drive rod is withdrawn. In hard or rocky formations this direct-push placement method may not be practical, and vapor probe placement may require boring a pilot hole with the hollow-stem augers. Pilot holes may be necessary to the full depth of the probe, or, if resistant formations such as compacted fill are only present near the surface, may extend only to the base of the resistant strata.

Two sets of vapor probes and thermocouples will be implanted at depths corresponding with the screened intervals of each SVE well. One set of probes will be installed near the top of the screened interval of each well and one near the bottom of the screened interval. Each bioventing well will have one set of probes installed opposite the middle of the screened interval. The three probes in each set of implants will be installed at 30%, 66%, and 100% of the assumed 100-ft radius of influence of the well and

will be spaced 120° apart. The orientation of the array of probes will be rotated on adjacent wells to maximize the data on the subsurface air flow which is collected (see Figures 2-4 and 2-5).

Vapor probes and thermocouples will be installed together in each probe location. The vapor probe/thermocouple implant assemblies to be installed each day will be assembled the previous day or will be assembled by a technician in a clean area away from drilling operations. The thermocouple leads for each location will be manufactured to specific lengths to match the placement depth. After selecting the appropriate thermocouple, the lead wire will be taped to the vapor probe tubing before the tubing is attached to the Swageloc® fitting on the probe so that the thermocouple is immediately above the vapor probe screen. Alternatively, if the probe/thermocouple assembly will not fit into the Geoprobe rod in this configuration, the probe wire will be threaded through the vapor probe tubing so that the thermocouple is located at the top of the vapor probe screen.

To install the vapor probe/thermocouple assembly, a post-run tubing implant anchor is fastened into the expendable point holder on the bottom of the Geoprobe drive rod and the rod is driven to the desired depth. To drive the probe rod, an adapter is used on the Geoprobe rod to attach it to a standard drill rod for use with the rig drive hammer. As each section of the probe rod is driven into the ground, new sections are threaded on and driven until the desired implant depth is reached. The drill rod and drive adapter are then removed from the Geoprobe rod and the vapor probe screen and thermocouple assembly are inserted into the Geoprobe rod. The vapor probe is then threaded onto the left-hand threads on the implant anchor end to fix it in place. A slotted drive cap is fastened onto the top of the Geoprobe rods to allow them to be pulled while the tubing remains in place. The rods are pulled up about 6

in. and sand or glass beads (about 1 cup) are poured down the probe rods to form a filter pack around the probe screen. The rods are then pulled up another 6 in. and about ½ cup fine (60 mesh) bentonite is poured down the probe rods to form a seal above the probe. The rods are then withdrawn to the surface, leaving the implants and tubing in place.

Probes installed below the water table will not have a filter pack or bentonite seal because of potential problems with plugging the probe rods. Previous experience at Galena Airport indicates that formation collapse around probes in the saturated zone will provide an adequate filter pack and seal. Figure 3-2 shows a diagram of vapor probe construction.

Vapor probe installation will be documented in the field log book. The total depth of each implant and the number of blows per foot (if appropriate) required to drive the probe rod will also be recorded in the field log book.

Surface completion of the vapor probes is also shown on Figure 3-2. A 5- to 6-ft section of metal conduit is placed over the top of the probe tubing and the thermocouple tubing and pushed down into the hole left by the Geoprobe rod to leave 30 in. sticking up. The end of the vapor probe tubing is trimmed to leave about an extra 1 ft and a piece of flexible silicone tubing is pushed over the end of the polyethylene tubing to allow easy connection to sampling devices. An outdoor-type electrical junction box is fastened onto the top of the conduit so that the extra length of silicone tubing and thermocouple lead can be easily coiled inside. Silicone caulk is used to fasten the polyethylene tubing into the top of the conduit so that the tubing will not be stressed during winter sampling when it is exposed to the cold and is brittle. Vapor probes installed in traffic areas will be completed flush with grade.

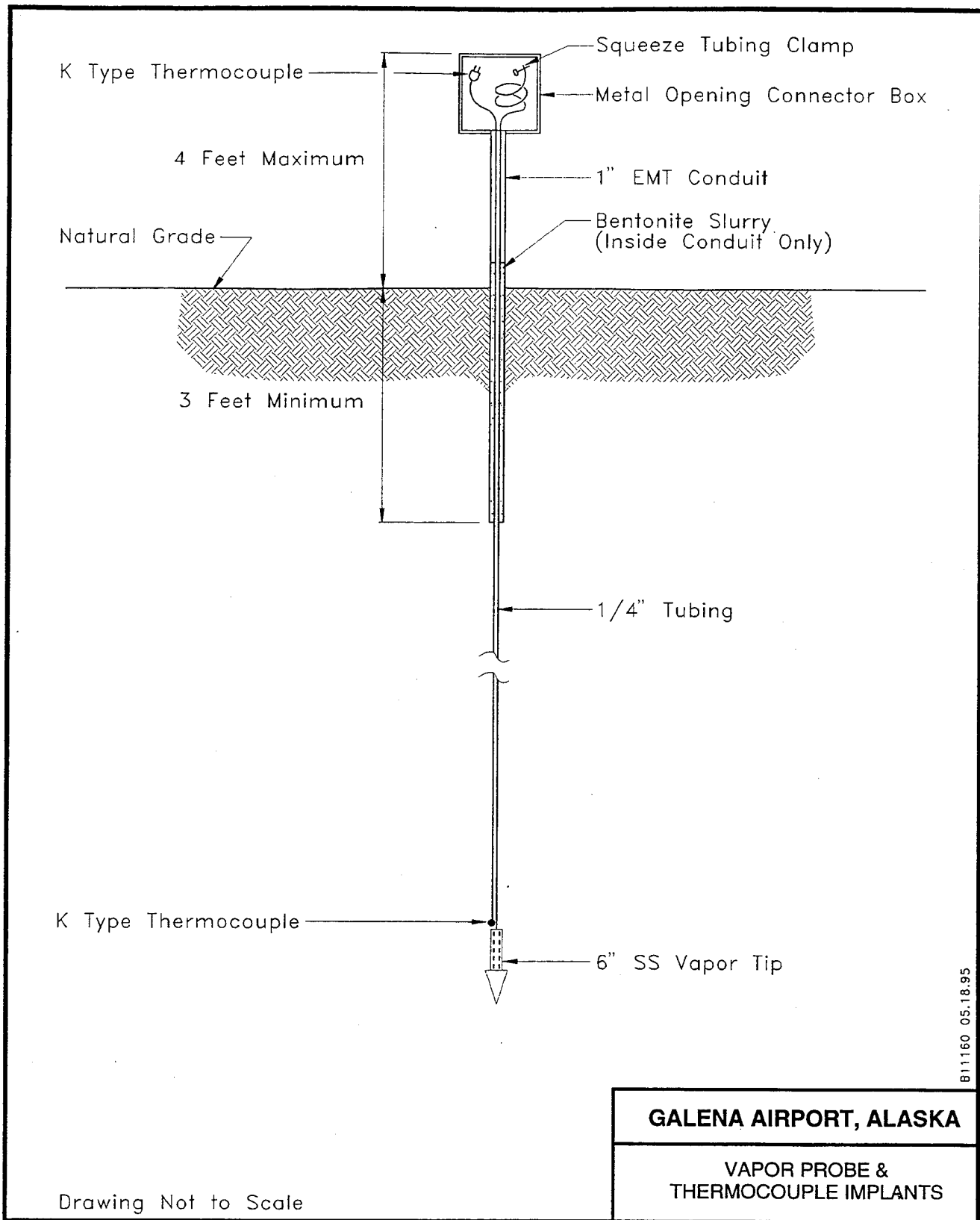


Figure 3-2. Construction Diagram of a Vapor Probe and Thermocouple Implant

### 3.2 Environmental Sampling

Environmental samples will be collected at three sites (Southeast Runway, POL Tank Farm, and CTDSA) in support of the risk assessment and/or remedial design activities. For more information regarding sample handling and documentation, refer to the 1992 SAP (USAF, 1992a).

#### 3.2.1 Soil Sampling

Soil samples will be collected as described in the 1992 FSP, with the exception of subsurface soil samples collected at the Southeast Runway Fuel Spill site. The proximity of this site to the Galena Airport runway makes it desirable to limit the amount of time the drill rig is on site. Therefore, the subsurface soil samples will be collected by boring a pilot hole to the desired sample interval with a decontaminated hand auger, then pushing a brass sampling sleeve into the soil samples collected for VOCs will be submitted to the laboratory in the brass sleeves. Following withdrawal of the brass sampling sleeve, a sheet of Teflon will be placed over each end and plastic end caps attached. The edges of the plastic caps will then be sealed with adhesiveless silicone tape. For nonvolatile analytes, the brass sleeve may be emptied into a glass jar.

#### 3.2.2 Groundwater Sampling

Monitoring wells will be purged and sampled according to the procedures outlined in the 1992 SAP (USAF, 1992a).

#### 3.2.3 Well Development

Monitoring wells at Galena Airport that have been deemed damaged beyond repair or are no longer necessary will be abandoned in accordance with State of Alaska Department of Environmental Conservation. A well

abandonment plan is being developed and will be submitted as a separate document.

### 3.3 Site Management

This section briefly discusses the general aspects of Site Management including the identification of key 611 CES, AFCEE/ERD, and Galena Airport personnel. The 1992 SAP gives more details regarding the duties of management and field personnel (USAF, 1992a).

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#### IRP Project Manager

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Fax: (907) 552-9562/1533

#### Space Mark Contact in Galena

Ted Alexander  
Space Mark  
Galena Airport, AK 98723  
(907) 446-3351

#### 3.3.1 Field Management

One field manager will be on site throughout the summer field program to ensure task execution and continuity. Depending on the active tasks, several field technicians (geologists, engineers, and other disciplines) will be on site to install remedial equipment, sample water and soil, or perform tests.

## Section 4

### QUALITY ASSURANCE PROJECT PLAN

This section serves as the 1995 addendum to the QAPP, and describes two analytical methods (EPA 524.2 and EPA 525) that have not been previously used for the Galena Airport RI. For other analytical methods, refer to Section 1 of the 1994 Addendum to the SAP (USAF, 1994).

#### 4.1 Identification of Methods

Methods to be used for sample analysis are presented in Table 4-1. Methods identified in this document were published in U.S. Environmental Protection Agency (EPA) (1988, 1989) and are presented in Appendix C.

**Table 4-1**  
**Sample Analysis Methods**

Parameter	Analytical Methods	
	Water	Soil
Purgeable Organics in Water by GC/MS.	EPA 524.2	NA
Semivolatile Organics in Water by GC/MS.	EPA 525	NA

GC/MS: Gas Chromatography/Mass Spectrometry

#### 4.2 Sampling Procedures

The field manager is responsible for ensuring that samples are collected with properly decontaminated equipment and placed in properly cleaned sample bottles. A summary of the recommended sample containers, volume, preservation, and hold times for each analytical method (EPA Methods 524.2 and 525) is provided in Table 4-2.

#### 4.3 Subcontractors

The competitive bid process was employed to subcontract a qualified laboratory to perform EPA Methods 524.2 and 525 analysis. Lancaster Laboratories, Inc., based in Lancaster, Pennsylvania, was chosen to perform this work.

#### 4.4 Method Description, Quantitation Limits, and Calibration

Analytical methods, corresponding estimated method detection limits (MDL), and key ion abundance criteria for EPA Methods 524.2 and 525 are presented in Tables 4-3 through 4-6, respectively.

##### 4.4.1 EPA Method 524.2—Purge and Trap Purgeable Organics

Volatile organics in samples will be analyzed according to EPA Method 524.2 by using an inert gas to purge the compounds with low water solubility from the sample matrix. The purged organic compounds are removed from the inert gas by passing it through a sorbent trap. After sample has been purged, the sorbent trap is backflushed and heated. This vapor is passed onto a gas chromatographic (GC) column where the compounds are separated and then detected on a mass spectrometer (MS). The species detected and the estimated detection limits are listed in Table 4-3.

The MS is tuned daily, prior to sample analyses, to give an acceptable spectrum for bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are given in EPA Method 524.2 and are listed in Table 4-4. An initial calibration, used for generating response factors, is performed. The initial calibration curve will consist of 4 to 5 points depending on calibration range. The lowest calibration standard will be 2-10 times the MDL. The relative percent difference (RPD) must be less than 20% for the response factors calculated for each of the calibration check compounds used. After initial calibration is successful, a continuing calibration (CCV) is required at the beginning of each 8-hour period in which analyses are performed.

**Table 4-2**  
**Sample Storage and Preservation Requirements**

Parameter	Method	Holding Time	Container	Preservation	Storage Requirements
Purgeable Organics in Water by GC/MS	E524.2	14 days	Three 40-mL glass vials with Teflon seals	25-mg ascorbic acid for samples with residual Cl, pH<2 with HCl	4 °C
Semivolatiles Organics in Water—GC/MS	E525	7 days to extraction, 30 days to analyze extract	Two 1-L glass bottles with Teflon seals	25-mg ascorbic acid for samples with residual Cl, pH<2 with HCl	4 °C

**Table 4-3**  
**Quantitation Limits for EPA Method 524.2**  
**Purge and Trap Purgeable Organics**

Method	Parameter	Analytes	Estimated Method Detection Limits <sup>a</sup>
			Water (µg/L)
EPA 524.2	Purge and Trap Purgeable Organics (Volatile Organics)	Benzene	0.04
		Bromobenzene	0.03
		Bromochloromethane	0.04
		Bromodichloromethane	0.08
		Bromoform	0.12
		Bromomethane	0.11
		n-Butylbenzene	0.11
		sec-Butylbenzene	0.13
		tert-Butylbenzene	0.14
		Carbon tetrachloride	0.21
		Chlorobenzene	0.04
		Chloroethane	0.10
		Chloroform	0.03
		Chloromethane	0.13
		2-Chlorotoluene	0.04
		4-Chlorotoluene	0.06
		Dibromochloromethane	0.05
		1,2-Dibromo-3-chloropropane	0.26
		1,2-Dibromoethane	0.06
		Dibromomethane	0.24
		1,2-Dichlorobenzene	0.03
		1,3-Dichlorobenzene	0.12
		1,4-Dichlorobenzene	0.03
		Dichlorodifluoromethane	0.10
		1,1-Dichloroethane	0.04
		1,2-Dichloroethane	0.06
		1,1-Dichloroethene	0.12
		cis-1,3-Dichloroethene	0.12
		trans-1,3-Dichloroethene	0.06
		1,2-Dichloropropane	0.04
		1,3-Dichloropropane	0.04
		2,2-Dichloropropane	0.35
		1,1-Dichloropropene	0.10
		cis-1,2-Dichloropropene	NS
trans-1,2-Dichloropropene	NS		
Ethylbenzene	0.06		

**Table 4-3**  
**(Continued)**

Method	Parameter	Analytes	Estimated Method Detection Limits*
			Water (µg/L)
EPA 524.2	Purge and Trap Purgeable Organics (Volatile Organics)	Hexachlorobutadiene	0.11
		Isopropylbenzene	0.15
		4-Isopropyltoluene	0.12
		Methylene chloride	0.03
		Naphthalene	0.04
		n-Propylbenzene	0.04
		Styrene	0.04
		1,1,1,2-Tetrachloroethane	0.05
		1,1,2,2-Tetrachloroethane	0.04
		Tetrachloroethene	0.14
		Toluene	0.11
		1,2,3-Trichlorobenzene	0.03
		1,2,4-Trichlorobenzene	0.04
		1,1,1-Trichloroethane	0.08
		1,1,2-Trichloroethane	0.10
		Trichloroethene	0.19
		Trichlorofluoromethane	0.08
		1,2,3-Trichloropropane	0.32
		1,2,4-Trimethylbenzene	0.13
		1,3,5-Trimethylbenzene	0.05
Vinyl chloride	0.17		
o-Xylene	0.11		
m-Xylene	0.05		
p-Xylene	0.13		

\*These are the estimated method detection limits given in EPA Method 524.2.

NS = Method detection limit is not specified in E524.2.

**Table 4-4**  
**BFB Key Ion Abundance Criteria For EPA Method 524.2**

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	Base peak, 100% relative abundance
96	5% to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5% to 9% of mass 174
176	Greater than 95%, but less than 101% of mass 174
177	5% to 9% of mass 176

**Table 4-5**  
**Quantitation Limits for EPA Method 525**  
**Organic Compounds in Water**

Method	Parameter	Analytes	Estimated Method Detection Limits <sup>a</sup>
			Water (µg/L)
EPA 525	Organic Compounds	Acenaphthylene	0.1
		Aldrin	0.1
		Anthracene	0.04
		Atrazine	0.1
		Benzo(a)anthracene	0.04
		Benzo(b)fluoranthene	NA <sup>b</sup>
		Benzo(k)fluoranthene	0.2
		Benzo(g,h,i)perylene	0.04
		Benzo(a)pyrene	0.1
		Butyl benzyl phthalate	0.3
		alpha-Chloradane	0.2
		gamma-Chloradane	0.1
		trans-Chloradane	0.3
		2-Chlorobiphenyl	0.1
		Chrysene	0.04
		Dibenz(a,h)anthracene	0.1
		Di-n-butylphthalate	0.3
		2,3-Dichlorobiphenyl	0.1
		Diethyl phthalate	0.8
		Di(2-ethylhexyl)phthalate <sup>c</sup>	0.6
		Di(2-ethylhexyl)adipate	0.6
		Dimethylphthalate	0.04
		Endrin	0.5
		Fluorene	0.2
		Heptachlor	0.04
		Heptachlor epoxide	0.2
		2,2,3,3,4,4,6-Heptachlorobiphenyl	0.1
		Hexachlorobenzene	0.1
		2,2,4,4,5,6- Hexachlorobiphenyl	0.1
		Hexachlorocyclopentadiene	0.03
		Indeno(1,2,3,c,d)pyrene	0.1
		Lindane	0.1
		Methoxychlor	0.04
		Octachlorobiphenyl	0.2
		Pentachlorobiphenyl	0.1
		Pentachlorophenol	0.3
		Phenathrene	0.01
		Pyrene	0.02
		Simazine	0.2
		Tetrachlorobiphenyl	0.1
2,4,5-Trichlorobiphenyl	0.06		
Alachlor	1.0		

<sup>a</sup>These are the estimated method detection limits given in EPA Method 525.

<sup>b</sup> Not available; coelutes with benzo(k)fluoroanthene.

<sup>c</sup> Bis(2-ethylhexyl)phthalate.

**Table 4-6**  
**DFTPP Key Ion Abundance Criteria for EPA Method 525**

<b>Mass</b>	<b>Ion Abundance Criteria</b>
51	10% to 80% of the base peak
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	10% to 80% of the base peak
197	Less than 2% of mass 198
198	Base peak >50% of 442
199	5% to 9% of mass 198
275	10% to 60% of the base peak
365	Greater than 1% of the base peak
441	Present, but less than mass 443
442	Base peak or >50% of mass 198
443	15% to 24% of mass 442

#### **4.4.2 EPA Method 525—Semivolatile Organics in Water**

The semivolatile compounds are extracted from the water sample by passing a 1-L sample through a cartridge containing 1 g of C<sub>18</sub> organic phase bonded onto a solid inorganic matrix (liquid-solid extraction, LSE). Organic compounds trapped by the column are eluted from the LSE cartridge using a small quantity of methylene chloride. This methylene chloride extract is then reduced in volume by evaporation. The extract components are separated, identified, and measured by injecting an aliquot of the concentrated extract into a high-resolution fused silica capillary column of a GC/MS system. The species detected and the estimated detection limits are listed in Table 4-5.

The MS is tuned daily, prior to sample analysis, to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). Relative ion abundance criteria for this compound are given in EPA Method 525 and are listed in Table 4-6. A 6-point initial calibration, used for generating response factors, is performed. The RPD must be less than 30% for the six response factors calculated for each of the calibration check compounds used. After initial calibration is successful, a CCV is required at the beginning of each 8-hour period in which analyses are performed.

#### **4.5 Internal Quality Control Checks for Laboratory Operations**

Control limits and acceptance criteria for EPA Methods 524.2 and 525 are presented by method in Tables 4-7 through 4-10.

**Table 4-7**  
**Quality Control Acceptance Criteria for EPA Method 524.2**  
**Purge and Trap Purgeable Organics**

Analytes	LCS Spike Accuracy (% Recovery)	Precision (RPD)
Benzene	80-120	20
Bromobenzene	80-120	20
Bromochloromethane	80-120	20
Bromodichloromethane	80-120	20
Bromoform	80-120	20
Bromomethane	80-120	20
n-Butylbenzene	80-120	20
sec-Butylbenzene	80-120	20
tert-Butylbenzene	80-120	20
Carbon tetrachloride	80-120	20
Chlorobenzene	80-120	20
Chloroethane	80-120	20
Chloroform	80-120	20
Chloromethane	80-120	20
2-Chlorotoluene	80-120	20
4-Chlorotoluene	80-120	20
Dibromochloromethane	80-120	20
1,2-Dibromo-3-chloropropane	80-120	20
1,2-Dibromoethane	80-120	20
Dibromomethane	80-120	20
1,2-Dichlorobenzene	80-120	20
1,3-Dichlorobenzene	80-120	20
1,4-Dichlorobenzene	80-120	20
Dichlorodifluoromethane	80-120	20
1,1-Dichloroethane	80-120	20
1,2-Dichloroethane	80-120	20
1,1-Dichloroethene	80-120	20
cis-1,3-Dichloroethene	80-120	20
trans-1,3-Dichloroethene	80-120	20
1,2-Dichloropropane	80-120	20
1,3-Dichloropropane	80-120	20
2,2-Dichloropropane	80-120	20
1,1-Dichloropropene	80-120	20
cis-1,2-Dichloropropene	80-120	20
trans-1,2-Dichloropropene	80-120	20
Ethylbenzene	80-120	20

**Table 4-7**  
**(Continued)**

<b>Analytes</b>	<b>LCS Spike Accuracy (% Recovery)</b>	<b>Precision (RPD)</b>
Hexachlorobutadiene	80-120	20
Isopropylbenzene	80-120	20
4-Isopropyltoluene	80-120	20
Methylene chloride	80-120	20
Naphthalene	80-120	20
n-Propylbenzene	80-120	20
Styrene	80-120	20
1,1,2-Tetrachloroethane	80-120	20
1,1,2,2-Tetrachloroethane	80-120	20
Tetrachloroethene	80-120	20
Toluene	80-120	20
1,2,3-Trichlorobenzene	80-120	20
1,2,4-Trichlorobenzene	80-120	20
1,1,1-Trichloroethane	80-120	20
1,1,2-Trichloroethane	80-120	20
Trichloroethene	80-120	20
Trichlorofluoromethane	80-120	20
1,2,3-Trichloropropane	80-120	20
1,2,4-Trimethylbenzene	80-120	20
1,3,5-Trimethylbenzene	80-120	20
Vinyl chloride	80-120	20
o-Xylene	80-120	20
m-Xylene	80-120	20
p-Xylene	80-120	20
<b>Surrogates</b>		
1,2-Dichlorobenzene-d <sub>4</sub>	80-120	20
4-Bromofluorobenzene	80-120	20

Table 4-8  
Summary of Calibration and Internal Quality Control Procedures for EPA Method 524.2

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action*
EPA 524.2	Purge and Trap Purgeable Organics Refer to: EPA 524.2	Mass scale calibration using BFB	Initially, prior to calibration, again prior to sample analyses, and once per every 8-hour shift.	See Table 4-4.	Repeat calibration.
		Initial calibration	Initial calibration and when daily calibration verification fails to meet acceptance criteria.	Percent relative standard deviation (RSD) $\leq 20\%$ .	Identify and repeat outlying point(s); recalculate curve using repeated points.
		Daily calibration check	Once per each 8-hour period, prior to sample analysis.	Agreement within 30% of value predicted from the most recent continuing calibration check and within 50% of the initial multipoint calibration.	Accuracy: 1) Repeat calibration verification. 2) If still out, identify and correct problem, run calibration verification again; if still out, recalibrate.
		Laboratory control sample (fortified reagent water)	One for each batch of up to 20 samples.	Recovery 80-120% with RSD $\leq 20\%$ .	If recovery criteria not achieved, problem must be located and corrected before additional samples are analyzed.
		Surrogate spikes	Every sample, standard, and reagent blank.	Recovery 80-120%.	1) Recalculate result, if still out. 2) Check instrument performance, take corrective action, if necessary. 3) Reanalyze sample, if still out. 4) Flag result if it does not meet criteria and document that 1 through 3 were performed.
EPA 524.2 (cont'd)	Purge and Trap Purgeable Organics Refer to: EPA 524.2	Method blank	Once per sample batch (up to 20 samples per batch).	No analytes > method reporting limits except for methylene chloride that are <3 times the method reporting limit.	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) All samples processed with a contaminated blank are to be reextracted and reanalyzed at no cost to the Air Force. 4) Sample results associated with method blank contamination at $\leq 3$ times the reporting limit are to be flagged.

\*All corrective actions with USAF project work shall be documented and the records maintained by the laboratory, as specified in the Handbook for the Installation Restoration Program (IRP); Remedial Investigations and Feasibility Studies (RI/FS); Air Force Center for Environmental Excellence (AFCEE) (USAF, 1993c).  
NA = Not applicable.

**Table 4-9**  
**Quality Control Acceptance Criteria for EPA Method 525**  
**Organic Compounds in Water**

Analyte	LCS Spike Accuracy (% Recovery)	Precision (RPD)
Acenaphthylene	70-130	30
Aldrin	70-130	30
Anthracene	70-130	30
Atrazine	70-130	30
Benzo(a)anthracene	35-130	30
Benzo(b)fluoranthene	35-130	30
Benzo(k)fluoranthene	35-130	30
Benzo(g,h,i)perylene	35-130	30
Benzo(a)pyrene	35-130	30
Butyl benzyl phthalate	70-130	30
alpha-Chloradane	70-130	30
gamma-Chloradane	70-130	30
trans-Chloradane	70-130	30
2-Chlorobiphenyl	70-130	30
Chrysene	35-130	30
Dibenz(a,h)anthracene	35-130	30
Di-n-butylphthalate	70-130	30
2,3-Dichlorobiphenyl	70-130	30
Diethyl phthalate	70-130	30
Di(2-ethylhexyl)phthalate	70-130	30
Di(2-ethylhexyl)adipate	70-130	30
Dimethylphthalate	70-130	30
Endrin	70-130	30
Fluorene	35-130	30
Heptachlor	70-130	30
Heptachlor epoxide	70-130	30
2,2,3,3,4,4,6-Heptachlorobiphenyl	70-130	30
Hexachlorobenzene	70-130	30
2,2,4,4,5,6- Hexachlorobiphenyl	70-130	30
Hexachlorocyclopentadiene	70-130	30
Indeno(1,2,3,c,d)pyrene	70-130	30
Lindane	70-130	30
Methoxychlor	70-130	30
Octachlorobiphenyl	70-130	30
Pentachlorobiphenyl	70-130	30
Pentachlorophenol	70-130	30
Phenathrene	35-130	30
Pyrene	35-130	30
Simazine	70-130	30
Tetrachlorobiphenyl	70-130	30
Toxaphene	70-130	30
2,4,5-Trichlorobiphenyl	70-130	30
Alachlor	70-130	30
<b>Surrogates</b> Perylene-d <sub>12</sub>	70-130	30

These are the limits listed in EPA Method 525.

**Table 4-10**  
**Summary of Calibration and Internal Quality Control Procedures for EPA Method 525**

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 525	Organics in Water Refer to: EPA 525	Mass scale calibration using DFTTP	Initially, prior to calibration, again prior to sample analyses, and once per every 8-hour shift.	See Table 4-6.	Repeat calibration.
		Six-point calibration	Initial calibration and when daily calibration verification fails to meet acceptance criteria.	Percent relative standard deviation (RSD) $\leq 30\%$ .	Identify and repeat outlying point(s); recalculate curve using repeated points.
		Laboratory control sample (fortified reagent water)	One for each batch of up to 20 samples.	See Table 4-9.	If recovery criteria not achieved, locate and correct problem before additional samples are analyzed.
		Daily calibration check	Once per each 8-hour period, prior to sample analysis.	Agreement within 30% of value predicted from multipoint calibration.	Accuracy: 1) Repeat calibration verification. 2) If still out, identify and correct problem, run calibration verification again; if still out, recalibrate.
		Surrogate spikes	Every sample, spike, standard, and reagent blank.	Recovery 70%-130%.	1) Recalculate result, if still out. 2) Check instrument performance, take corrective action, if necessary. 3) Reanalyze sample, if still out. 4) Flag result if it does not meet criteria and document that 1 through 3 were performed.
EPA 525 (cont'd)	Organics in Water Refer to: EPA 525	Method blank	Once per extraction batch (up to 20 sample/batch) and each time there is a new batch of LSE cartridges.	No analytes > method reporting limits except for phthalates that are <3 times the method reporting limit.	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) All samples processed with a contaminated blank are to be reextracted and reanalyzed at no cost to the Air Force. 4) Sample results associated with method blank contamination at $\leq 3$ times the reporting limit are to be flagged.

\*All corrective actions with USAF project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook (USAF, 1993c).  
NA = Not applicable.

## Section 5

### HEALTH AND SAFETY PLAN

No activities that present any new hazards to health and safety will be conducted at Galena Airport during the 1995 field season. Therefore, refer to the *Health and Safety Plan, Galena and Campion Air Force Stations, Alaska* (USAF,

1993d) for all site procedures concerning health and safety. Table 5-1 provides updated emergency and information phone numbers.

**Table 5-1**  
**Emergency and Information Numbers, Galena**

Organization	Number
Fire Department for Emergency Only	911
Police Department	656-1303
Galena City Health Clinic	656-1266
Galena Public Utilities	656-1444
State Troopers	656-1233
BLM—To Report Forest Fires	656-1222
Air Ambulance, Humana Hospital, Alaska	(907)258-3822
Poison Control Center, Providence Hospital	800-478-3193
Providence Hospital Emergency Department	(907)261-3111
Search and Rescue Air Force Rescue Coordination Center, Elmendorf AFB	Call Collect 552-5375

## Section 6

### REFERENCES

- U.S. Air Force. *Installation Restoration Program Phase I: Records Search, AAC-Northern Region*. 1985.
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- U.S. EPA. *Method 524.2. Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*. Environmental Monitoring Systems Laboratory, Office of Research and Development. Cincinnati, Ohio. Revision 2.0. 1986.
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U.S. EPA. *Method 525. Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry*. Environmental Monitoring Systems Laboratory, Office of Research and Development. Cincinnati, Ohio. Revision 1.0, 2.0, 2.1. 1988.

**APPENDIX A**  
**Statement of Work**

**STATEMENT OF WORK**

**for**

**PRELIMINARY ASSESSMENT/SITE INSPECTION  
AT KALAKAKET CREEK RADIO RELAY STATION (RRS), AK**

**and**

**REMEDIAL INVESTIGATION/FEASIBILITY STUDY AT  
GALENA AIRPORT & CAMPION AFS, AK**

**Date: 12 March 1995**

## **I. INTRODUCTION**

### **1.0 PURPOSE**

The purpose of this Statement of Work (SOW) is to provide services, technical man-hours and materials for toxic and hazardous contamination studies; water and wastewater treatment plant investigations, geological, geophysical and geotechnical investigations; hydrogeological studies; bioassay and relative potency determinations; limnological studies; jar testing, drum testing and pilot plant investigations; laboratory testing and/or field evaluations of environmental equipment and landfill leachate monitoring and landfill siting investigations; of environmental waste sites. In addition, this SOW is to provide services for the collection, testing, analysis and reporting of contaminants present in soil, water and wastewater samples in support of Air Force Hazardous and Toxic Waste Programs.

### **1.1 SCOPE**

**1.1.1** In carrying out any work assignment issued, the Contractor shall furnish the necessary personnel, services, equipment, materials, facilities and otherwise do everything necessary for or incidental to, the performance of work set forth herein.

**1.1.2** Primary services shall include, : Services to perform Preliminary Assessment/Site Inspection (PA/SI) at Kalakaket RRS, Alaska, Remedial Investigation/Feasibility Studies (RI/FS) and to support conceptual modeling for Galena Airport and Campion AFS, Alaska This shall include, environmental monitoring, studies and modeling (production of drawings, plans, and specifications) in accordance with the final Decision Documents for Galena Airport and Campion AFS, AK.

**1.1.3** Secondary services incidental to these services include technical requirements found in Annex A of the Basic SOW. They include topographical and geophysical surveys, sampling of soil, tank, drum and pipeline contents; treatability studies, bench scale and/or pilot studies necessary to obtain data to establish/verify the extent and parameters of remediation activities.

## **II. GUIDANCE DOCUMENTS**

### **2.0 Handbook**

The Handbook to Support the Installation Restoration Program (IRP) Statements of Work, dated May 1991, referred to in this SOW as "The Handbook," is provided under separate cover as general guidance only. Any reference within the Handbook language regarding compliance and/or formats for reports as a requirement of this Delivery Order shall be considered deleted. If a conflict is identified between this general guidance and any OSWER, U.S. Environmental Protection Agency (EPA), or other regulatory guidance or requirements, the Handbook shall be disregarded. Also, references to requirements for approval for deviations throughout the Handbook shall be considered invalid. Finally, the Method Detection Limits (MDLs) identified in the Handbook are a consolidation of numerous Code of Federal Regulations (CFR) documents which incorporate current EPA requirements. However, the Contractor shall be responsible for incorporating applicable information available in any updates in the CFR.

### **2.1 Background Guidance**

The following are guidance documents which provide direction for, or otherwise outline, the scope of Air Force major environmental quality activities. These assessments, studies, design activities, and additional related technical activities, as may be required, shall be performed in accordance with rules and regulations set forth by the U.S. Environmental Protection Agency (US EPA), Occupational Safety and Health Administration (OSHA), Nuclear Regulatory Commission (NRC), Food and Drug Administration (FDA), other federal agencies, individual state regulatory agencies, foreign regulations, international laws, treaties and agreements, as well as applicable requirements of other guidance documents including, but not limited to, the most current versions of the applicable portions of the documents cited below:

- a) Occupational Safety and Health Administration (OSHA) regulations.
- b) Department of Transportation regulations.
- c) National Environmental Policy Act (NEPA).
- d) Clean Water Act (CWA).
- e) Clean Air Act (CAA).
- f) Endangered Species Act (ESA).
- g) Toxic Substances Control Act (TSCA).
- h) Resource Conservation and Recovery Act (RCRA), as amended by the Hazardous and Solid Waste Amendments.
- i) Comprehensive Environmental Response Compensation and Liabilities Act (CERCLA) as amended by the Superfund Amendments and Reauthorization Act (SARA).
- j) National Oil & Hazardous Substances Contingency Plan (NCP) 40 CFR 300
- k) Air Force Engineering Technical Letters (AF ETLs).
- l) Guidance for Oversight of Remedial Designs and Remedial Actions Performed by Potentially Responsible Parties, Interim Final U.S. Environmental Protection Agency

(EPA)/540/G-90/OOI; EPA Office of Solid Waste and Emergency Response (OSWER) Directive 9355.5-01, 4/90.

m) Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (OSWER Directive 9335.3-01), 1988.

n) Risk Assessment Guidance and Superfund, Volume 1, Human Health Evaluation Manual (Part A), Interim Final (EPA/540/1-89/002), 1989.

o) Risk Assessment Guidance and Superfund, Volume 2, Environmental Evaluation Manual, Interim Final (EPA/540/1-89/001), 1989.

p) Test Methods for Evaluating Solid Waste (SW-846), Third Edition (1986), and 1987 updates.

q) Guidance on Remedial Action for Contaminated Groundwater at Superfund Sites (OSWER Directive 9283.1-2), 1988.

r) A Compendium of Superfund Field Operation Methods, (EPA/540/P-87/OOI; OSWER Directive 9335.0-14), Dec 1987.

s) National Fire Protection Association Standards

t) AFM 88-29, Engineering Weather Data, 1 Jul 1978.

u) National Standard Plumbing Code

v) HQ AFCEE Handbook for the Installation Restoration Program (IRP) Remedial Investigations and Feasibility Studies (RI/FS), dated Sept 1993, referred to as "The Handbook".

w) Project-specific Quality Program Plans (QPP) prepared by the Contractor. Includes Sampling and Analysis Plans (SAP), Health and Safety Plans (HSP), and Quality Assurance Project Plans (QAPP).

x) OSWER 9345.0-01, Section 2.0 - Guidance for Conducting New Preliminary Assessments

y) American Petroleum Institute

aa) Section 1447(a) of the Safe Drinking Water Act, Public Law 93-523, et. seq.

ab) Executive Order (EO) 12088, Federal Compliance with Pollution Control Standards, 13 October 78

ac) Code 40 of Federal Regulations (CFR), Chapter I and V, Protection of Environment.

ad Air Force Regulations (AFR) 19-1, "Pollution Abatement and Environmental Quality," 9 Jan 78.

ae) AFR 19-2, "Environmental Impact Analysis Process (EIAP)," 23 Sep 81.

af) AFR 19-6, "Air Pollution Control Systems for Boilers and Incinerators," Mar 88.

ag) AFR 19-7, "Environmental Pollution Monitoring," 19 Apr 85.

ah) AFR 19-8, "Environmental Protection Committees and Environmental Reporting," Aug 88.

ai) AFR 19-9, "Interagency and Intergovernmental Coordination of Land, Facility and Environmental Plans, Programs and Projects," 14 Feb 86.

aj) AFR 19-10, "Planning in the Noise Environment," 15 Jun 78.

ak) AFR 19-11, "Hazardous Waste Management and Minimization," Jul 89

- al) AFR 19-14, "Management of Recoverable and Unusable Liquid Petroleum Products," Aug 90
- am) AFR 91-8, "Solid Waste Management" Mar 90
- an) AFR 161-17, "USAF Occupational and Environmental Health Laboratory (OEHL) Services," 3 Aug 81.
- ao) AFR 161-44, "Management of the Drinking Water Surveillance Program," 29 May 79.
- ap) "Defense Environmental Quality Program Policy Memorandum
- aq) E.O. 12316, "Response to Environmental Damage," 14 August 1981.

### **III. GENERAL REQUIREMENTS**

#### **3.0 MEETINGS, CONFERENCES, SITE VISITS**

##### **3.0.1 Post Award Meeting**

After the issuance of a delivery order, the Contractor shall attend a post award meeting at the base, or other location specified by the Contracting Officer's Representative (COR). The purpose of the meeting shall be to familiarize the Contractor with the work and/or hazardous waste sites addressed under the delivery order.

##### **3.0.2 Progress Meetings**

The Contractor shall attend progress meetings with the base and Air Force Center for Environmental Excellence (AFCEE) as specified by the AFCEE COR. The Contractor shall be responsible for preparing minutes from each of the meetings. The contractor shall deliver the minutes to AFCEE ten (10) working days after the completion of the meeting.

##### **3.0.3 Design Integration Meetings.**

Not applicable

#### **3.1 Special Notification**

##### **3.1.1 Health Risks**

The contractor shall immediately report to the AFCEE COR, and the Base point-of-contact (POC), via telephone, any data or results generated during investigations pursuant to delivery orders which may indicate any potential imminent health risk to contracted or federal personnel, or the public at large. Following this telephone notification, a written notice with supporting documentation shall be prepared and delivered within three (3) working days. Upon request of the Air Force, the contractor shall provide pertinent raw laboratory data (i.e. chromatograms) within three (3) weeks of the telephone notification.

##### **3.1.2 Change of Contractor Personnel**

An organizational chart displaying key personnel involved in the effort and their respective labor categories shall be submitted with the first monthly Status Report. The Contractor shall notify the AFCEE COR of all professional personnel to work on specific tasks. The Contractor shall notify the AFCEE COR of any significant changes in project personnel along with the steps that the Contractor is taking to ensure there are no impacts to the schedule or individual tasks.

## **3.2 Laboratories**

### **3.2.1 General**

The Contractor shall submit laboratory reporting limits and the methods by which they were derived to the AFCEE COR concurrently along with a laboratory Quality Assurance Project Plan (QAPP) prior to usage of a laboratory. All laboratories shall be capable of meeting Data Quality Objectives (DQOs) specified in the project-specific Sampling and Analysis Plan (SAP). All laboratories shall screen for analytes and perform Quality Assurance/Quality Control (QA/QC) requirements as specified in the project/site specific SAP. All analyses shall be reported on a dry weight basis to facilitate comparison with the off-site laboratory data. The analytical capabilities of the all laboratories shall be sufficient for the methods specified in the SAP, and all laboratories shall have sufficient through-put capacity to handle the necessary analytical load during all field activities.

### **3.2.2 On-site Laboratories**

Not applicable

## **3.3 Work-site Requirements**

### **3.3.1 Safety Requirements**

The contractor shall provide for protecting the lives and health of employees and other persons; preventing damage to property, materials, supplies, and equipment; and avoiding work interruptions. For these purposes, the contractor shall comply with OSHA Safety and health regulations and pertinent provisions of the Air Force Occupational Safety and Health Standard (AFOSH).

### **3.3.2 Work-site Maintenance**

The work-site shall be maintained in accordance with the requirements of Section 2.1 of the Handbook so as to: 1) prevent the spread of contamination, 2) provide for the integrity of the samples obtained, and 3) provide for the safety of federal workers, contracted personnel, and/or other individuals in the vicinity of the project areas.

The work site shall be well marked to prevent inadvertent entry into all work areas. Access to work areas shall be monitored and thoroughly controlled. Standard work zones and access points for hazardous waste operations shall be established and maintained as the site conditions warrant. The contractor shall, at all times, keep the work area free from accumulation of waste materials. The contractor shall remove non-essential equipment from the work site when not in use. The work-site shall be maintained to present an orderly appearance and to maximize work efficiency.

Before completing the work at each sampling site, the contractor shall remove, from the work premises, any rubbish, tools, equipment, and materials that are not property of the Government. Upon completing the work, the contractor shall leave the area clean, neat, orderly, and return work sites to the original condition. The contractor shall also ensure compliance with any federal and state regulations for decontaminating tools, equipment, or other materials, as required.

### **3.3.3 Operations Impact Minimization**

The contractor shall mark the field locations of all points of ground penetration during the planning/mobilization phase of the field investigation. The base POC shall be consulted to properly position sampling locations (wells, borings, soil gas probes, etc.) with respect to site locations, to minimize the disruption of base activities, and to avoid penetrating underground utilities. Additionally, the contractor may be required to coordinate with other base personnel to attain these objectives. The contractor shall provide for the detection of underground utilities independent of base Civil Engineering services utilizing geophysical or other techniques. All necessary permits shall be obtained, and necessary coordination shall be completed, prior to commencement of individual sampling operations. Frequent communication and coordination with base personnel shall be necessary to accomplish these goals.

### **3.3.4 Storage**

The contractor shall be responsible for the security of his equipment. Contractor's equipment or materials used in the work, requiring storage on base, shall be placed at sites as designated by the Base POC. The contractor shall be responsible for security and weather proofing of any stored material and equipment. Missing or damaged material shall be replaced at no additional cost to the Government. At the completion of the work, all temporary fences and structures (the contractor used to protect materials and equipment) shall be removed from the base. The contractor shall clean the storage area of all debris and material and perform all repairs as required to return the site to its original condition.

### **3.3.5 Security**

The contractor is responsible for obtaining and monitoring contractor security badges for all areas for the duration of this contract. All security badges or passes shall be returned to the Base POC upon expiration of the badge, upon completion of the project, or when possession of the badge is no longer necessary (e.g., upon removal of contracted personnel from specific projects). Photography of any kind must be coordinated through the Base POC or Public Relations representative.

### 3.4 Work Breakdown Structure

In response to Requests for Proposals (RFPs) for individual Delivery Orders (DOs), the contractor shall prepare proposals, project schedules, and monthly financial reports organized according to the following work breakdown structure (WBS):

#### 5 PRELIMINARY ASSESSMENT/SITE INVESTIGATION

- 5.01 PA/SI Scoping
- 5.02 Site Assessment
- 5.03 Soil Borings
- 5.04 Groundwater Monitoring Wells
- 5.05 Sampling and Analysis
- 5.06 Recommendations

#### 10 REMEDIAL INVESTIGATION/FEASIBILITY STUDY

- 10.01 RI/FS Scoping
- 10.02 Development of Alternatives
- 10.03 Site Characterization
- 10.04 Screening of Alternatives
- 10.05 Treatability Investigation
- 10.06 Analysis of Remedial Alternatives
- 10.07 Remedy Selection
- 10.08 Groundwater Monitoring Wells
- 10.09 Sampling and Analysis
- 10.10 Site-work and Utilities

#### **IV. WORK TASKS**

All work performed pursuant to any paragraph of Section IV of this SOW shall comply with the technical requirements of Annex A of the Basic SOW. The work shall be accomplished at Kalakaket RRS, Galena Airport and Campion AFS, AK. The work shall include :

##### **4.0 PLAN DEVELOPMENT**

The Contractor shall prepare for approval by the AFCEE COR a Quality Assurance Project Plan (QAPP) for this work. In addition, the Contractor shall prepare project specific schedules, Work Plans (WPs), Management Action Plan (MAP), Sampling and Analysis Plan (SAP), Field Sampling Plan (FSP), Community Relations Plans (CRPs), and discretely prioritized cost estimates and modeling plans. The CO, the AFCEE COR and the Base POC shall be notified in writing prior to any modification to, or deviation from, any activity described in these documents.

##### **4.1 Delivery Order Scoping**

###### **4.1.1 Pre-survey**

Not applicable

###### **4.1.2 Pre-mobilization Survey**

Not applicable

##### **4.2 Preliminary Assessment/Site Inspection (PA/SI)**

The Contractor shall conduct PA/SI to define the environmental setting of Kalakaket RRS and to identify preliminary sites which may potentially be contaminated, and to develop a preliminary assessment of the potential sources of contamination. The Contractor shall make all preliminary studies of monitoring or sampling locations and accessibility, number of sampling locations, number and type of personnel required, number and type of tests or samples desired, special or modified sampling equipment and procedures required, personnel protective equipment required, and type of analytical protocol or procedures to assure that activities shall comply with US EPA or state NPDES regulations or other laws, regulations or standards which are applicable. Meetings with USAF, US EPA and/or state regulatory agency officials may be required to discuss tentative test plans.

#### **4.2.1 Preliminary Assessment (PA)**

The Contractor shall conduct a literature search to define the installation environmental setting and to identify potentially contaminated sites and potential sources of contaminants. The goals of the PA are to: 1) identify potentially contaminated sites or Areas of Concern (AOC); 2) document the need for no further investigation at sites where CERCLA remedial action is not required; 3) identify sites that require emergency response; 4) compile information necessary to develop preliminary projected Hazard Ranking Scoring; 5) set priorities for SIs; and 6) to develop a preliminary conceptual model for each AOC presenting hypotheses regarding the contaminants present, their potential migration pathways, and their potential impact on sensitive receptors. Sources of information include federal, state, and local agencies, base personnel and former employees, aerial photographs, academic institutions, and reports of previous investigations. Document the findings in a PA report using the guidance in OSWER 9345.0.01. All references, personal communications, etc., shall be cited in an appendix to the report.

#### **4.2.2 Site Inspection (SI)**

The Contractor shall visit the AOCs to ensure a complete understanding of site conditions. Coordinate this visit with the AFCEE COR. The Contractor shall visit and inspect all AOCs identified as requiring further investigation in the PA Report. The Contractor shall look for evidence of contamination at each AOC visited (e.g., leaking drums, vegetative stress, leachate seeps, etc.). The Contractor shall observe the physical setting of each site visited to formulate specific recommendations concerning well and boring placement, use of geophysical techniques, and other aspects of the proposed field investigation. The Contractor shall perform field screening and limited sampling at each of the AOCs. Document the findings in a SI Report. Using the information from the PA/SI, the Contractor shall perform Hazard Ranking Scoring (HRS) for each of the AOCs. The findings of the PA/SI shall be used to prepare the Work Plan and Sampling and Analysis Plan required for the follow-up effort, if needed.

#### **4.3 Remedial Investigation/Feasibility Study (RI/FS)**

##### **4.3.1 Remedial Investigation (RI)**

The Contractor shall conduct a remedial investigation (RI) to characterize environmental conditions, define the nature and extent of contamination, and quantitatively estimate the risk to human health and the environment at AOCs through the collection of geologic, geophysical, hydrogeological, ecological, chemical, physical, and hydrologic data, and environmental samples; the laboratory analysis of those samples for potential contaminants; the evaluation of the analytical results and field measurements with respect to quality control data; and the interpretation and analysis of validated data. The purpose of data collection, sample collection and laboratory analysis is to determine whether any contaminants generated from

installation activities have entered the environment and pose a risk to human health or the environment.

The field investigation is used to determine the source of any identified contaminants, and the magnitude of contamination relative to Applicable or Relevant and Appropriate Requirements (ARARs) and any naturally occurring or background concentrations for specific compounds. The remedial investigation shall comply with the specifications, procedures, and methodologies presented in project-specific SAPs.

#### **4.3.2 Feasibility Study (FS)**

The FS is performed concurrently with the RI. As much of the FS as possible shall be performed early on in the RI/FS process and refined as additional RI data are obtained. Use the information from the RI and the baseline risk assessment to develop and evaluate remedial action alternatives for each site where a threat to human health or the environment exists. Follow the procedures specified in USEPA OSHA Directive 9355.3-01, "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA." Employ streamlining methods wherever possible. Develop and evaluate the minimum number of alternatives needed to provide a range of promising treatment, and containment actions. Eliminate impracticable alternatives from further consideration early in the FS process. The scope and level of detail shall be consistent with the nature and complexity of site problems.

#### **4.4 Remedial Design (RD)**

Not applicable.

#### **4.5 Treatability Studies, Pilot Tests, Bench Scale Tests**

The Contractor shall conduct treatability studies, pilot tests, and/or bench-scale tests to determine optimum methods of contaminant delineation and/or removal of contaminants from soils, ground and surface waters, and the degree of treatment anticipated using various processes. Pilot plant studies shall also be conducted to permit the Air Force to determine the feasibility of the implementation of various environmental processes at selected Air Force facilities. This shall include the development and utilization of innovative site investigation and/or remedial technologies, and cost estimates. The contractor shall implement a treatability study to assess the potential for the optimum technology to treat the pesticide and petroleum oil and lubricant soil stock pile at Galena Airport. In addition, the Contractor shall implement the recovery of free-phase product removal, and innovative treatment technology for trichlorethylene (TCE) from drinking groundwater wells. The Contractor shall include the plans for implementation of the study in the Phase I modeling and monitoring plan pursuant to paragraph 4.7.4.1 and 4.7.4.1.1

## **4.6 Subtasks**

Sub-tasks, shall include the following:

### **4.6.1 Conceptual Site Model**

For each site, use validated data supported by acceptable QA/QC results (as measured against QAPP requirements) and site characterization information to develop or refine, based on newly collected data, the conceptual site model. The model shall define the nature and extent of contamination, the hydrogeologic regime, and the transport and fate of those contaminants. The conceptual site model may be prepared using the minimum requirements given in Section 2 of the Handbook as guidance. The complexity and detail of the site model shall be consistent with the nature of the site and site problems, and the amount of data available. Use the conceptual site model in the baseline risk assessment.

### **4.6.2 Ecological/Baseline Risk Assessment**

For each site, use validated data supported by acceptable QA/QC results (as measured against QAPP requirements) and the conceptual site model to estimate numerically the risk posed by site contaminants to public health and the environment. The methodology in Section 2 of the Handbook may be used as guidance. Identify all Applicable or Relevant and Appropriate Requirements (ARARs) that were not identified in previous reports for those contaminants detected in environmental samples at each site. Provide the results of the baseline and/or ecological risk assessment in the Risk Assessment Technical Memorandum. The formats in the Handbook may be used as guidance.

The Contractor shall Identify those sites posing minimal or no threat to human health, welfare, or the environment and for which no further action is appropriate. Use the results of the risk assessment in establishing remedial action objectives and developing remedial alternatives in the Feasibility Study.

### **4.6.3 Alternatives Development**

Not applicable.

### **4.6.4 Alternatives Analysis**

Conduct a detailed analysis of each alternative selected and identified in par. 2.3.15, and approved by the AFCEE COR. Using the methodology in OSWER Directive 9355.3-01, evaluate each alternative against US EPA's nine criteria for conducting Feasibility Studies. Focus the analysis on sub-factors and criteria most pertinent to each site and the scope and complexity of the proposed action. Select a recommended alternative for each site or operable unit. Provide a summary of the detailed analysis of alternatives following task completion.

Include summary tables of the individual analysis that shall be used in the Remedial Investigation Report. For those sites or zones where sites are grouped together, where a preferred alternative is identified, prepare a decision document after the receipt of Air Force review comments on the Remedial Investigation Report to support the selection process. The format specified in Section 3 of the Handbook may be used as guidance.

#### **4.6.5 Evaluation of Remedial Systems and Environmental Equipment**

The Contractor shall conduct an independent evaluation of remediation systems to determine their effectiveness. This includes the collection of data needed to assess the ability of the remediation system to remediate the site.

The Contractor shall perform laboratory and field tests of environmental monitoring and testing equipment, to include validation of manual/instrumental methods, continuous monitors, analytical support and mathematical models using US EPA, ASTM, NRC, and/or equivalent procedures specified by the Air Force.

#### **4.6.6 Administrative Record**

The Contractor shall update the Administrative Record, provided under a separate cover.

#### **4.7 Other Environmental Activities**

The Contractor shall conduct other investigations, studies, assessments, and/or designs related to environmental issues at Kalakaket RRS, Galena Airport and Campion AFS not described in the PA/SI/RI/FS/RD process described in the sections above. This shall include, RFAs, RFIs, COFAs, the analysis, development and/or utilization of emerging processes and other environmental studies, investigations/, and/or analyses. All work undertaken in accordance with this paragraph shall comply with the technical requirements of Annex A of the Basic SOW.

##### **4.7.1 Not Applicable**

##### **4.7.2 Miscellaneous Analyses**

##### **4.7.3 Environmental Monitoring**

Includes continuous and/or discrete measuring, sampling and analysis of groundwater, surface water, effluent, air emissions, soils and any other environmental media .

#### **4.7.4 Sampling for Remedial Action**

The Contractor shall prepare and implement approved work plans for the geophysical sampling required as part of remedial action contracts. This includes sampling needed to determine the type and quantity of contamination. Sampling shall be conducted on the site being remediated prior to excavation/remediation, as well as on material following excavation/remediation, such as stockpiled materials excavated as part of tank removals. This information is needed to determine remediation required, as well as suitability of stockpiled material for use as backfill.

##### **4.7.4.1 Modeling Plans, Drawings and Specifications**

The contractor shall review all existing IRP documentation associated with the Galena Airport and Campion AFS RI/FS which contributed to the determination of the selected alternatives. This shall include the Galena Airport and Campion AFS RI/FS Report, the Galena Airport and Campion AFS Decision Documents, and all aerial photos. The contractor shall be required to review additional background data information as provided for modeling plans, drawings and specifications for an effective remedial action. The modeling plans, drawings and specifications shall reflect the chosen remedial alternatives for seeps, groundwater and soil.

###### **4.7.4.1.1 Work Plan**

The contractor shall develop a modeling work plan. The work plan shall document the overall management and implementation strategy for modeling plans, drawings and specifications activities. It shall include the responsibilities and authorities of all organizations and key personnel involved in the tasks. Site-specific aspects of the Contractor's proposed work plan shall be detailed, and any deviations from the existing or previous RI/FS Work plan shall be highlighted. The contractor shall receive approval for the work plan before proceeding to the next modeling phase. The work plan shall detail the following areas:

- a) Requirements for additional field data collection
- b) Requirements for treatability studies
- c) Develop requirements for Permits/Access Agreements for RD/RA
- d) Schedule for completion
- e) Modeling criteria
- f) Tentative treatment schemes
- g) Health and Safety Plan for modeling activities
- h) Quality Assurance/Quality Control for modeling activities

#### **4.7.4.1.2 Modeling Phases**

The modeling shall be submitted for AFCEE review in three (3) phases. The following lists the modeling submittal phases and components of the modeling which shall be completed at each phase:

##### **4.7.4.1.2.1 Phase I (Preliminary) Modeling**

Submitted at development of preliminary concept of the modeling at which time the technical requirements of the project have been addressed and outlined. This includes, modeling plans and specifications, cost estimates, project schedule, and analysis and rationale.

##### **4.7.4.1.2.2 Phase II (Intermediate) Modeling**

Submitted at establishment of modeling specifications. This includes, but is not limited to, modeling plans and specifications, cost estimates, analyses and rational, project schedule, operations procedures and maintenance requirements.

##### **4.7.4.1.2.3 Phase III (Final) Modeling**

Submitted at final completion of the modeling, and includes all modeling components from paragraph 4.7.4.1.2.2.

#### **4.7.4.1.3 Modeling Reviews**

The contractor shall submit the stated work at all phases specified: preliminary , intermediate and final. Submittals shall be reviewed by the AFCEE and written comments shall be provided. Disposition of the comments shall be determined at the respective review meeting. The contractor shall incorporate the results of each modeling review into the next required modeling phases submittal.

### **4.8 Deliverables**

#### **4.8.1.1 Monthly Financial and Management Reports**

The Contractor shall submit financial and management reports utilizing the standardized Work Breakdown Structure per paragraph 3.4 of this SOW to describe the status of expenditure of funds correlated with the progress of the work completed. Reports shall provide current status and projected requirements of funds, man-hours, and work completion; indicate the progress of work and the status of the program and assigned tasks; and inform of existing or potential problem areas. (A001, A002, A003)

#### **4.8.1.2 Health and Safety Plan**

The Contractor shall prepare and deliver a Health and Safety Plan to comply with USAF, Occupational Safety and Health Administration (OSHA), US EPA, state, and local health and safety regulations regarding the proposed work effort at Kalakaket RRS. The Contractor shall utilize to the fullest extent possible existing corporate Health and Safety Plans, tailoring them to the current effort. Use US EPA guidelines for designating the appropriate levels of protection needed at the study sites. Coordinate the Health and Safety Plan directly with applicable regulatory agencies prior to submittal to AFCEE. Provide the AFCEE COR with evidence of Health and Safety Plan coordination prior to the start of field work. The Contractor shall certify to AFCEE that it has reviewed the approved Health and Safety Plan with each employee and subcontractor's employees prior to the time each employee engages in field activities. (A004)

#### **4.8.1.3 Management Action Plan (MAP)**

In accordance with paragraph 4.0, the Contractor shall deliver and update the MAP to describe the overall approach, major tasks and scope, time sequencing of events, and major decision points to complete all IRP efforts to ensure consistency with the NCP. This Plan is intended as a planning document and management tool to track the progress of IRP efforts. (A005)

#### **4.8.1.4 Community Relations Plan**

In accordance with paragraph 4.0, the contractor shall finalize the Community Relations Plan (CRP) for Galena Airport and Campion AFS, provided under a separate cover, outlining the specific public communication and involvement techniques to be used in coordination with remedial site activities. Follow the guidance contained in OSWER Directive 9230.0-3b, "Community Relations in Superfund, A Handbook." Propose a detailed format for the CRP consistent with this guidance for AF and AFCEE approval prior to preparing the plan. The CRP shall include a description of the site and the community, an overview of the community involvement to date, key community concerns regarding the site and AF site activities. A list of elected officials, agency representatives, and interested groups and individuals shall be included. Contractor activities to develop the CRP shall include conducting a review of site information provided by the base. (A005)

##### **4.8.1.4.1 Photo Notebook Update**

The Contractor shall update the Photo Notebook, provided under a separate cover. After review and approval by the COR and the 611 CES/CEVR Community Relations Coordinator, the Contractor shall submit final copies of the photo notebooks (A005)

#### **4.8.1.4.2 Community Meeting Support**

The Contractor shall attend community meetings and shall provide public information posters and fact sheet handouts at the meetings. The Contractor shall also provide information on the use of the Administrative Record at Galena and Elmendorf AFB, Alaska (A005)

#### **4.8.1.4.3 Newsletters**

The contractor shall prepare up to a six (6)-page newsletters to provide the community with background information on investigation, remediation approaches, technology and regulatory issues and other pertinent information on the IRP. After review and approval by the COR and the 611 CES/CEVR Community Relations Coordinator, the contractor shall submit final copies of each newsletter (A005).

#### **4.8.1.4.4 Fact Sheets**

The contractor shall prepare two (2)-sided fact sheets to provide the community with updates on IRP activities. These fact sheets shall be produced quarterly. After review and approval by the COR and the 611 CES/CEVR Community Relations Coordinator, the contractor shall submit final copies of each Fact Sheet. (A005)

#### **4.8.1.5 Cost Estimates**

In accordance with paragraphs 3.4, the contractor shall deliver Cost Estimates for Galena Airport and Campion AFS and Kalakaket RRS. (A004)

#### **4.8.1.6.1 PA/SI, RI/FS Work Plans**

In accordance with paragraphs 3.4 and 4.0 the Contractor shall deliver an RI/FS Work Plan. The Handbook may be used as guidance. The Contractor shall provide an addendum describing the work being completed during the 1995 field season. (A005)

#### **4.8.1.6.2 Remedial Design Work Plan**

Not applicable.

#### **4.8.1.7 Quality Assurance Project Plans (QAPPs)**

The Contractor shall deliver one QAPP addendum for Galena Airport, Campion AFS, and Kalakaket for all phases of work. The Contractor shall update the QAPP addendum, if applicable, to describe the effort carried out in the upcoming field season at Galena Airport. As a component of the Sampling and Analysis Plan described in Section 4.8.1.9, the Contractor shall

deliver a project/site specific addendum to the QAPP in accordance with paragraph 4.0 of this SOW. The Handbook may be used as guidance. (A007)

**4.8.1.7.1 General QAPP**

Not applicable.

**4.8.1.7.2 RI/FS Project/Site Specific Addendum to QAPP**

Not applicable.

**4.8.1.8 RD Title II Associate Contractor Agreement and Plan Evaluation Report**

Not applicable.

**4.8.1.9 Sampling and Analysis Plan (SAP)**

The Contractor shall deliver and comply with the SAP per paragraph 4.0 of this SOW. The Handbook may be used as guidance. The contractor shall deliver one SAP for Galena Airport, Campion AFS, and Kalakaket Creek, combined. (A007)

**4.8.1.10 Field Sampling Plan (FSP)**

As a component of the SAP described in Section 4.8.1.9 of this SOW, the Contractor shall deliver and comply with a FSP in accordance with Section 4.0 of this SOW. The Handbook may be used as guidance. The FSP shall be considered as an evolving document by which the Contractor provides recommendations and then incorporates Air Force acceptance for field sampling and analysis. The Contractor shall submit an annotated outline of each section of the FSP for approval by the AFCEE COR prior to preparation of the report. The Contractor shall prepare the report as specified in the accepted annotated outline. All sampling and analysis recommendations shall include the Contractor's supporting rationale. Upon Air Force acceptance of sampling and analysis recommendations a phased FSP shall be compiled. The FSP shall include sufficient data to support recommendations and a description of the work to be conducted. FSP shall be updated by site as phase recommendations are accepted by AFCEE. A prime objective shall be to incorporate AFCEE comments in an on-going manner and thereby minimize the volume of comments on the working copy and final submittals. The Contractor shall cite the Base-specific QAPP as a reference document, but completely describe any modifications or additions to the content of these documents. Specific plans shall be developed to conduct sampling as part of remedial actions in accordance with paragraph 4.7.4 of this SOW. The contractor shall deliver two separate FSPs, one for Galena Airport and Campion AFS, and the other for Kalakaket RRS. (A007)

**4.8.1.11 Long Term Groundwater Sampling Plan**

Not applicable.

**4.8.1.12 Test Plans (TPS)**

Not applicable.

**4.8.1.13 Schedules**

**4.8.1.13.1 PA/SI & RI/FS Project Schedule**

In accordance with paragraph 4.0 of this SOW, the Contractor shall deliver a computer generated network analysis which is a detailed task plan for all WBS tasks for approval by the AFCEE COR. The Network Analysis (e.g., GANTT, PERT, CPM) shall be in the form of a progress chart of suitable scale to indicate appropriately the percentage of work scheduled for completion by any given date during the performance period of this SOW. The Network Analysis shall show both serial and parallel sub-tasks leading to a deliverable product/report. Show early and late start and completion date with float. (A013)

**4.8.1.13.2 Remedial Design Project Schedule**

Not applicable.

**4.8.1.13.3 Remedial Action Project Schedule**

Not applicable.

**4.8.2 Primary Documents**

All primary documents shall be prepared and submitted in draft, and final form. Provide microfiche copies of each final primary document at the direction of the AFCEE COR. Draft and final written responses to comments received on draft primary documents shall be provided. The contractor shall deliver advanced drafts to the AFCEE COR for approval. The following primary documents shall be provided:

**4.8.2.1 Technical Reports**

**4.8.2.1.1 Preliminary Assessment/Site Inspection (PA/SI) Report**

In accordance with paragraph 4.2 the contractor shall deliver a report documenting the results of the Preliminary Assessment and/or Site Inspection for Kalakaket RRS. This report shall include the results of the literature search, describing the environmental setting of the base

and identifying potential sources of contamination. The report shall also document the results of all site investigations conducted. (A005)

#### **4.8.2.1.2 Remedial Investigation (RI) Technical Memorandum**

In accordance with paragraph 4.3.1 the Contractor shall update Remedial Investigation Technical Memoranda, provided under a separate cover, in accordance with OSWER 9355.3-01, "Guidance for Conducting Remedial Investigation and Feasibility Studies under CERCLA," October 1988. (A005)

#### **4.8.2.1.3 Feasibility Study (FS) Technical Memorandum**

In accordance with paragraph 4.3.2 a Feasibility Study Technical Memorandum shall be prepared in accordance with OSWER 9355.3-01, "Guidance for Conducting Remedial Investigation and Feasibility Studies under CERCLA," October 1988. The Report shall include the detailed analysis of alternatives and reflect regulatory agency comments to the corresponding Screening of Alternatives Technical Report. The FS Technical Memorandum shall be a separate report from the RI Technical Memoranda. (A005)

#### **4.8.2.3 Decision Documents (DD)**

The contractor shall deliver separate DDs for each site, according to OSWER 9355.3-02. DDs shall be prepared using a format approved by the AFCEE/COR. The contractor shall deliver an Interim Decision Document describing the remedy for the Galena Airport pesticide, petroleum oil and lubricant (POL) stock pile (A005).

#### **4.8.2.4 Engineering Evaluation/Cost Analysis (EE/CA)**

The Contractor shall deliver EE/CAs as part of Action Memorandum Decision Documents. The EE/CA shall evaluate possible alternative technologies for removal actions for remediating the pesticide and POL soil stockpile, the POL area, the fire protection training area, the million gallon hill area, the control tower drum storage area, the southeast spill area, and the base drinking water supply area at Galena Airport. An EE/CA is required by the NCP for any removal action which is determined to be non-time-critical. (A005)

#### **4.8.2.5 Administrative Record Index**

In accordance with paragraph 4.6.6, the Contractor shall update the Administrative Record Index. (A004)

#### **4.8.2.6 Drawings, Plans and Specifications**

#### **4.8.2.6.1 General**

The following lists the drawings, plans and specifications submittal deliverables, the components of these deliverable, and the approximate percentage of the drawings, plans and specifications which shall be completed at each stage. Submittals shall be reviewed by the AFCEE COR and written comments shall be provided. Disposition of the comments shall be determined at the respective review meeting. The contractor shall incorporate the results for each modeling review phase into the next required phase submittal.

##### **4.8.2.6.1.1 Phase I (Preliminary) Drawings/Plans/Specifications**

In accordance with paragraph 4.7.4.1.2 submit at approximately 35% completion of the submittal at which time the technical requirements of the project have been addressed and outlines. (A004, A008)

##### **4.8.2.6.1.2 Phase II (Intermediate) Drawings/Plans/Specifications**

Submitted at approximately 65% completion of the submittal in accordance with paragraph 4.7.4.1.2.2. (A004, A008)

##### **4.8.2.6.1.3 Phase III (final) Drawings/Plans/Specifications**

Submitted at approximately 95% completion of the submittal in accordance with paragraph 4.7.4.1.2.3. (A004, A008)

#### **4.8.2.7 Remedial Design Title II Documents**

Not applicable

#### **4.8.3 Secondary Documents**

Secondary documents are used as input to subsequent primary documents. Draft secondary documents shall be prepared and submitted for review and comment. Following receipt of comments to draft secondary documents, a draft written response to each comment shall be provided for Air Force review. The draft written responses shall be revised based on Air Force input, and final responses shall be provided. The following secondary documents shall be provided:

##### **4.8.3.1 Informal Technical Information Reports (ITIRs)**

**4.8.3.1.1 Analytical Data ITIR**

Submit all analytical data, including QC results and cross reference tables, in a hard and/or electronic copy ITIR. The format in Section 3 of the Handbook may be used as guidance. (A004)

**4.8.3.1.2 Accelerated Remediation Project Definition ITIR**

Not applicable.

**4.8.3.1.3 Conceptual Site Model ITIR**

Not applicable.

**4.8.3.1.4 Site Characterization Summary (SCS—ITIR)**

Not applicable.

**4.8.3.1.5 Ecological and Baseline Risk Assessment ITIR**

The Contractor shall submit in accordance with paragraph 4.6.2. (A004)

**4.8.3.1.6 Remedial Systems and Environmental Equipment ITIR**

Not applicable.

**4.8.3.2 Initial Screening of Alternatives (ISA) Report**

Not applicable.

**4.8.3.3 Detailed Analyses of Alternatives (DAA) Report**

Not applicable.

**4.8.3.4 Installation Restoration Program Information Management System (IRPIMS)**

**Data Management**—The Contractor shall meet the data deliverable requirements of the Installation Restoration Program Information Management System (IRPIMS). The Contractor shall be responsible for recording field and laboratory data into a computerized format as required by the most current version of the IRPIMS Data Loading Handbook (mailed under separate cover). In order to perform this task, the Contractor shall use the latest version of the IRPIMS Quality Control Tool (QC Tool), a PC software utility (mailed under separate cover

with software manual), to quality check ASCII data files and to check all data files for compliance with requirements in the IRPIMS Data Loading Handbook. Upon request, the IRPIMS Contractor Data Loading Tool (CDLT) is available. This PC software is designed to assist the Contractor in preparing the various ASCII data files.

Individual IRPIMS data files (e. g. analytical results, groundwater level data, etc.), including resubmissions, shall be delivered with a transmittal letter by the Contractor to the Air Force Center for Environmental Excellence (AFCEE) IN SEQUENCE according to a controlled time schedule as identified in the current version of the IRPIMS Data Loading Handbook. The Contractor shall include a copy of the Quality Control Tool error report, i.e. output from the QC tool, for each IRPIMS file submission. The error report shall be submitted as hard copy with the transmittal letter.

All Contractor IRPIMS data deliverables shall be sent to:

AFCEE/MSC  
ENVIRONMENTAL DATA MANAGEMENT DIVISION  
ATTN: IRPIMS Data Management  
8106 Chenault Rd (BLDG 1161)  
Brooks AFB TX 78235-5318

In addition, the Contractor shall provide a copy of the transmittal letter to the CO, HSC/PKV (8005 9th St, Brooks AFB, TX 78235-5353). This letter shall identify the files included or otherwise omitted (with an appropriate explanation), the government contract and delivery order number and the Air Force point of contact that is responsible for monitoring the government contract.

The Contractor shall be responsible for the accuracy and completeness of all data submitted. All data entered into the IRPIMS data files and submitted by the Contractor shall correspond exactly with the data contained in the original laboratory reports and other documents associated with sampling and laboratory contractual tasks.

Each file delivered by the Contractor will be electronically evaluated by AFCEE/MSC for format compliance and data integrity in order to verify acceptance. All files delivered by the Contractor are required to be ERROR-FREE and in compliance with the IRPIMS Data Loading Handbook. Any errors identified by AFCEE/MSC in the submission shall be corrected by the Contractor.

#### **4.8.3.5 Letter Reports**

##### **4.8.3.5.1 General**

The Contractor shall deliver letter reports. The purpose of the letter reports is to provide data and the Contractors' evaluation of the data to enable the AFCEE COR and Base

POC to be involved in the decisions based on that data. The letter report shall briefly describe the task performed, the Contractor's evaluation of the data collected, and recommendations for subsequent tasks. All data collected as part of this task shall be provided as an attachment to the letter report. (A004)

#### **4.8.3.5.2 Health Risk**

In accordance with paragraph 3.1.1, the Contractor shall deliver letter reports concerning imminent health risks encountered (A015)

#### **4.8.3.6 Environmental Report**

The Contractor shall deliver reports, photographs, data, drawings, designs, documentation as required by each DO, documenting the results of various environmental investigations, studies, assessments, designs, and/or analyses conducted under section 4.7 above. (A004, A005, A008, A009, A011)

#### **4.8.3.7 Presentation Materials**

The Contractor shall prepare and present briefing packages at meetings coordinated by the Air Force. As part of the presentation materials, the Contractor shall deliver electronic and paper copies of all slides, analytical data Graphical Interface System material, and overheads as specified in each DO. (A010)

#### **4.8.3.8 Photo Documentation**

The Contractor shall prepare and deliver a Photo Notebook with descriptive captions at Kalakaket RRS. Include photos of sites under investigation, field activities and sample locations. (A011)

#### **4.8.3.9 Community Relations Newsletters/Fact Sheets**

The Contractor shall submit Newsletters and Fact sheets in accordance with paragraphs 4.8.1.4.3 and 4.8.1.4.4

#### **4.8.3.10 Meeting Minutes**

The Contractor shall be responsible for generating meeting minutes, documenting all items discussed at the meetings and shall include a list of meeting attendees. (A012)

**4.8.3.11 Contractor Personnel Chart**

Per paragraph 3.1.2 the Contractor shall deliver Contractor personnel charts to the AFCEE COR. (A003)

**4.8.3.12 Treatability Study Technical Report**

The Contractor shall finalize the draft report, provided under a separate cover. (A004)

**4.8.3.13 Aquifer Test Technical Report**

The Contractor shall incorporate all AFCEE and 611 CES/CEVR comments and finalize the Draft Aquifer Test Technical Report, provided under a separate cover. (A004)

## **V. DATA**

### **5.0 DATA MANAGEMENT**

The Contractor shall collect, prepare, publish, and distribute the data in the quantities and types designated on the Contract Data Requirements List (CDRL). The Contractor shall designate a focal point who shall integrate the total data management effort and manage changes, additions or deletions of data items. In addition, the Contractor shall identify items to be added, recommend revisions or deletion of items already listed on the CDRL as appropriate and maintain the status of all data deliverables.

#### **5.1 Data Deliverables**

Deliverables shall be submitted in accordance with the attached CDRLs.

**APPENDIX B**

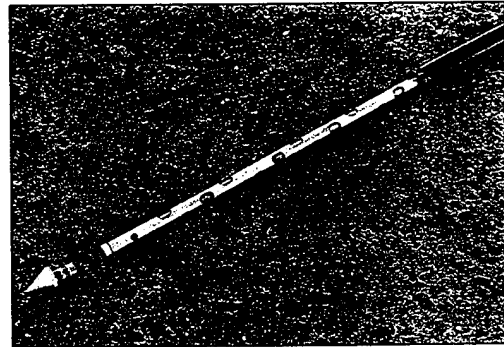
**Standard Operating Procedures for DPT Field Screening**

## Appendix C:

# Screen Point Ground Water Sampler Operation

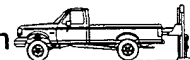
Appendix C

C



After the Screen Point Ground Water Sampler is driven to depth, the rods are retracted and the screen insert is pushed out into the formation.

The Tools for Site Investigation



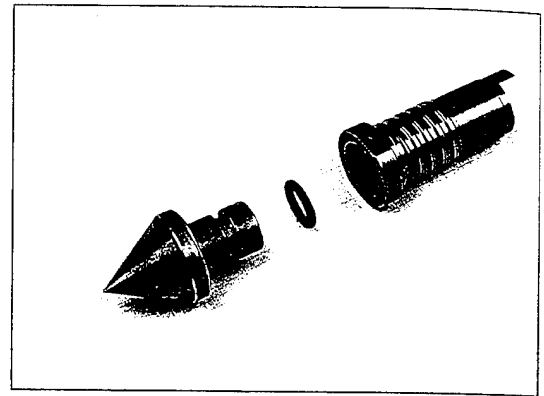
C.1

# Screen Point Ground Water Sampler – Operation

## Assembly

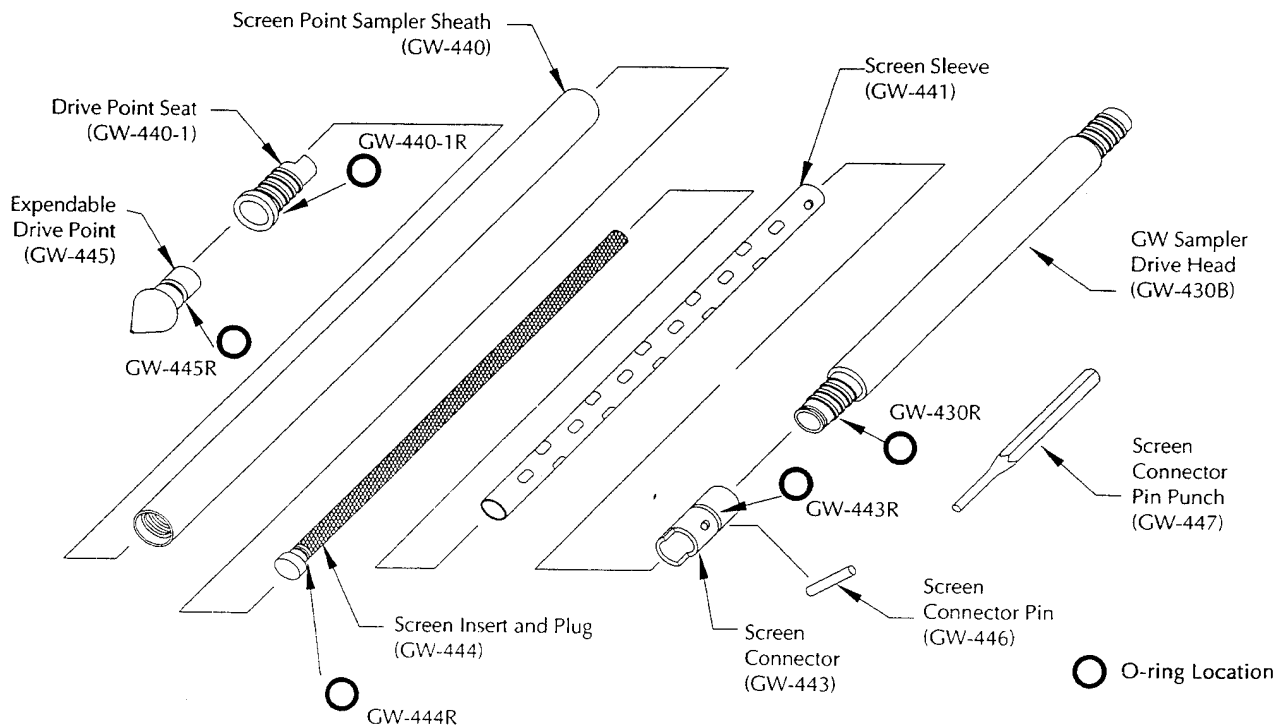
Clean all parts thoroughly before assembly. An uncontaminated screen insert should be used for each new sample. It is recommended that new O-rings be installed at each O-ring location prior to each sample. O-ring numbers correspond to the individual part numbers. After O-rings have been installed, follow these steps:

1. Push the Screen Insert and Plug into the Screen Sleeve from the bottom. The bottom end has one drain hole (Figure 1).
2. Push the Screen Connector over the top end of the Screen Sleeve and push the Screen Connector Pin into place (Figure 2). It has a loose fit so use your thumb and forefinger to hold it in place.
3. Insert the Screen Sleeve, Screen Connector first, halfway into the Sampler Sheath (either end is okay) (Figure 3).
4. Slide the Drive Point Seat over the end of the screen assembly that protrudes from the Sampler Sheath (Figure 4). Thread it in until tight using a 7/8-in. wrench.
5. Push the screen assembly just far enough into the Sampler Sheath that an Expendable Drive Point (GW-445) can be pushed into place in the Drive Seat (Figure 5).
6. Screw the Drive Head with the O-ring end first into the open end of the Sampler Sheath (Figure 6).



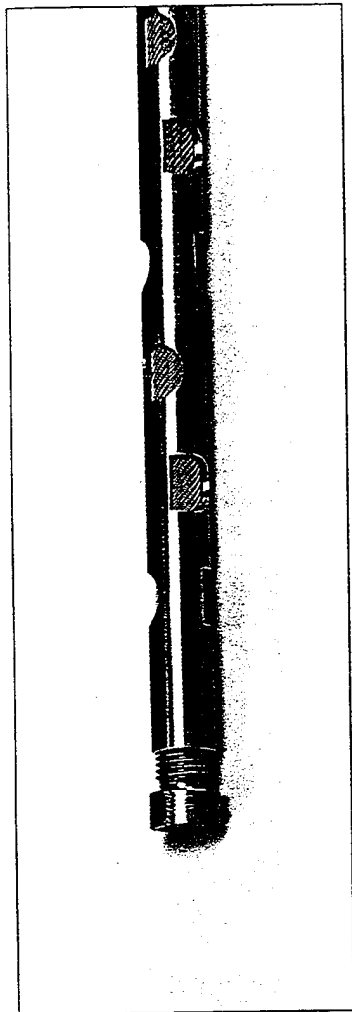
New O-rings should be used at each O-ring location prior to each sample. Shown here is the Expendable Drive Point (GW-445), O-ring (GW-445R), and Drive Point Seat (GW-440-1).

**NOTE:** These parts must be assembled so as to allow free movement of the screen assembly inside the Sampler Sheath. There should be no internal binding. The assembled sampler is now ready to be driven into the subsurface. Wetting the O-rings with a small amount of distilled water will aid in free movement of the parts.



▲ Screen Point Sampler (GW-440K)

# Screen Point Ground Water Sampler – Operation



Wire mesh stainless steel Screen Insert inside stainless steel Screen Sleeve. Screen Insert has 0.145-mm pore openings which filter out sediment.

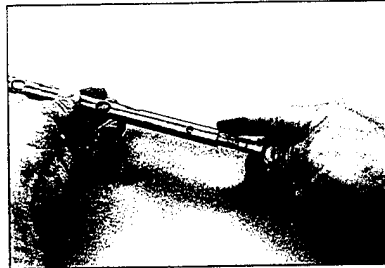


Figure 1. Push the Screen Insert and Plug into Screen Sleeve.

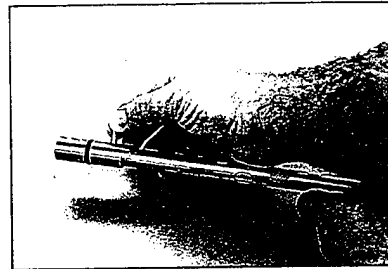


Figure 2. Push the Screen Connector Pin into place.

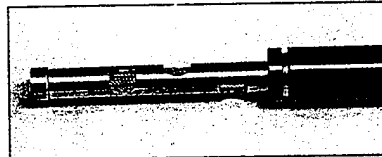


Figure 3. Insert Screen Sleeve halfway into Screen Point Sampler Sheath.

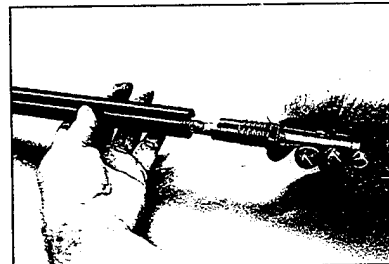


Figure 4. Slide Drive Point Seat over end of Screen Sleeve and screw into Sampler Sheath.

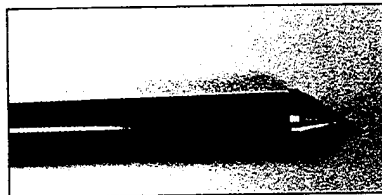


Figure 5. Insert Expendable Drive Point into Drive Point Seat.

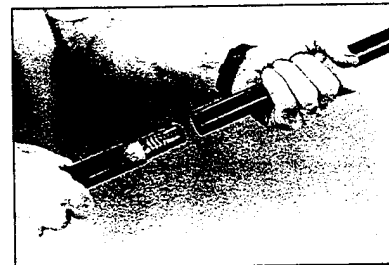
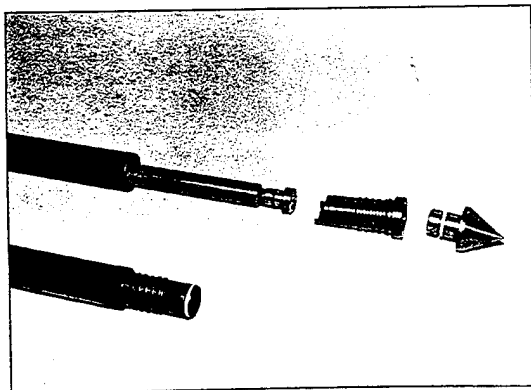


Figure 6. Screw O-ring end of Drive Head into top of Sampler Sheath.



Partially assembled Screen Point Sampler (GW-440K).

# Screen Point Ground Water Sampler – Operation

## Probing

Place a drive cap on the assembled sampler and drive it into the subsurface (Figure 1). Continue driving by adding Geoprobe probe rods until the sampler tip has been driven about one foot (1225 mm) below the target sampling depth (Figure 2). Once that depth has been reached, disengage the expendable drive point by replacing the drive cap with a pull cap and pulling the rods back a distance of about 2 ft. (1 m) (Figure 3).

## Exposing the Screen

In stable formations, the screen assembly may be pushed out into the open borehole by lowering 3/8-in. tubing affixed with a PRT Adapter (TB-25L and PR-25S) to the top end of the screen assembly (Figure 4). The threads on the PRT adapter are engaged with the threads on the Screen Connector by pushing gently downward on the tubing and rotating it counterclockwise. When properly connected, the screen assembly can be pushed out of the Sampler Sheath by pushing down on the tubing. A water sample can be drawn through the tubing.

In unstable formations, the screen assembly may have to be pushed out of the Sampler Sheath by means of extension rods coupled together and inserted down the inside of the probe rods (Figure 5). The leading end of the extension rods should be equipped with an extension rod coupler to protect the threads on the Screen Connector. A steady push is sufficient. Excessive hammering on the rods should be avoided. After pushing the screen into the formation, the extension rods need to be removed in order to begin sampling.

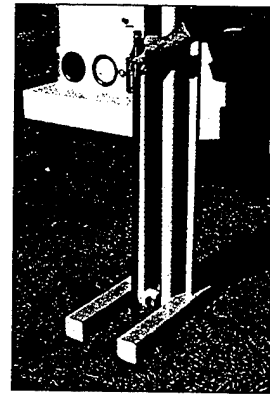


Figure 1. Assembled Screen Point Sampler is driven to depth.

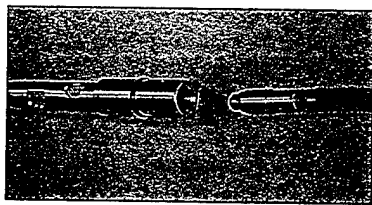


Figure 4. PRT-Adapter (PR-25S) inserted in tubing (TB-25L) prior to connection down-hole with Screen Connector.

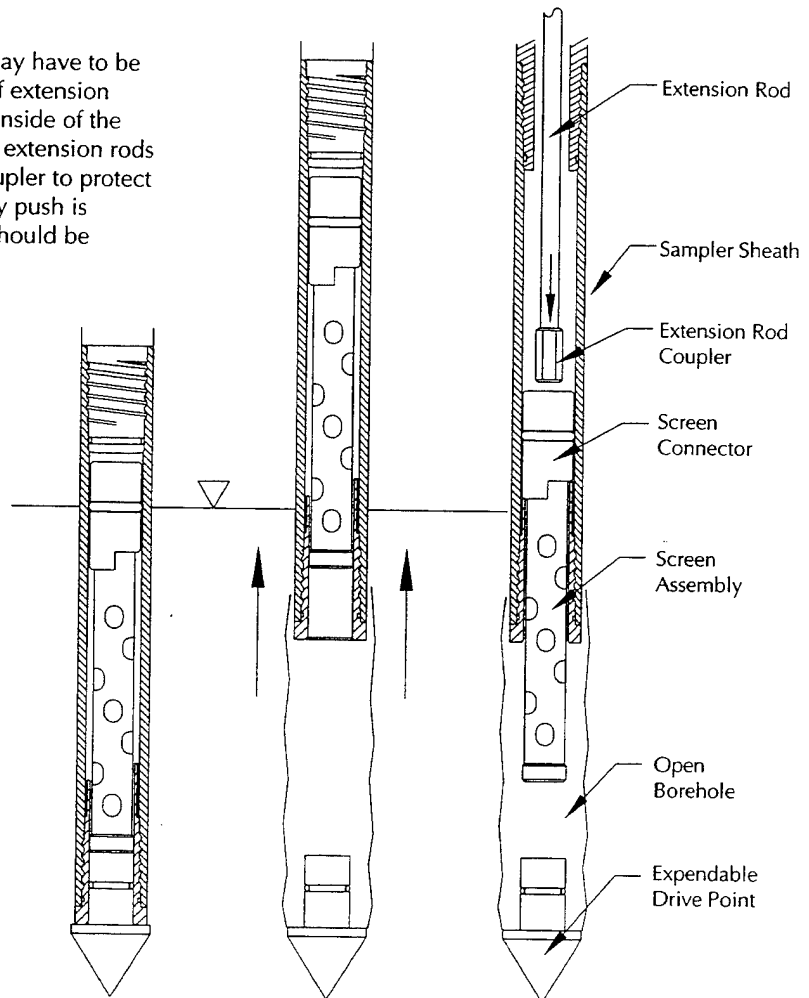


Figure 2. Sample Depth is Reached.

Figure 3. Pull Back Sampler 2 ft. (1 m).

Figure 5. Push Out Screen Assembly with Extension Rods.

# Screen Point Ground Water Sampler – Operation

## Sampling

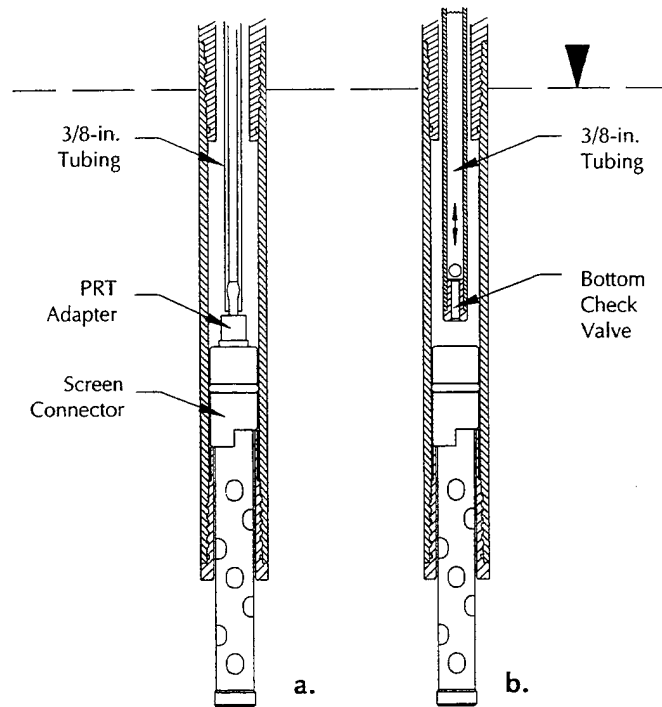
Water sampling may be accomplished by using 3/8-inch tubing and a stainless steel PRT adapter as previously described (TB-25L and PR-25S). Once the PRT adapter has made connection with the Screen Connector, a vacuum may be applied to the top of the tubing (Figure 6a). This may be done with a peristaltic pump (Figure 7) or by using a vacuum pump with an in-line trap.

If the PRT system is not used, the same tubing equipped with a Tubing Bottom Check Valve (GW-42) may be used (Figures 8 and 9). The tubing is oscillated up and down and the water sample is pushed upward into the tubing as the ball repeatedly lifts and seats (Figure 6b). The tubing will begin to feel heavier as it fills with several feet of water. It can then be lifted out of the probe rods, cut, and the water poured into a vial for analysis. This same tubing/check valve arrangement has been used to pump multi-liter samples from the probe rod.

## Removal

When the sampling procedure is finished, the probe rods and sampler may be extracted. If the PRT system is used, remove the tubing by pulling up firmly on it until it disconnects from the PRT adapter down-hole. The PRT adapter will remain attached to the Screen Connector.

After the sampler has been recovered, examine all parts for wear, damage, or contamination. Thoroughly clean all parts, replace all O-rings, and prepare for the next sample.



Figures 6a and 6b. Sampling options.

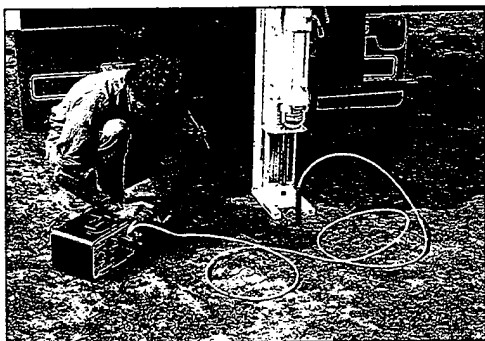


Figure 7. Using a peristaltic pump to collect a groundwater sample using the Screen Point Sampler.



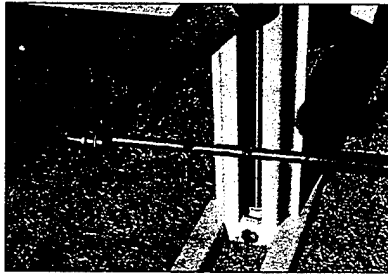
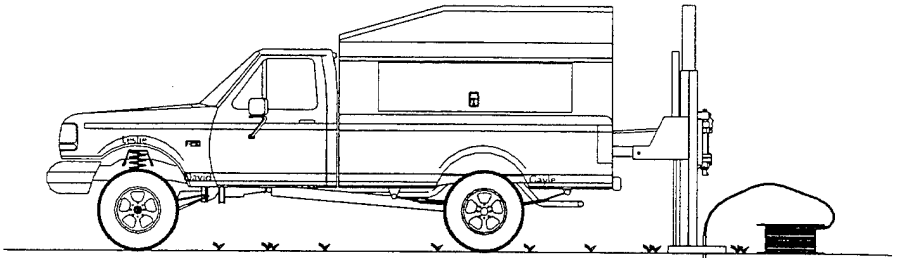
Figure 8. Tubing Bottom Check Valve and Check Ball are installed onto Tubing . . .



Figure 9. . . . and are then fed through the diameter of the probe rods to retrieve the water sample.



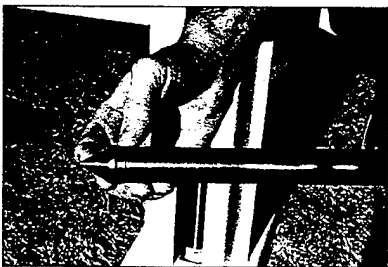
# Screen Point Ground Water Sampler – Operation



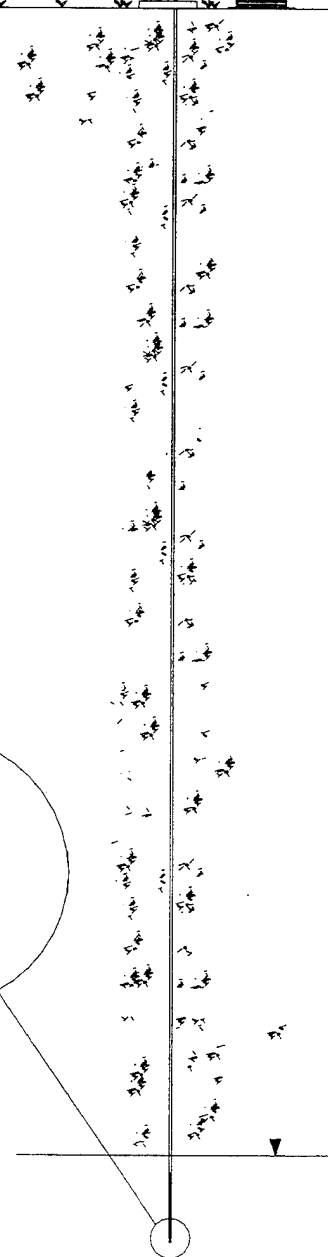
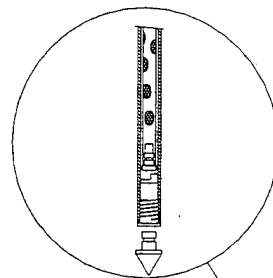
An Expendable Drive Point, Screen Insert, and Screen Sleeve are inserted into Sampler Sheath . . .



. . . the Screen Insert and Sleeve are inserted just far enough for the Expendable Drive Point to be inserted . . .



. . . and the assembled Screen Point Sampler is ready to be driven to depth.



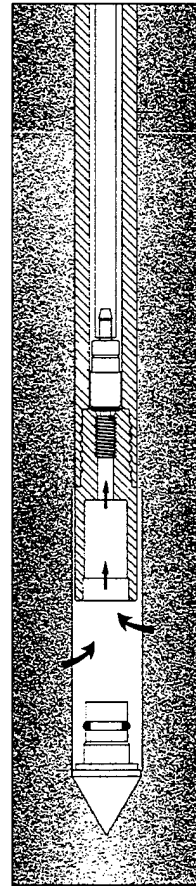
Screen Point Sampler at depth using Tubing Bottom Check Valve system for retrieving groundwater sample.



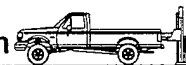
# Appendix D: Soil Gas Sampling — PRT System Operation

Appendix D

**D**



Soil Gas Sampling using the Post-Run Tubing (PRT) System.



# Soil Gas Sampling — PRT System Operation

## Basics

Using the Post-Run Tubing System, one can drive probe rods to the desired sampling depth, then insert and seal an internal tubing for soil gas sampling. The usual Geoprobe probe rods and driving accessories and the following tools are required:

- PRT Expendable Point Holder
- PRT Adapter
- Selected PRT Tubing

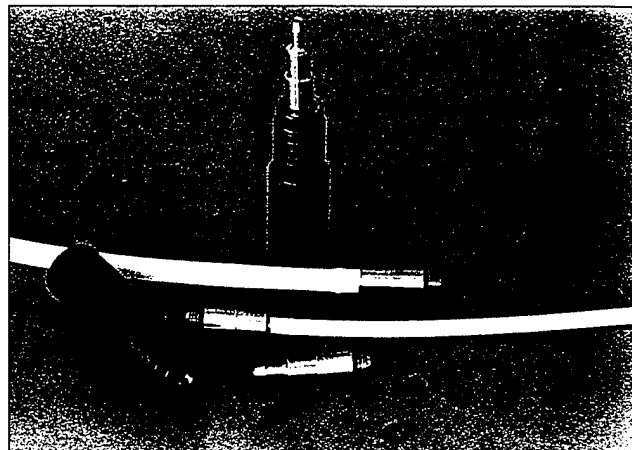
## Preparation

1. Clean all parts prior to use. Install O-rings on the PR-13B and the PRT adapter.
2. Inspect the probe rods and clear them of all obstructions.
3. TEST FIT the adapter with the PRT fitting on the expendable point holder to assure that the threads are compatible and fit together smoothly.

NOTE: PRT fittings are left-hand threaded.

4. Push the adapter into the end of the selected tubing. Tape may be used on the outside of the adapter and tubing to prevent the tubing from spinning freely around the adapter during connection – especially when using Teflon tubing (Figure 1).

REMEMBER: The sample will not contact the outside of the tubing or adapter.



PRT SYSTEM PARTS  
PRT Expendable Point Holder, PRT Adapters, Tubing, and O-rings.

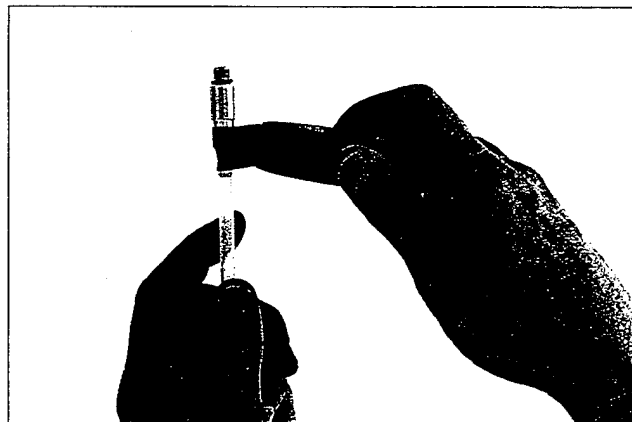


Figure 1. Securing adapter to tubing with tape. NOTE: Tape does not contact soil gas sample.

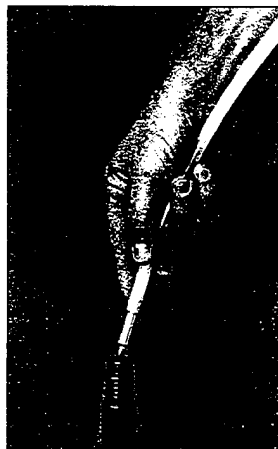
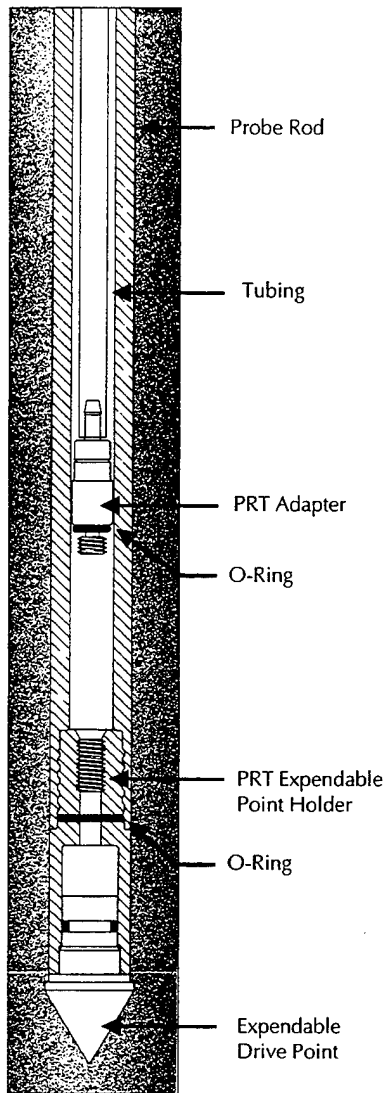


Figure 2. Insertion of tubing and PRT adapter.



Figure 3. Engaging threads by rotating tubing.

# Soil Gas Sampling — PRT System Operation



A cross section of probe rods driven to depth and then retracted to allow for soil gas sampling. The PRT adapter and tubing are now fed through the rods and rotated to form a vacuum-tight connection at the point holder. The result is a continuous run of tubing from the sample level to the surface.

## Probing

Drive the PRT tip configuration into the ground. Connect probe rods as necessary to reach the desired depth. After depth has been reached, disengage the expendable point by pulling up on the probe rods. Remove the pull cap from the top probe rod, and position the Geoprobe unit to allow room to work.

## Connection

1. Insert the adapter end of the tubing down the inside diameter of the probe rods (Figure 2).
2. Feed the tubing down the rod bore until it hits bottom on the expendable point holder. Allow about 2 ft. (610 mm) of tubing to extend out of the hole before cutting it.
3. Grasp the excess tubing and apply some downward pressure while turning it in a counterclockwise motion to engage the adapter threads with the expendable point holder (Figure 3).
4. Pull up lightly on the tubing to test engagement of the threads. (Failure of adapter to thread could mean that intrusion of soil may have occurred during driving of probe rods or disengagement of drive point.)



# Soil Gas Sampling — PRT System Operation

## Sampling

1. Connect the outer end of the tubing to the Silicone Tubing Adapter and vacuum hose (or other sampling apparatus).
2. Follow the appropriate sampling procedure for collecting a soil gas sample (Figure 1).

## Removal

1. After collecting a sample, disconnect the tubing from the vacuum hose or sampling system.
2. Pull up firmly on the tubing until it releases from the adapter at the bottom of the hole. (Taped tubing requires a stronger pull.)
3. Remove the tubing from the probe rods. Dispose of polyethylene tubing or decontaminate Teflon tubing as protocol dictates.
4. Retrieve the probe rods from the ground and recover the expendable point holder with the attached PRT adapter.
5. Inspect the O-ring at the base of the PRT adapter to verify that proper sealing was achieved during sampling. The O-ring should be compressed. This seal can be tested by capping the open end of the point holder applying vacuum to the PRT adapter.
6. Prepare for the next sample.



Figure 1. Taking a soil gas sample for direct injection into a GC with the PRT system.



**APPENDIX C**  
**Analytical Methods**

**METHOD 524.2. MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN  
WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY**

**Revision 3.0**

A. Alford-Stevens, J. W. Eichelberger, W. L. Budde - Method 524, Revision 1.0  
(1983)

R. W. Slater, Jr. - Method 524.2, Revision 2.0 (1986)

J. W. Eichelberger, W. L. Budde - Method 524.2, Revision 3.0 (1989)

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## METHOD 524.2

### MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY

#### 1. SCOPE AND APPLICATION

1.1 This is a general purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage (1-2). The method is applicable to a wide range of organic compounds, including the four trihalomethane disinfection by-products, that have sufficiently high volatility and low water solubility to be efficiently removed from water samples with purge and trap procedures. The following compounds can be determined by this method.

<u>Compound</u>	<u>Chemical Abstract Service Registry Number</u>
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-4
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9

2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
Isopropylbenzene	98-82-8
4-Isopropyltoluene	99-87-6
Methylene chloride	75-09-2
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

- 1.2 Method detection limits (MDLs) (3) are compound and instrument dependent and vary from approximately 0.02-0.35  $\mu\text{g/L}$ . The applicable concentration range of this method is primarily column dependent and is approximately 0.02 to 200  $\mu\text{g/L}$  for the wide-bore thick-film columns. Narrow-bore thin-film columns may have a capacity which limits the range to about 0.02 to 20  $\mu\text{g/L}$ . Analytes that are inefficiently purged from water will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present in sufficient amounts.
- 1.3 Analytes that are not separated chromatographically, but which have different mass spectra and non-interfering quantitation ions, can be identified and measured in the same calibration mixture or water sample (Sect 11.6.2). Analytes which have very similar mass spectra cannot be individually identified and measured in the same calibration mixture or water sample unless they have different retention times (Sect.11.6.3). Coeluting compounds with very similar mass spectra, typically many structural isomers, must be reported as an isomeric group or pair. Two of the three isomeric xylenes and two of the three dichlorobenzenes are examples of structural isomers that may not be resolved on the capillary column, and if not, must be reported as isomeric pairs.

## 2. SUMMARY OF METHOD

2.1 Volatile organic compounds and surrogates with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through the aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb the trapped sample components into a capillary gas chromatography (GC) column interfaced to a mass spectrometer (MS). The column is temperature programmed to separate the method analytes which are then detected with the MS. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a data base. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure.

## 3. DEFINITIONS

- 3.1 Internal standard -- A pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.
- 3.2 Surrogate analyte -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of a surrogate analyte is to monitor method performance with each sample.
- 3.3 Laboratory duplicates (LD1 and LD2) -- Two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 3.4 Field duplicates (FD1 and FD2) -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.5 Laboratory reagent blank (LRB) -- An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method

analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

- 3.6 Field reagent blank (FRB) -- Reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.
- 3.7 Laboratory performance check solution (LPC) -- A solution of one or more compounds (analytes, surrogates, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 3.8 Laboratory fortified blank (LFB) -- An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit.
- 3.9 Laboratory fortified sample matrix (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 3.10 Stock standard solution -- A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assayed reference compound. Stock standard solutions are used to prepare primary dilution standards.
- 3.11 Primary dilution standard solution -- A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 3.12 Calibration standard (CAL) -- a solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.13 Quality control sample (QCS) -- a sample matrix containing method analytes or a solution of method analytes in a water miscible solvent which is used to fortify reagent water or environmental samples. The QCS is obtained from a source external to the laboratory, and is used

to check laboratory performance with externally prepared test materials.

#### 4. INTERFERENCES

- 4.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) plastic tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in laboratory reagent blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.
- 4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross contamination.
- 4.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

#### 5. SAFETY

- 5.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available (4-6) for the information of the analyst.
- 5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloro-

form, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

## 6. APPARATUS AND EQUIPMENT

6.1 SAMPLE CONTAINERS -- 60-mL to 120-mL screw cap vials (Pierce #19832 or equivalent) each equipped with a PTFE-faced silicone septum (Pierce #12718 or equivalent). Prior to use, wash vials and septa with detergent and rinse with tap and distilled water. Allow the vials and septa to air dry at room temperature, place in a 105°C oven for 1 hr, then remove and allow to cool in an area known to be free of organics.

6.2 PURGE AND TRAP SYSTEM -- The purge and trap system consists of three separate pieces of equipment: purging device, trap, and desorber. Systems are commercially available from several sources that meet all of the following specifications.

6.2.1 The all glass purging device (Figure 1) should be designed to accept 25-mL samples with a water column at least 5 cm deep. A smaller (5-mL) purging device is recommended if the GC/MS system has adequate sensitivity to obtain the method detection limits required. Gaseous volumes above the sample must be kept to a minimum (< 15 mL) to eliminate dead volume effects. A glass frit should be installed at the base of the sample chamber so the purge gas passes through the water column as finely divided bubbles with a diameter of < 3 mm at the origin. Needle spargers may be used, however, the purge gas must be introduced at a point about 5 mm from the base of the water column.

6.2.2 The trap (Figure 2) must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap should contain 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. If it is not necessary to determine dichlorodifluoromethane, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

6.2.3 The use of the methyl silicone coated packing is recommended, but not mandatory. The packing serves a dual purpose of protecting the Tenax adsorbant from aerosols, and also of

insuring that the Tenax is fully enclosed within the heated zone of the trap thus eliminating potential cold spots. Alternatively, silanized glass wool may be used as a spacer at the trap inlet.

- 6.2.4 The desorber (Figure 2) must be capable of rapidly heating the trap to 180°C either prior to or at the beginning of the flow of desorption gas. The polymer section of the trap should not be heated higher than 200°C or the life expectancy of the trap will decrease. Trap failure is characterized by a pressure drop in excess of 3 pounds per square inch across the trap during purging or by poor bromoform sensitivities. The desorber design illustrated in Fig. 2 meets these criteria.

### 6.3 GAS CHROMATOGRAPHY/MASS SPECTROMETER/DATA SYSTEM (GC/MS/DS)

- 6.3.1 The GC must be capable of temperature programming and should be equipped with variable-constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation. The column oven must be cooled to 10°C; therefore, a subambient oven controller is required. If syringe injections of BFB will be used, a split/splitless injection port is required.

- 6.3.2 Capillary Gas Chromatography Columns. Any gas chromatography column that meets the performance specifications of this method may be used. Separations of the calibration mixture must be equivalent or better than those described in this method. Three useful columns have been identified.

6.3.2.1 Column 1 -- 60 m x 0.75 mm ID VOCOL (Supelco, Inc.) glass wide-bore capillary with a 1.5 μm film thickness.

Column 2 -- 30 m x 0.53 mm ID DB-624 (J&W Scientific, Inc.) fused silica capillary with a 3 μm film thickness.

Column 3 -- 30 m x 0.32 mm ID DB-5 (J&W Scientific, Inc.) fused silica capillary with a 1 μm film thickness.

- 6.3.3 Interfaces between the GC and MS. The interface used depends on the column selected and the gas flow rate.

- 6.3.3.1 The wide-bore columns 1 and 2 have the capacity to accept the standard gas flows from the trap during thermal desorption, and chromatography can begin with the onset of thermal desorption. Depending on the pumping capacity of the MS, an additional interface between the end of the column and the MS may be required. An open split interface (7), an all-glass jet separator, or a cryogenic (Sect. 6.3.3.2) device

are acceptable interfaces. Any interface can be used if the performance specifications described in this method can be achieved. The end of the transfer line after the interface, or the end of the analytical column if no interface is used, should be placed within a few mm of the MS ion source.

- 6.3.3.2 The narrow bore column 3 cannot accept the thermal desorption gas flow, and a cryogenic interface is required. This interface (Tekmar Model 1000 or equivalent) condenses the desorbed sample components at liquid nitrogen temperature, and allows the helium gas to pass through to an exit. The condensed components are frozen in a narrow band on an uncoated fused silica precolumn. When all components have been desorbed from the trap, the interface is rapidly heated under a stream of carrier gas to transfer the analytes to the analytical column. The end of the analytical column should be placed with a few mm of the MS ion source. A potential problem with this interface is blockage of the interface by frozen water from the trap. This condition will result in a major loss in sensitivity and chromatographic resolution.
- 6.3.4 The mass spectrometer must be capable of electron ionization at a nominal electron energy of 70 eV. The spectrometer must be capable of scanning from 35 to 260 amu with a complete scan cycle time (including scan overhead) of 2 sec or less. (Scan cycle time = Total MS data acquisition time in seconds divided by number of scans in the chromatogram). The spectrometer must produce a mass spectrum that meets all criteria in Table 3 when 25 ng or less of 4-bromofluorobenzene (BFB) is introduced into the GC. An average spectrum across the BFB GC peak may be used to test instrument performance.
- 6.3.5 An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectra from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software must allow integration of the ion abundance of any specific ion between specified time or scan number limits. The software should also allow calculation of response factors as defined in Sect. 9.2.6 (or construction of a second or third order regression calibration curve), calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the equation in Sect. 12.

#### 6.4 SYRINGE AND SYRINGE VALVES

- 6.4.1 Two 5-mL or 25-mL glass hypodermic syringes with Luer-Lok tip (depending on sample volume used).
- 6.4.2 Three 2-way syringe valves with Luer ends.
- 6.4.3 One 25- $\mu$ L micro syringe with a 2 in x 0.006 in ID, 22<sup>o</sup> bevel needle (Hamilton #702N or equivalent).
- 6.4.4 Micro syringes - 10, 100  $\mu$ L.
- 6.4.5 Syringes - 0.5, 1.0, and 5-mL, gas tight with shut-off valve.

#### 6.5 MISCELLANEOUS

- 6.5.1 Standard solution storage containers -- 15-mL bottles with PTFE-lined screw caps.

### 7. REAGENTS AND CONSUMABLE MATERIALS

#### 7.1 TRAP PACKING MATERIALS

- 7.1.1 2,6-Diphenylene oxide polymer, 60/80 mesh, chromatographic grade (Tenax GC or equivalent).
- 7.1.2 Methyl silicone packing (optional) -- OV-1 (3%) on Chromosorb W, 60/80 mesh, or equivalent.
- 7.1.3 Silica gel -- 35/60 mesh, Davison, grade 15 or equivalent.
- 7.1.4 Coconut charcoal -- Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through 26 mesh screen.

#### 7.2 REAGENTS

- 7.2.1 Methanol -- Demonstrated to be free of analytes.
- 7.2.2 Reagent water -- Prepare reagent water by passing tap water through a filter bed containing about 0.5 kg of activated carbon, by using a water purification system, or by boiling distilled water for 15 min followed by a 1-h purge with inert gas while the water temperature is held at 90<sup>o</sup>C. Store in clean, narrow-mouth bottles with PTFE-lined septa and screw caps.
- 7.2.3 Hydrochloric acid (1+1) -- Carefully add measured volume of conc. HCl to equal volume of reagent water.
- 7.2.4 Vinyl chloride -- Certified mixtures of vinyl chloride in nitrogen and pure vinyl chloride are available from several

sources (for example, Matheson, Ideal Gas Products, and Scott Gases).

7.2.5 Ascorbic acid -- ACS reagent grade, granular.

7.3 STOCK STANDARD SOLUTIONS -- These solutions may be purchased as certified solutions or prepared from pure standard materials using the following procedures. One of these solutions is required for every analyte of concern, every surrogate, and the internal standard. A useful working concentration is about 1-5 mg/mL.

7.3.1 Place about 9.8 mL of methanol into a 10-mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg.

7.3.2 If the analyte is a liquid at room temperature, use a 100- $\mu$ L syringe and immediately add two or more drops of reference standard to the flask. Be sure that the reference standard falls directly into the alcohol without contacting the neck of the flask. If the analyte is a gas at room temperature, fill a 5-mL valved gas-tight syringe with the standard to the 5.0-mL mark, lower the needle to 5 mm above the methanol meniscus, and slowly inject the standard into the neck area of the flask. The gas will rapidly dissolve in the methanol.

7.3.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in  $\mu$ g/ $\mu$ L from the net gain in weight. When compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

7.3.4 Store stock standard solutions in 15-mL bottles equipped with PTFE-lined screw caps. Methanol solutions prepared from liquid analytes are stable for at least 4 weeks when stored at 4°C. Methanol solutions prepared from gaseous analytes are not stable for more than 1 week when stored at <0°C; at room temperature, they must be discarded after 1 day.

7.4 PRIMARY DILUTION STANDARDS -- Use stock standard solutions to prepare primary dilution standard solutions that contain all the analytes of concern and the surrogates (but not the internal standard!) in methanol. The primary dilution standards should be prepared at concentrations that can be easily diluted to prepare aqueous calibration solutions that will bracket the working concentration range. Store the primary dilution standard solutions with minimal headspace and check frequently for signs of deterioration or evaporation, especially just before preparing calibration solutions. Storage times described for stock standard solutions in Sect. 7.4.4 also apply to primary dilution standard solutions.

## 7.5 FORTIFICATION SOLUTIONS FOR INTERNAL STANDARD AND SURROGATES

7.5.1 A solution containing the internal standard and the surrogates is required to prepare laboratory reagent blanks (also used as a laboratory performance check solution), and to fortify each sample. Prepare a fortification solution containing fluoro-benzene (internal standard), 1,2-dichlorobenzene- $d_4$  (surrogate), and BFB (surrogate) in methanol at concentrations of 5  $\mu\text{g}/\text{mL}$  of each. A 5- $\mu\text{L}$  aliquot of this solution added to a 25-mL water sample volume gives concentrations of 1  $\mu\text{g}/\text{L}$  of each. A 5- $\mu\text{L}$  aliquot of this solution added to a 5-mL water sample volume gives a concentration of 5  $\mu\text{g}/\text{L}$  of each). Additional internal standards and surrogate analytes are optional.

7.5.2 A solution of the internal standard alone is required to prepare calibration standards and laboratory fortified blanks. The internal standard should be in methanol at a concentration of 5  $\mu\text{g}/\text{mL}$ .

7.6 PREPARATION OF LABORATORY REAGENT BLANK -- Fill a 25-mL (or 5-mL) syringe with reagent water and adjust to the mark (no air bubbles). Inject 10  $\mu\text{L}$  of the fortification solution containing the internal standard and surrogates through the Luer Lok valve into the reagent water. Transfer the LRB to the purging device. See Sect. 11.1.2.

7.7 PREPARATION OF LABORATORY FORTIFIED BLANK -- Prepare this exactly like a calibration standard (Sect. 7.8). This is a calibration standard that is treated as a sample.

## 7.8 PREPARATION OF CALIBRATION STANDARDS

7.8.1 The number of calibration solutions (CALs) needed depends on the calibration range desired. A minimum of three CAL solutions is required to calibrate a range of a factor of 20 in concentration. For a factor of 50, use at least four standards, and for a factor of 100 at least five standards. One calibration standard should contain each analyte of concern and each surrogate at a concentration of 2-10 times the method detection limit (Tables 4-6) for that compound. The other CAL standards should contain each analyte of concern and each surrogate at concentrations that define the range of the method. Every CAL solution contains the internal standard at the same concentration (5  $\mu\text{g}/\text{L}$  suggested for a 5-mL sample; 1  $\mu\text{g}/\text{L}$  for a 25-mL sample).

7.8.2 To prepare a calibration standard, add an appropriate volume of a primary dilution standard (containing analytes and surrogates) to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the methanol solutions into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the

flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable in a volumetric flask and should be discarded after 1 hr unless transferred to a sample bottle and sealed immediately.

## 8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

### 8.1 SAMPLE COLLECTION, DECHLORINATION, AND PRESERVATION

8.1.1 Collect all samples in duplicate. If samples contain residual chlorine, and measurements of the concentrations of disinfection by-products (trihalomethanes, etc.) at the time of sample collection are desired, add about 25 mg of ascorbic acid to the sample bottle before filling. Fill sample bottles to overflowing, but take care not to flush out the rapidly dissolving ascorbic acid. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. Adjust the pH of the duplicate samples to <2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume. Seal the sample bottles, PFTE-face down, and shake vigorously for 1 min.

8.1.2 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 mL/min and collect duplicate samples from the flowing stream.

8.1.3 When sampling from an open body of water, fill a 1-quart wide-mouth bottle or 1-liter beaker with sample from a representative area, and carefully fill duplicate sample bottles from the 1-quart container.

8.1.4 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

### 8.2 SAMPLE STORAGE

8.2.1 Store samples at 4°C until analysis. The sample storage area must be free of organic solvent vapors.

8.2.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

### 8.3 FIELD REAGENT BLANKS

8.3.1 Duplicate field reagent blanks must be handled along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill field blank sample bottles with reagent

water, seal, and ship to the sampling site along with empty sample bottles and back to the laboratory with filled sample bottles. Wherever a set of samples is shipped and stored, it is accompanied by appropriate blanks.

- 8.3.2 Use the same procedures used for samples to add ascorbic acid and HCl to blanks (Sect. 8.1.1).

## 9. CALIBRATION

9.1 Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successful, a continuing calibration check is required at the beginning of each 8 hr. period during which analyses are performed. Additional periodic calibration checks are good laboratory practice.

### 9.2 Initial calibration

9.2.1 Calibrate the mass and abundance scales of the MS with calibration compounds and procedures prescribed by the manufacturer with any modifications necessary to meet the requirements in Sect. 9.2.2.

9.2.2 Introduce into the GC (either by purging a laboratory reagent blank or making a syringe injection) 25 ng of BFB and acquire mass spectra for m/z 35-260 at 70 eV (nominal). Use the purging procedure and/or GC conditions given in Sect. 11. If the spectrum does not meet all criteria in Table 2, the MS must be retuned and adjusted to meet all criteria before proceeding with calibration. An average spectrum across the GC peak may be used to evaluate the performance of the system.

9.2.3 Purge a medium CAL solution, for example 10-20 µg/L, using the procedure given in Sect. 11.

9.2.4 Performance criteria for the medium calibration. Examine the stored GC/MS data with the data system software. Figure 3 shows an acceptable total ion chromatogram.

9.2.4.1 GC performance. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are broad, or sensitivity poor, see Sect. 9.3.6 for some possible remedial actions.

9.2.4.2 MS sensitivity. The GC/MS/DS peak identification software should be able to recognize a GC peak in the appropriate retention time window for each of the compounds in calibration solution, and make correct tentative identifications. If fewer than 99% of the

compounds are recognized, system maintenance is required. See Sect. 9.3.6.

- 9.2.5 If all performance criteria are met, purge an aliquot of each of the other CAL solutions using the same GC/MS conditions.
- 9.2.6 Calculate a response factor (RF) for each analyte, surrogate, and isomer pair for each CAL solution using the internal standard fluorobenzene. Table 1 contains suggested quantitation ions for all compounds. This calculation is supported in acceptable GC/MS data system software (Sect. 6.3.4), and many other software programs. RF is a unitless number, but units used to express quantities of analyte and internal standard must be equivalent.

$$RF = \frac{(A_x)(Q_{is})}{(A_{is})(Q_x)}$$

where:  $A_x$  = integrated abundance of the quantitation ion of the analyte.  
 $A_{is}$  = integrated abundance of the quantitation ion of the internal standard.  
 $Q_x$  = quantity of analyte purged in ng or concentration units.  
 $Q_{is}$  = quantity of internal standard purged in ng or concentration units.

- 9.2.6.1 For each analyte and surrogate, calculate the mean RF from the analyses of the CAL solutions. Calculate the standard deviation (SD) and the relative standard deviation (RSD) from each mean:  $RSD = 100 (SD/M)$ . If the RSD of any analyte or surrogate mean RF exceeds 20%, either analyze additional aliquots of appropriate CAL solutions to obtain an acceptable RSD of RFs over the entire concentration range, or take action to improve GC/MS performance. See Sect. 9.2.7.
- 9.2.7 As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a second or third order regression calibration curve.
- 9.3 Continuing calibration check. Verify the MS tune and initial calibration at the beginning of each 8-hr work shift during which analyses are performed using the following procedure.
- 9.3.1 Introduce into the GC (either by purging a laboratory reagent blank or making a syringe injection) 25 ng of BFB and acquire a mass spectrum that includes data for  $m/z$  35-260. If the spectrum does not meet all criteria (Table 2), the MS must be

retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.

- 9.3.2 Purge a medium concentration CAL solution and analyze with the same conditions used during the initial calibration.
- 9.3.3 Demonstrate acceptable performance for the criteria shown in Sect. 9.2.4.
- 9.3.4 Determine that the absolute areas of the quantitation ions of the internal standard and surrogates have not decreased by more than 30% from the areas measured in the most recent continuing calibration check, or by more than 50% from the areas measured during initial calibration. If these areas have decreased by more than these amounts, adjustments must be made to restore system sensitivity. These adjustments may require cleaning of the MS ion source, or other maintenance as indicated in Sect. 9.3.6, and recalibration. Control charts are useful aids in documenting system sensitivity changes.
- 9.3.5 Calculate the RF for each analyte and surrogate from the data measured in the continuing calibration check. The RF for each analyte and surrogate must be within 30% of the mean value measured in the initial calibration. Alternatively, if a second or third order regression is used, the point from the continuing calibration check for each analyte and surrogate must fall, within the analyst's judgement, on the curve from the initial calibration. If these conditions do not exist, remedial action must be taken which may require re-initial calibration.
- 9.3.6 Some possible remedial actions. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.
  - 9.3.6.1 Check and adjust GC and/or MS operating conditions; check the MS resolution, and calibrate the mass scale.
  - 9.3.6.2 Clean or replace the splitless injection liner; silanize a new injection liner.
  - 9.3.6.3 Flush the GC column with solvent according to manufacturer's instructions.
  - 9.3.6.4 Break off a short portion (about 1 meter) of the column from the end near the injector; or replace GC column. This action will cause a change in retention times.
  - 9.3.6.5 Prepare fresh CAL solutions, and repeat the initial calibration step.
  - 9.3.6.6 Clean the MS ion source and rods (if a quadrupole).

- 9.3.6.7 Replace any components that allow analytes to come into contact with hot metal surfaces.
- 9.3.6.8 Replace the MS electron multiplier, or any other faulty components.
- 9.4 Optional calibration for vinyl chloride using a certified gaseous mixture of vinyl chloride in nitrogen can be accomplished by the following steps.
- 9.4.1 Fill the purging device with 25.0 mL (or 5-mL) of reagent water or aqueous calibration standard.
- 9.4.2 Start to purge the aqueous mixture. Inject a known volume (between 100 and 2000  $\mu\text{L}$ ) of the calibration gas (at room temperature) directly into the purging device with a gas tight syringe. Slowly inject the gaseous sample through a septum seal at the top of the purging device at 2000  $\mu\text{L}/\text{min}$ . If the injection of the standard is made through the aqueous sample inlet port, flush the dead volume with several mL of room air or carrier gas. Inject the gaseous standard before 5 min of the 11-min purge time have elapsed.
- 9.4.3 Determine the aqueous equivalent concentration of vinyl chloride standard, in  $\mu\text{g}/\text{L}$ , injected with the equation:

$$S = 0.102 (C)(V)$$

where S = Aqueous equivalent concentration of vinyl chloride standard in  $\mu\text{g}/\text{L}$ ;  
C = Concentration of gaseous standard in ppm (v/v);  
V = Volume of standard injected in milliliters.

## 10. QUALITY CONTROL

- 10.1 Quality control (QC) requirements are the initial demonstration of laboratory capability followed by regular analyses of laboratory reagent blanks, field reagent blanks, and laboratory fortified blanks. The laboratory must maintain records to document the quality of the data generated. Additional quality control practices are recommended.
- 10.2 Initial demonstration of low system background. Before any samples are analyzed, it must be demonstrated that a laboratory reagent blank (LRB) is reasonably free of contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbants, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next section. In general, background from method analytes should be below the method detection limit.

- 10.3 Initial demonstration of laboratory accuracy and precision. Analyze five to seven replicates of a laboratory fortified blank containing each analyte of concern at a concentration in the range of 0.2-5  $\mu\text{g/L}$  (see regulations and maximum contaminant levels for guidance on appropriate concentrations).
- 10.3.1 Prepare each replicate by adding an appropriate aliquot of a quality control sample to reagent water. If a quality control sample containing the method analytes is not available, a primary dilution standard made from a source of reagents different than those used to prepare the calibration standards may be used. Also add the appropriate amounts of internal standard and surrogates if they are being used. Analyze each replicate according to the procedures described in Section 11, and on a schedule that results in the analyses of all replicates over a period of several days.
- 10.3.2 Calculate the measured concentration of each analyte in each replicate, the mean concentration of each analyte in all replicates, and mean accuracy (as mean percentage of true value) for each analyte, and the precision (as relative standard deviation, RSD) of the measurements for each analyte. Calculate the MDL of each analyte using the procedures described in Sect. 13.2 (2).
- 10.3.3 For each analyte and surrogate, the mean accuracy, expressed as a percentage of the true value, should be 80-120% and the RSD should be <20%. Some analytes, particularly the early eluting gases and late eluting higher molecular weight compounds, are measured with less accuracy and precision than other analytes. The method detection limits must be sufficient to detect analytes at the required levels. If these criteria are not met for an analyte, take remedial action and repeat the measurements for that analyte to demonstrate acceptable performance before samples are analyzed.
- 10.3.4 Develop and maintain a system of control charts to plot the precision and accuracy of analyte and surrogate measurements as a function of time. Charting of surrogate recoveries is an especially valuable activity since these are present in every sample and the analytical results will form a significant record of data quality.
- 10.4 Monitor the integrated areas of the quantitation ions of the internal standards and surrogates in continuing calibration checks. These should remain reasonably constant over time. A drift of more than 50% in any area is indicative of a loss in sensitivity, and the problem must be found and corrected. These integrated areas should also be reasonably constant in laboratory fortified blanks and samples.

- 10.5 Laboratory reagent blanks. With each batch of samples processed as a group within a work shift, analyze a laboratory reagent blank to determine the background system contamination. A FRB (Sect. 10.7) may be used in place of a LRB.
- 10.6 With each batch of samples processed as a group within a work shift, analyze a single laboratory fortified blank (LFB) containing each analyte of concern at a concentration as determined in 10.3. If more than 20 samples are included in a batch, analyze one LFB for every 20 samples. Use the procedures described in 10.3.3 to evaluate the accuracy of the measurements, and to estimate whether the method detection limits can be obtained. If acceptable accuracy and method detection limits cannot be achieved, the problem must be located and corrected before further samples are analyzed. Add these results to the on-going control charts to document data quality.
- 10.7 With each set of field samples a field reagent blank (FRB) should be analyzed. The results of these analyses will help define contamination resulting from field sampling and transportation activities. If the FRB shows unacceptable contamination, a LRB must be measured to define the source of the impurities.
- 10.8 At least quarterly, replicates of laboratory fortified blanks should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.
- 10.9 At least quarterly, analyze a quality control sample (QCS) from an external source. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem source.
- 10.10 Sample matrix effects have not been observed when this method is used with distilled water, reagent water, drinking water, and ground water. Therefore, analysis of a laboratory fortified sample matrix (LFM) is not required. It is recommended that sample matrix effects be evaluated at least quarterly using the QCS described in 10.9.
- 10.11 Numerous other quality control measures are incorporated into other parts of this procedure, and serve to alert the analyst to potential problems.

## 11. PROCEDURE

### 11.1 SAMPLE INTRODUCTION AND PURGING

- 11.1.1 This method is designed for a 25-mL sample volume, but a smaller (5 mL) sample volume is recommended if the GC/MS system has adequate sensitivity to achieve the required method detection limits. Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the

purging device and open the syringe valve on the purging device.

- 11.1.2 Remove the plungers from two 25-mL (or 5-mL depending on sample size) syringes and attach a closed syringe valve to each. Warm the sample to room temperature, open the sample bottle, and carefully pour the sample into one of the syringe barrels to just short of overflowing. Replace the syringe plunger, invert the syringe, and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 25.0-mL (or 5-mL). For samples and blanks, add 5- $\mu$ L of the fortification solution containing the internal standard and the surrogates to the sample through the syringe valve. For calibration standards and laboratory fortified blanks, add 5- $\mu$ L of the fortification solution containing the internal standard only. Close the valve. Fill the second syringe in an identical manner from the same sample bottle. Reserve this second syringe for a reanalysis if necessary.
- 11.1.3 Attach the sample syringe valve to the syringe valve on the purging device. Be sure that the trap is cooler than 25 $^{\circ}$ C, then open the sample syringe valve and inject the sample into the purging chamber. Close both valves and initiate purging. Purge the sample for 11.0 min at ambient temperature.

## 11.2 SAMPLE DESORPTION

- 11.2.1 Non-cryogenic interface -- After the 11-min purge, place the purge and trap system in the desorb mode and preheat the trap to 180 $^{\circ}$ C without a flow of desorption gas. Then simultaneously start the flow of desorption gas at 15-mL/min for about 4 min, begin the temperature program of the gas chromatograph, and start data acquisition.
- 11.2.2 Cryogenic interface -- After the 11-min purge, place the purge and trap system in the desorb mode, make sure the cryogenic interface is a -150 $^{\circ}$ C or lower, and rapidly heat the trap to 180 $^{\circ}$ C while backflushing with an inert gas at 4 mL/min for about 5 min. At the end of the 5 min desorption cycle, rapidly heat the cryogenic trap to 250 $^{\circ}$ C, and simultaneously begin the temperature program of the gas chromatograph, and start data acquisition.
- 11.2.3 While the trapped components are being introduced into the gas chromatograph (or cryogenic interface), empty the purging device using the sample syringe and wash the chamber with two 25-mL flushes of reagent water. After the purging device has been emptied, leave syringe valve open to allow the purge gas to vent through the sample introduction needle.

- 11.3 GAS CHROMATOGRAPHY/MASS SPECTROMETRY -- Acquire and store data over the mass range 35-260 with a total cycle time (including scan overhead time) of 2 sec or less. Cycle time must be adjusted to measure five or more spectra during the elution of each GC peak. Several alternative temperature programs can be used.
- 11.3.1 Single ramp linear temperature program for wide bore columns 1 and 2 with a jet separator. Adjust the helium carrier gas flow rate to about 15 mL/min. The column temperature is reduced 10°C and held for 5 min from the beginning of desorption, then programmed to 160°C at 6°C/min, and held until all components have eluted.
- 11.3.2 Multi-ramp linear temperature program for wide bore column 2 with the open split interface. Adjust the helium carrier gas flow rate to about 4.6 mL/min. The column temperature is reduced 10°C and held for 6 min from the beginning of desorption, then heated to 70°C at 10°C/min, heated to 120°C at 5°C/min, heated to 180°C at 8°C/min, and held at 180°C until all compounds have eluted.
- 11.3.3 Single ramp linear temperature program for narrow bore column 3 with a cryogenic interface. Adjust the helium carrier gas flow rate to about 4 mL/min. The column temperature is reduced 10°C and held for 5 min from the beginning of vaporization from the cryogenic trap, programmed at 6°C/min for 10 min, then 15°C/min for 5 min to 145°C, and held until all components have eluted.
- 11.4 TRAP RECONDITIONING -- After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 sec, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.
- 11.5 TERMINATION OF DATA ACQUISITION -- When all the sample components have eluted from the GC, terminate MS data acquisition. Use appropriate data output software to display full range mass spectra and appropriate plots of ion abundance as a function of time. If any ion abundance exceeds the system working range, dilute the sample aliquot in the second syringe with reagent water and analyze the diluted aliquot.
- 11.6 IDENTIFICATION OF ANALYTES -- Identify a sample component by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time of the sample component should be within three standard deviations of the mean retention time of the compound in the calibration mixture.

- 11.6.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10 to 50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.
- 11.6.2 Identification requires expert judgement when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.
- 11.6.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes and two of the three dichlorobenzenes are examples of structural isomers that may not be resolved on the capillary columns. If unresolved, these groups of isomers must be reported as isomeric pairs.
- 11.6.4 Methylene chloride and other background components appear in variable quantities in laboratory and field reagent blanks, and generally cannot be accurately measured. Subtraction of the concentration in the blank from the concentration in the sample is not acceptable because the concentration of the background in the blank is highly variable.

## 12. CALCULATIONS

- 12.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations if unique ions with adequate intensities are available for quantitation.

12.1.1 Calculate analyte and surrogate concentrations.

$$C_x = \frac{(A_x)(Q_{is}) 1000}{(A_{is}) RF V}$$

where:  $C_x$  = concentration of analyte or surrogate in  $\mu\text{g/L}$  in the water sample.  
 $A_x$  = integrated abundance of the quantitation ion of the analyte in the sample.  
 $A_{is}$  = integrated abundance of the quantitation ion of the internal standard in the sample.  
 $Q_{is}$  = total quantity (in micrograms) of internal standard added to the water sample.  
 $V$  = original water sample volume in mL.  
 $RF$  = mean response factor of analyte from the initial calibration.

12.1.2 Alternatively, use the GC/MS system software or other available proven software to compute the concentrations of the analytes and surrogates from the second or third order regression curves.

12.1.3 Calculations should utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty). Experience indicates that three significant figures may be used for concentrations above  $99 \mu\text{g/L}$ , two significant figures for concentrations between  $1-99 \mu\text{g/L}$ , and one significant figure for lower concentrations.

12.1.4 Calculate the total trihalomethane concentration by summing the four individual trihalomethane concentrations in  $\mu\text{g/L}$ .

### 13. ACCURACY AND PRECISION

13.1 Single laboratory accuracy and precision data were obtained for the method analytes using laboratory fortified blanks with analytes at concentrations between 1 and  $5 \mu\text{g/L}$ . Four sets of results were obtained using the three columns specified (Sect. 6.3.2) and the open split, cryogenic, and jet separator interfaces (Sect. 6.3.3). These data are shown in Tables 4-6.

13.2 With these data, method detection limits were calculated using the formula (2):

$$MDL = S t_{(n-1, 1-\alpha = 0.99)}$$

where:

$t_{(n-1, 1-\alpha = 0.99)}$  = Student's t value for the 99% confidence level with  $n-1$  degrees of freedom,

n = number of replicates

S = the standard deviation of the replicate analyses.

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TABLE 1. MOLECULAR WEIGHTS AND QUANTITATION IONS FOR METHOD ANALYTES

Compound	MW <sup>a</sup>	Primary Quantitation Ion	Secondary Quantitation Ions
<u>Internal standard</u>			
Fluorobenzene	96	96	77
<u>Surrogates</u>			
4-Bromofluorobenzene	174	95	174,176
1,2-Dichlorobenzene-d4	150	152	115,150
<u>Target Analytes</u>			
Benzene	78	78	77
Bromobenzene	156	156	77,158
Bromochloromethane	128	128	49,130
Bromodichloromethane	162	83	85,127
Bromoform	250	173	175,252
Bromomethane	94	94	96
n-Butylbenzene	134	91	134
sec-Butylbenzene	134	105	134
tert-Butylbenzene	134	119	91
Carbon tetrachloride	152	117	119
Chlorobenzene	112	112	77,114
Chloroethane	64	64	66
Chloroform	118	83	85
Chloromethane	50	50	52
2-Chlorotoluene	126	91	126
4-Chlorotoluene	126	91	126
Dibromochloromethane	206	129	127
1,2-Dibromo-3-Chloropropane	234	75	155,157
1,2-Dibromoethane	186	107	109,188
Dibromomethane	172	93	95,174
1,2-Dichlorobenzene	146	146	111,148
1,3-Dichlorobenzene	146	146	111,148
1,4-Dichlorobenzene	146	146	111,148
Dichlorodifluoromethane	120	85	87
1,1-Dichloroethane	98	63	65,83
1,2-Dichloroethane	98	62	98
1,1-Dichloroethene	96	96	61,63
cis-1,2-Dichloroethene	96	96	61,98
trans-1,2-Dichloroethene	96	96	61,98
1,2-Dichloropropane	112	63	112
1,3-Dichloropropane	112	76	78
2,2-Dichloropropane	112	77	97
1,1-Dichloropropene	110	75	110,77

TABLE 1. (continued)

Compound	MW <sup>a</sup>	Primary Quantitation Ion	Secondary Quantitation Ions
cis-1,3-dichloropropene	110	75	110
trans-1,3-dichloropropene	110	75	110
Ethylbenzene	106	91	106
Hexachlorobutadiene	258	225	260
Isopropylbenzene	120	105	120
4-Isopropyltoluene	134	119	134,91
Methylene chloride	84	84	86,49
Naphthalene	128	128	
n-Propylbenzene	120	91	120
Styrene	104	104	78
1,1,1,2-Tetrachloroethane	166	131	133,119
1,1,2,2-Tetrachloroethane	166	83	131,85
Tetrachloroethene	164	166	168,129
Toluene	92	92	91
1,2,3-Trichlorobenzene	180	180	182
1,2,4-Trichlorobenzene	180	180	182
1,1,1-Trichloroethane	132	97	99,61
1,1,2-Trichloroethane	132	83	97,85
Trichloroethene	130	95	130,132
Trichlorofluoromethane	136	101	103
1,2,3-Trichloropropane	146	75	77
1,2,4-Trimethylbenzene	120	105	120
1,3,5-Trimethylbenzene	120	105	120
Vinyl Chloride	62	62	64
o-Xylene	106	106	91
m-Xylene	106	106	91
p-Xylene	106	106	91

<sup>a</sup>Monoisotopic molecular weight calculated from the atomic masses of the isotopes with the smallest masses.

TABLE 2. CHROMATOGRAPHIC RETENTION TIMES FOR METHOD ANALYTES ON THREE COLUMNS WITH FOUR SETS OF CONDITIONS<sup>a</sup>

Compound	Retention		Time (min:sec)	
	Column 1 <sup>b</sup>	Column 2 <sup>b</sup>	Column 2 <sup>c</sup>	Column 3 <sup>d</sup>
<u>Internal standard</u>				
Fluorobenzene	8:49	6:27	14:06	8:03
<u>Surrogates</u>				
4-Bromofluorobenzene	18:38	15:43	23:38	
1,2-Dichlorobenzene-d4	22:16	19:08	27:25	
<u>Target Analytes</u>				
Benzene	8:14	5:40	13:30	7:25
Bromobenzene	18:57	15:52	24:00	16:25
Bromochloromethane	6:44	4:23	12:22	5:38
Bromodichloromethane	10:35	8:29	15:48	9:20
Bromoform	17:56	14:53	22:46	15:42
Bromomethane	2:01	0:58	4:48	1:17
n-Butylbenzene	22:13	19:29	27:32	17:57
sec-Butylbenzene	20:47	18:05	26:08	17:28
tert-Butylbenzene	20:17	17:34	25:36	17:19
Carbon Tetrachloride	7:37	5:16	13:10	7:25
Chlorobenzene	15:46	13:01	20:40	14:20
Chloroethane	2:05	1:01		1:27
Chloroform	6:24	4:48	12:36	5:33
Chloromethane	1:38	0:44	3:24	0:58
2-Chlorotoluene	19:20	16:25	24:32	16:44
4-Chlorotoluene	19:30	16:43	24:46	16:49
Cyanogen chloride				1:03
Dibromochloromethane	14:23	11:51	19:12	12:48
1,2-Dibromo-3-Chloropropane	24:32	21:05		18:02
1,2-Dibromoethane	14:44	11:50	19:24	13:36
Dibromomethane	10:39	7:56	15:26	9:05
1,2-Dichlorobenzene	22:31	19:10	27:26	17:47
1,3-Dichlorobenzene	21:13	18:08	26:22	17:28
1,4-Dichlorobenzene	21:33	18:23	26:36	17:38
Dichlorodifluoromethane	1:33	0:42	3:08	0:53
1,1-Dichloroethane	4:51	2:56	10:48	4:02
1,2-Dichloroethane	8:24	5:50	13:38	7:00
1,1-Dichloroethene	2:53	1:34	7:50	2:20
cis-1,2-Dichloroethene	6:11	3:54	11:56	5:04
trans-1,2-Dichloroethene	3:59	2:22	9:54	3:32
1,2-Dichloropropane	10:05	7:40	15:12	8:56
1,3-Dichloropropane	14:02	11:19	18:42	12:29
2,2-Dichloropropane	6:01	3:48	11:52	5:19
1,1-Dichloropropene	7:49	5:17	13:06	7:10

TABLE 2. (continued)

Compound	Retention		Time (min:sec)	
	Column 1 <sup>b</sup>	Column 2 <sup>b</sup>	Column 2 <sup>c</sup>	Column 3 <sup>d</sup>
cis-1,3-dichloropropene			17:54	
trans-1,3-dichloropropene			16:42	
Ethylbenzene	15:59	13:23	21:00	14:44
Hexachlorobutadiene	26:59	23:41	32:04	19:14
Isopropylbenzene	18:04	15:28	23:18	16:25
4-Isopropyltoluene	21:12	18:31	26:30	17:38
Methylene Chloride	3:36	2:04	9:16	2:40
Naphthalene	27:10	23:31	32:12	19:04
n-Propylbenzene	19:04	16:25	24:20	16:49
Styrene	17:19	14:36	22:24	15:47
1,1,1,2-Tetrachloroethane	15:56	13:20	20:52	14:44
1,1,2,2-Tetrachloroethane	18:43	16:21	24:04	15:47
Tetrachloroethene	13:44	11:09	18:36	13:12
Toluene	12:26	10:00	17:24	11:31
1,2,3-Trichlorobenzene	27:47	24:11	32:58	19:14
1,2,4-Trichlorobenzene	26:33	23:05	31:30	18:50
1,1,1-Trichloroethane	7:16	4:50	12:50	6:46
1,1,2-Trichloroethane	13:25	11:03	18:18	11:59
Trichloroethene	9:35	7:16	14:48	9:01
Trichlorofluoromethane	2:16	1:11	6:12	1:46
1,2,3-Trichloropropane	19:01	16:14	24:08	16:16
1,2,4-Trimethylbenzene	20:20	17:42	31:30	17:19
1,3,5-Trimethylbenzene	19:28	16:54	24:50	16:59
Vinyl chloride	1:43	0:47	3:56	1:02
o-Xylene	17:07	14:31	22:16	15:47
m-Xylene	16:10	13:41	21:22	15:18
p-Xylene	16:07	13:41	21:18	15:18

<sup>a</sup>Columns 1-3 are those given in Sect. 6.3.2.1; retention times were measured from the beginning of thermal desorption from the trap (columns 1-2) or from the beginning of thermal release from the cryogenic interface (column 3).

<sup>b</sup>GC conditions given in Sect. 11.3.1.

<sup>c</sup>GC conditions given in Sect. 11.3.2.

<sup>d</sup>GC conditions given in Sect. 11.3.3.

TABLE 3. ION ABUNDANCE CRITERIA FOR 4-BROMOFLUOROBENZENE (BFB)

Mass (M/z)	Relative Abundance Criteria
50	15 to 40% of mass 95
75	30 to 80% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

TABLE 4. ACCURACY AND PRECISION DATA FROM 16-31 DETERMINATIONS OF THE METHOD ANALYTES IN REAGENT WATER USING WIDE BORE CAPILLARY COLUMN 1<sup>a</sup>

Compound	True Conc. Range ( $\mu\text{g/L}$ )	Mean Accuracy (% of True Value)	Rel. Std. Dev. (%)	Method Det. Limit ( $\mu\text{g/L}$ )
Benzene	0.1-10	97	5.7	0.04
Bromobenzene	0.1-10	100	5.5	0.03
Bromochloromethane	0.5-10	90	6.4	0.04
Bromodichloromethane	0.1-10	95	6.1	0.08
Bromoform	0.5-10	101	6.3	0.12
Bromomethane	0.5-10	95	8.2	0.11
n-Butylbenzene	0.5-10	100	7.6	0.11
sec-Butylbenzene	0.5-10	100	7.6	0.13
tert-Butylbenzene	0.5-10	102	7.3	0.14
Carbon tetrachloride	0.5-10	84	8.8	0.21
Chlorobenzene	0.1-10	98	5.9	0.04
Chloroethane	0.5-10	89	9.0	0.10
Chloroform	0.5-10	90	6.1	0.03
Chloromethane	0.5-10	93	8.9	0.13
2-Chlorotoluene	0.1-10	90	6.2	0.04
4-Chlorotoluene	0.1-10	99	8.3	0.06
Dibromochloromethane	0.1-10	92	7.0	0.05
1,2-Dibromo-3-chloropropane	0.5-10	83	19.9	0.26
1,2-Dibromoethane	0.5-10	102	3.9	0.06
Dibromomethane	0.5-10	100	5.6	0.24
1,2-Dichlorobenzene	0.1-10	93	6.2	0.03
1,3-Dichlorobenzene	0.5-10	99	6.9	0.12
1,4-Dichlorobenzene	0.2-20	103	6.4	0.03
Dichlorodifluoromethane	0.5-10	90	7.7	0.10
1,1-Dichloroethane	0.5-10	96	5.3	0.04
1,2-Dichloroethane	0.1-10	95	5.4	0.06
1,1-Dichloroethene	0.1-10	94	6.7	0.12
cis-1,2 Dichloroethene	0.5-10	101	6.7	0.12
trans-1,2-Dichloroethene	0.1-10	93	5.6	0.06
1,2-Dichloropropane	0.1-10	97	6.1	0.04
1,3-Dichloropropane	0.1-10	96	6.0	0.04
2,2-Dichloropropane	0.5-10	86	16.9	0.35
1,1-Dichloropropene	0.5-10	98	8.9	0.10
cis-1,2-Dichloropropene				
trans-1,2-Dichloropropene				
Ethylbenzene	0.1-10	99	8.6	0.06
Hexachlorobutadiene	0.5-10	100	6.8	0.11
Isopropylbenzene	0.5-10	101	7.6	0.15
4-Isopropyltoluene	0.1-10	99	6.7	0.12
Methylene chloride	0.1-10	95	5.3	0.03
Naphthalene	0.1-100	104	8.2	0.04
n-Propylbenzene	0.1-10	100	5.8	0.04
Styrene	0.1-100	102	7.2	0.04

TABLE 4. (Continued)

Compound	True Conc. Range ( $\mu\text{g/L}$ )	Mean Accuracy (% of True Value)	Rel. Std. Dev. (%)	Method Det. Limit ( $\mu\text{g/L}$ )
1,1,1,2-Tetrachloroethane	0.5-10	90	6.8	0.05
1,1,2,2-Tetrachloroethane	0.1-10	91	6.3	0.04
Tetrachloroethene	0.5-10	89	6.8	0.14
Toluene	0.5-10	102	8.0	0.11
1,2,3-Trichlorobenzene	0.5-10	109	8.6	0.03
1,2,4-Trichlorobenzene	0.5-10	108	8.3	0.04
1,1,1-Trichloroethane	0.5-10	98	8.1	0.08
1,1,2-Trichloroethane	0.5-10	104	7.3	0.10
Trichloroethene	0.5-10	90	7.3	0.19
Trichlorofluoromethane	0.5-10	89	8.1	0.08
1,2,3-Trichloropropane	0.5-10	108	14.4	0.32
1,2,4-Trimethylbenzene	0.5-10	99	8.1	0.13
1,3,5-Trimethylbenzene	0.5-10	92	7.4	0.05
Vinyl chloride	0.5-10	98	6.7	0.17
o-Xylene	0.1-31	103	7.2	0.11
m-Xylene	0.1-10	97	6.5	0.05
p-Xylene	0.5-10	104	7.7	0.13

<sup>a</sup>Data obtained by Robert W. Slater using column 1 with a jet separator interface and a quadrupole mass spectrometer (Sect. 11.3.1) with analytes divided among three solutions.

TABLE 5. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS OF THE METHOD ANALYTES IN REAGENT WATER USING THE CRYOGENIC TRAPPING OPTION AND A NARROW BORE CAPILLARY COLUMN 3<sup>a</sup>

Compound	True Conc. ( $\mu\text{g/L}$ )	Mean Accuracy (% of True Value)	Rel. Std. Dev. (%)	Method Dect. Limit ( $\mu\text{g/L}$ )
Benzene	0.1	99	6.2	0.03
Bromobenzene	0.5	97	7.4	0.11
Bromochloromethane	0.5	97	5.8	0.07
Bromodichloromethane	0.1	100	4.6	0.03
Bromoform	0.1	99	5.4	0.20
Bromomethane	0.1	99	7.1	0.06
n-Butylbenzene	0.5	94	6.0	0.03
sec-Butylbenzene	0.5	90	7.1	0.12
tert-Butylbenzene	0.5	90	2.5	0.33
Carbon tetrachloride	0.1	92	6.8	0.08
Chlorobenzene	0.1	91	5.8	0.03
Chloroethane	0.1	100	5.8	0.02
Chloroform	0.1	95	3.2	0.02
Chloromethane	0.1	99	4.7	0.05
2-Chlorotoluene	0.1	99	4.6	0.05
4-Chlorotoluene	0.1	96	7.0	0.05
Cyanogen chloride <sup>b</sup>		92	10.6	0.30
Dibromochloromethane	0.1	99	5.6	0.07
1,2-Dibromo-3-chloropropane	0.1	92	10.0	0.05
1,2-Dibromoethane	0.1	97	5.6	0.02
Dibromomethane	0.1	93	6.9	0.03
1,2-Dichlorobenzene	0.1	97	3.5	0.05
1,3-Dichlorobenzene	0.1	99	6.0	0.05
1,4-Dichlorobenzene	0.1	93	5.7	0.04
Dichlorodifluoromethane	0.1	99	8.8	0.11
1,1-Dichloroethane	0.1	98	6.2	0.03
1,2-Dichloroethane	0.1	100	6.3	0.02
1,1-Dichloroethene	0.1	95	9.0	0.05
cis-1,2 Dichloroethene	0.1	100	3.7	0.06
trans-1,2-Dichloroethene	0.1	98	7.2	0.03
1,2-Dichloropropane	0.1	96	6.0	0.02
1,3-Dichloropropane	0.1	99	5.8	0.04
2,2-Dichloropropane	0.1	99	4.9	0.05
1,1-Dichloropropene	0.1	98	7.4	0.02
cis-1,3-Dichloropropene				
trans-1,3-Dichloropropene				
Ethylbenzene	0.1	99	5.2	0.03
Hexachlorobutadiene	0.1	100	6.7	0.04
Isopropylbenzene	0.5	98	6.4	0.10
4-Isopropyltoluene	0.5	87	13.0	0.26
Methylene chloride	0.5	97	13.0	0.09
Naphthalene	0.1	98	7.2	0.04

TABLE 5. (Continued)

Compound	True Conc. ( $\mu\text{g/L}$ )	Mean Accuracy (% of True Value)	Rel. Std. Dev. (%)	Method Dect. Limit ( $\mu\text{g/L}$ )
n-Propylbenzene	0.1	99	6.6	0.06
Styrene	0.1	96	19.0	0.06
1,1,1,2-Tetrachloroethane	0.1	100	4.7	0.04
1,1,2,2-Tetrachloroethane	0.5	100	12.0	0.20
Tetrachloroethene	0.1	96	5.0	0.05
Toluene	0.1	100	5.9	0.08
1,2,3-Trichlorobenzene	0.1	98	8.9	0.04
1,2,4-Trichlorobenzene	0.1	91	16.0	0.20
1,1,1-Trichloroethane	0.1	100	4.0	0.04
1,1,2-Trichloroethane	0.1	98	4.9	0.03
Trichloroethene	0.1	96	2.0	0.02
Trichlorofluoromethane	0.1	97	4.6	0.07
1,2,3-Trichloropropane	0.1	96	6.5	0.03
1,2,4-Trimethylbenzene	0.1	96	6.5	0.04
1,3,5-Trimethylbenzene	0.1	99	4.2	0.02
Vinyl chloride	0.1	96	0.2	0.04
o-Xylene	0.1	94	7.5	0.06
m-Xylene	0.1	94	4.6	0.03
p-Xylene	0.1	97	6.1	0.06

<sup>a</sup>Data obtained by Caroline A. Madding using column 3 with a cryogenic interface and a quadrupole mass spectrometer (Sect 11.3.3).

<sup>b</sup>Reference 8.

TABLE 6. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS OF THE METHOD ANALYTES IN REAGENT WATER USING WIDE BORE CAPILLARY COLUMN 2<sup>a</sup>

Compound	No. <sup>b</sup>	Mean Accuracy (% of True Value, 2 µg/L Conc.)		Mean Accuracy (% of True Value, 0.2 µg/L Conc.)	
			RSD (%)		RSD (%)
<u>Internal Standard</u>					
Fluorobenzene	1	-	-	-	-
<u>Surrogates</u>					
4-Bromofluorobenzene	2	98	1.8	96	1.3
1,2-Dichlorobenzene-d <sub>4</sub>	3	97	3.2	95	1.7
<u>Target Analytes</u>					
Benzene	37	97	4.4	113	1.8
Bromobenzene	38	102	3.0	101	1.9
Bromochloromethane	4	99	5.2	102	2.9
Bromodichloromethane	5	96	1.8	100	1.8
Bromoform	6	89	2.4	90	2.2
Bromomethane	7	55	27.	52	6.7
n-Butylbenzene	39	89	4.8	87	2.3
sec-Butylbenzene	40	102	3.5	100	2.8
tert-Butylbenzene	41	101	4.5	100	2.9
Carbon tetrachloride	8	84	3.2	92	2.6
Chlorobenzene	42	104	3.1	103	1.6
Chloroethane <sup>c</sup>					
Chloroform	9	97	2.0	95	2.1
Chloromethane	10	110	5.0	d	
2-Chlorotoluene	43	91	2.4	108	3.1
4-Chlorotoluene	44	89	2.0	108	4.4
Dibromochloromethane	11	95	2.7	100	3.0
1,2-Dibromo-3-chloropropane <sup>c</sup>					
1,2-Dibromoethane <sup>c</sup>					
Dibromomethane	13	99	2.1	95	2.2
1,2-Dichlorobenzene	45	93	2.7	94	5.1
1,3-Dichlorobenzene	46	100	4.0	87	2.3
1,4-Dichlorobenzene	47	98	4.1	94	2.8
Dichlorodifluoromethane	14	38	25.	d	
1,1-Dichloroethane	15	97	2.3	85	3.6
1,2-Dichloroethane	16	102	3.8	100	2.1
1,1-Dichloroethene	17	90	2.2	87	3.8
cis-1,2-Dichloroethene	18	100	3.4	89	2.9
trans-1,2-Dichloroethene	19	92	2.1	85	2.3

TABLE 6. (Continued)

Compound	No. <sup>b</sup>	Mean Accuracy (% of True Value, 2 $\mu\text{g/L}$ Conc.)		Mean Accuracy (% of True Value, 0.2 $\mu\text{g/L}$ Conc.)	
			RSD (%)		RSD (%)
1,2-Dichloropropane	20	102	2.2	103	2.9
1,3-Dichloropropane	21	92	3.7	93	3.2
2,2-Dichloropropane <sup>c</sup>					
1,1-Dichloropropene <sup>c</sup>					
cis-1,3-Dichloropropene <sup>c</sup>					
trans-1,3-Dichloropropene	25	96	1.7	99	2.1
Ethylbenzene	48	96	9.1	100	4.0
Hexachlorobutadiene	26	91	5.3	88	2.4
Isopropylbenzene	49	103	3.2	101	2.1
4-Isopropyltoluene	50	95	3.6	95	3.1
Methylene chloride	27	e		e	
Naphthalene	51	93	7.6	78	8.3
n-Propylbenzene	52	102	4.9	97	2.1
Styrene	53	95	4.4	104	3.1
1,1,1,2-Tetrachloroethane	28	99	2.7	95	3.8
1,1,2,2-Tetrachloroethane	29	101	4.6	84	3.6
Tetrachloroethene	30	97	4.5	92	3.3
Toluene	54	105	2.8	126	1.7
1,2,3-Trichlorobenzene	55	90	5.7	78	2.9
1,2,4-Trichlorobenzene	56	92	5.2	83	5.9
1,1,1-Trichloroethane	31	94	3.9	94	2.5
1,1,2-Trichloroethane	32	107	3.4	109	2.8
Trichloroethene	33	99	2.9	106	2.5
Trichlorofluoromethane	34	81	4.6	48	13.
1,2,3-Trichloropropane	35	97	3.9	91	2.8
1,2,4-Trimethylbenzene	57	93	3.1	106	2.2
1,3,5-Trimethylbenzene	58	88	2.4	97	3.2
Vinyl chloride	36	104	3.5	115	14.
o-Xylene	59	97	1.8	98	1.7
m-Xylene	60	f		f	
p-Xylene	61	98	2.3	103	1.4

<sup>a</sup>Data obtained by James W. Eichelberger using column 2 with the open split interface and an ion trap mass spectrometer (Sect. 11.3.2) with all method analytes in the same reagent water solution.

<sup>b</sup>Designation in Figures 1 and 2.

<sup>c</sup>Not measured; authentic standards were not available.

<sup>d</sup>Not found at 0.2  $\mu\text{g/L}$ .

<sup>e</sup>Not measured; methylene chloride was in the laboratory reagent blank.

<sup>f</sup>m-xylene coelutes with and cannot be distinguished from its isomer p-xylene, No 61.

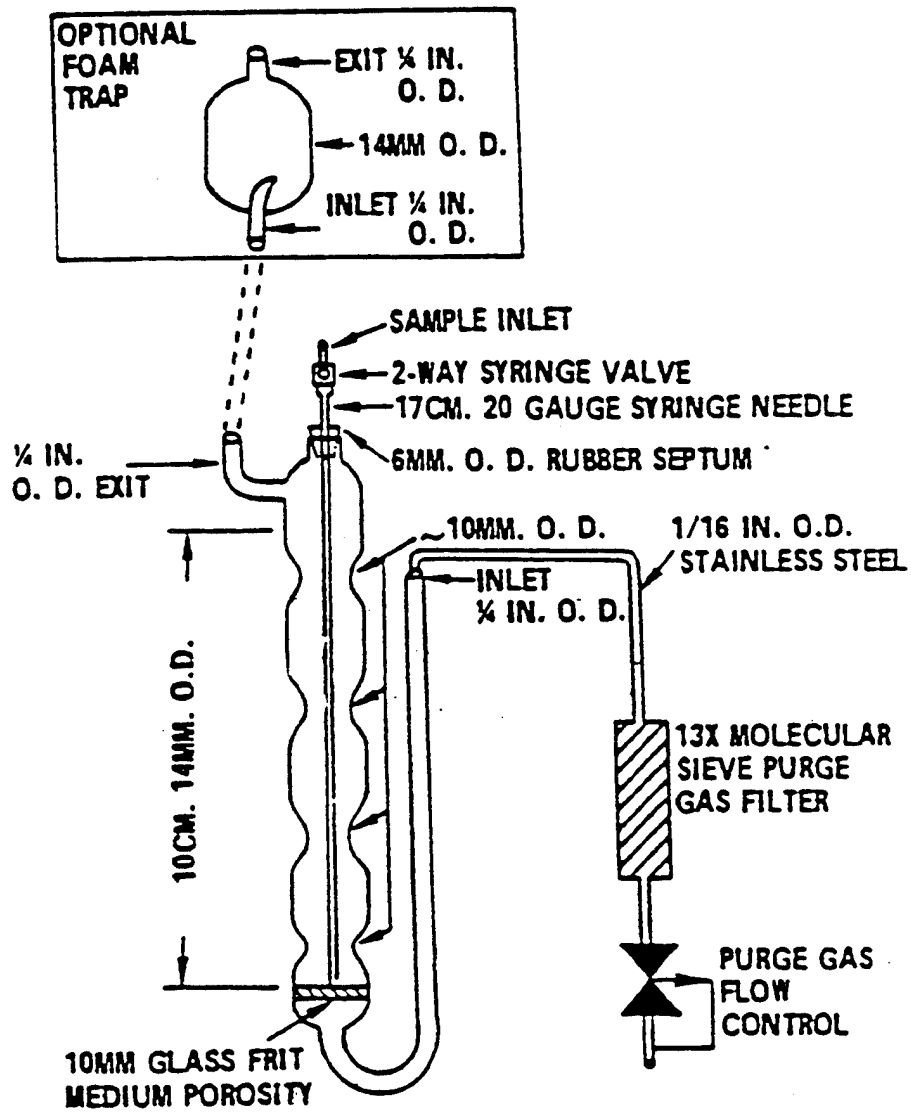


FIGURE 1. PURGING DEVICE

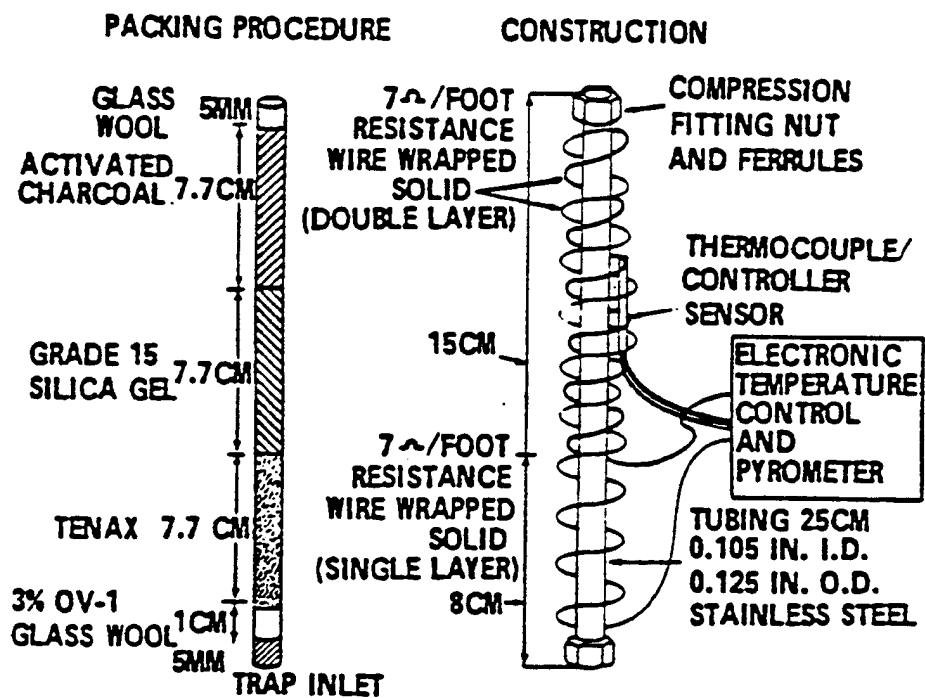


FIGURE 2. TRAP PACKINGS AND CONSTRUCTION TO INCLUDE DESORB CAPABILITY

FIGURE 3. NORMALIZED TOTAL ION CURRENT CHROMATOGRAM FROM A VOLATILE COMPOUND CALIBRATION MIXTURE CONTAINING 25 ng (5 µg/L) OF MOST COMPOUNDS. THE COMPOUND IDENTIFICATION NUMBERS ARE GIVEN IN TABLE 6.

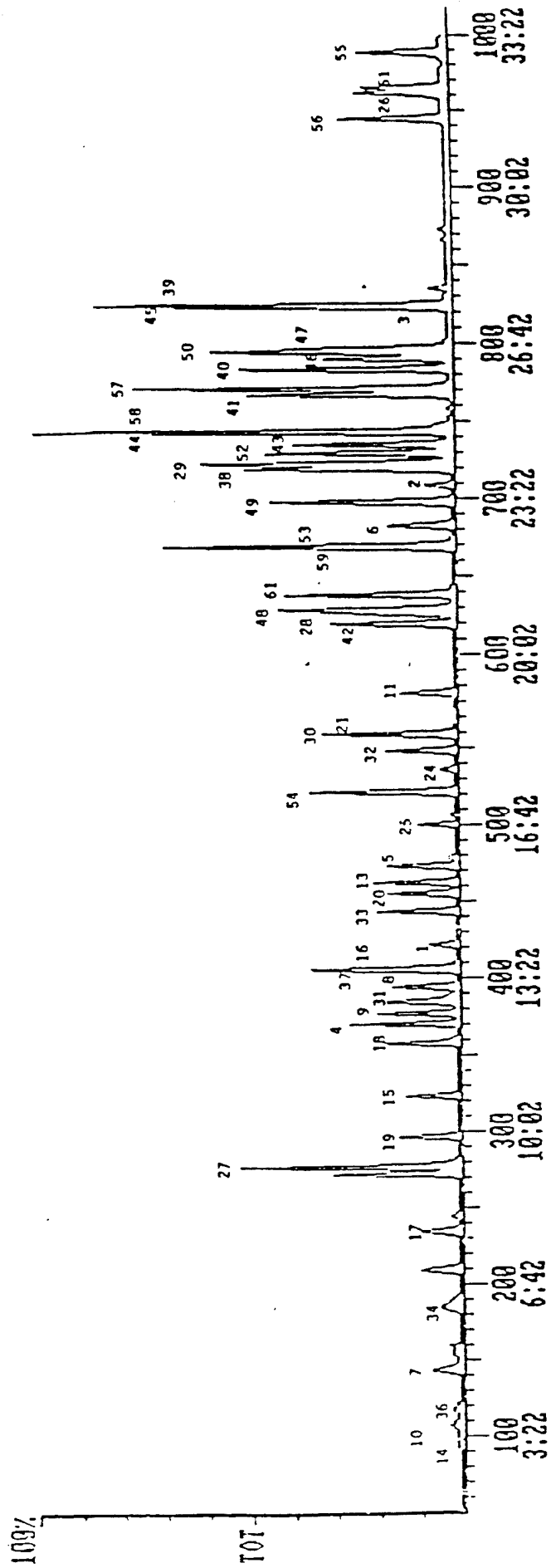
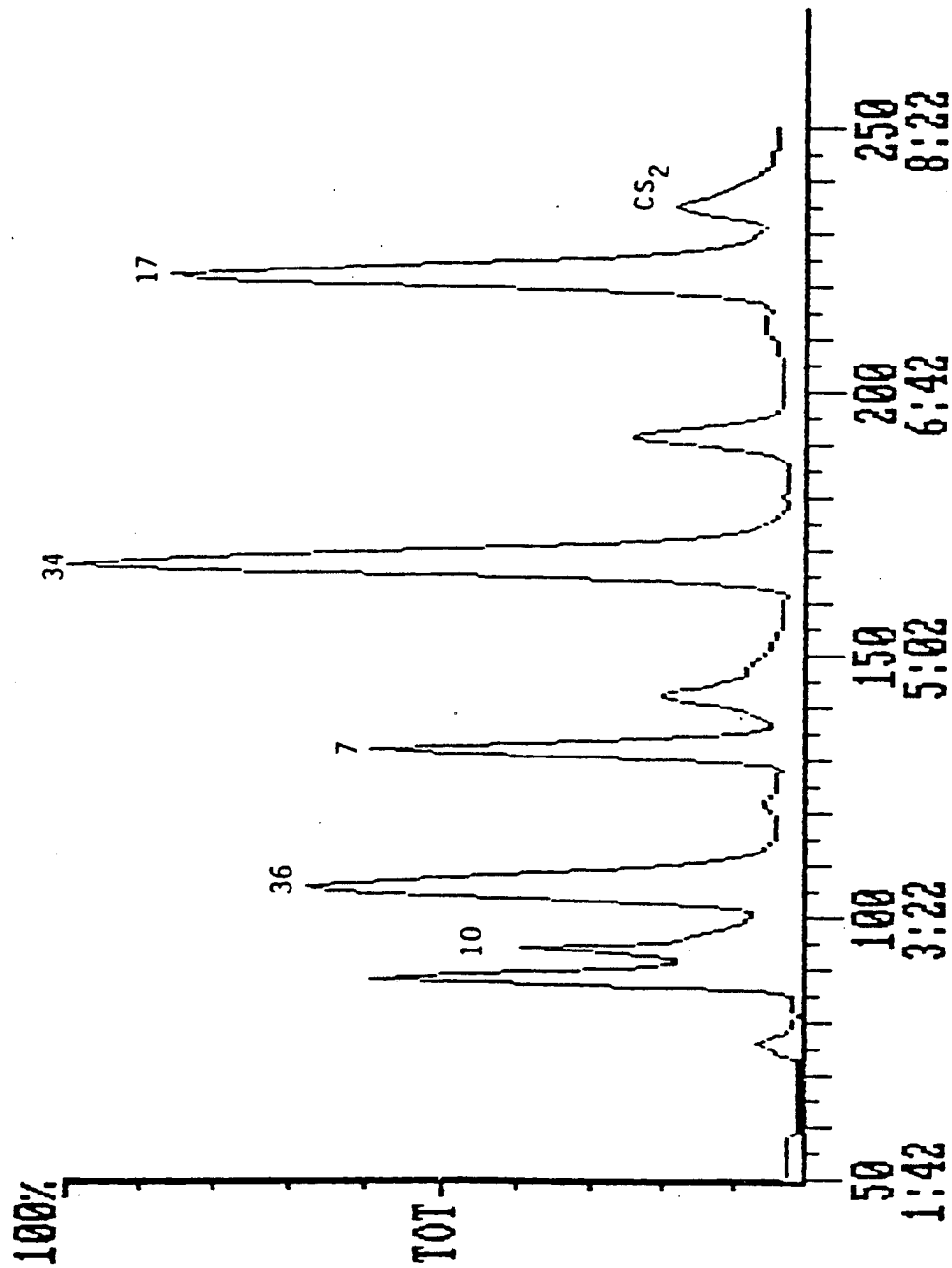


FIGURE 4. AMPLIFIED FIRST EIGHT MINUTES OF A TOTAL ION CURRENT CHROMATOGRAM FROM A VOLATILE COMPOUND CALIBRATION MIXTURE CONTAINING 25 ng (5 µg/L) OF EACH COMPONENT. THE COMPOUND IDENTIFICATION NUMBERS ARE GIVEN IN TABLE 6.



**METHOD 525. DETERMINATION OF ORGANIC COMPOUNDS IN DRINKING WATER  
BY LIQUID-SOLID EXTRACTION AND CAPILLARY COLUMN  
GAS CHROMATOGRAPHY/MASS SPECTROMETRY**

**Revision 2.1**

**J. W. Eichelberger, T. D. Behymer, W. L. Budde - Method 525,  
Revision 1.0, 2.0, 2.1 (1988)**

**ENVIRONMENTAL MONITORING SYSTEMS LABORATORY  
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## METHOD 525

### DETERMINATION OF ORGANIC COMPOUNDS IN DRINKING WATER BY LIQUID-SOLID EXTRACTION AND CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY

#### 1. SCOPE AND APPLICATION

1.1 This is a general purpose method that provides procedures for determination of organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage. The method is applicable to a wide range of organic compounds that are efficiently partitioned from the water sample onto a C<sub>18</sub> organic phase chemically bonded to a solid inorganic matrix, and sufficiently volatile and thermally stable for gas chromatography. Particulate bound organic matter will not be partitioned, and more than trace levels of particulates in the water may disrupt the partitioning process. Single-laboratory accuracy and precision data have been determined at two concentrations with two instrument systems for the following compounds:

<u>Compound</u>	<u>MW<sup>1</sup></u>	<u>Chemical Abstracts Service Registry Number</u>
Acenaphthylene	152	208-96-8
Alachlor	269	15972-60-8
Aldrin	362	309-00-2
Anthracene	178	120-12-7
Atrazine	215	1912-24-9
Benz[a]anthracene	228	56-55-3
Benzo[b]fluoranthene	252	205-82-3
Benzo[k]fluoranthene	252	207-08-9
Benzo[a]pyrene	252	50-32-8
Benzo[g,h,i]perylene	276	191-24-2
Butylbenzylphthalate	312	85-68-7
Chlordane components		
Alpha-chlordane	406	5103-71-9
Gamma-chlordane	406	5103-74-2
Trans nonachlor	440	39765-80-5
2-Chlorobiphenyl	188	2051-60-7
Chrysene	228	218-01-9
Dibenz[a,h]anthracene	278	53-70-3
Di-n-butylphthalate	278	84-72-2
2,3-Dichlorobiphenyl	222	16605-91-7
Diethylphthalate	222	84-66-2
Di(2-ethylhexyl)adipate	370	103-23-1
Di(2-ethylhexyl)phthalate	390	117-81-7
Dimethylphthalate	194	131-11-3
Endrin	378	72-20-8
Fluorene	166	86-73-7
Heptachlor	370	76-44-8

Heptachlor epoxide	386	1024-57-3
2,2',3,3',4,4',6-Heptachloro- biphenyl	392	52663-71-5
Hexachlorobenzene	282	118-74-1
2,2',4,4',5,6'-Hexachloro- biphenyl	358	60145-22-4
Hexachlorocyclopentadiene	270	77-47-4
Indeno[1,2,3,c,d]pyrene	276	193-39-5
Lindane	288	58-89-9
Methoxychlor	344	72-43-5
2,2',3,3',4,5',6,6'-Octa- chlorobiphenyl	426	40186-71-8
2,2',3',4,6-Pentachloro- biphenyl	324	60233-25-2
Pentachlorophenol	264	87-86-5
Phenanthrene	178	85-01-8
Pyrene	202	129-00-0
Simazine	201	122-34-9
2,2',4,4'-Tetrachlorobiphenyl	290	2437-79-8
Toxaphene mixture		8001-35-2
2,4,5-Trichlorobiphenyl	256	15862-07-4

<sup>1</sup>Monoisotopic molecular weight calculated from the atomic masses of the isotopes with the smallest masses.

A laboratory may use this method to identify and measure additional analytes after the laboratory obtains acceptable (defined in Sect. 10) accuracy and precision data for each added analyte.

- 1.2 Method detection limit (MDL) is defined as the statistically calculated minimum amount that can be measured with 99% confidence that the reported value is greater than zero (1). The MDL is compound dependent and is particularly dependent on extraction efficiency and sample matrix. For the listed analytes, MDLs vary from 0.01 to 15  $\mu\text{g/L}$ . The concentration calibration range of this method is 0.1  $\mu\text{g/L}$  to 10  $\mu\text{g/L}$ .

## 2. SUMMARY OF METHOD

Organic compound analytes, internal standards, and surrogates are extracted from a water sample by passing 1 liter of sample water through a cartridge containing about 1 gram of a solid inorganic matrix coated with a chemically bonded  $\text{C}_{18}$  organic phase (liquid-solid extraction, LSE). The organic compounds are eluted from the LSE cartridge with a small quantity of methylene chloride, and concentrated further by evaporation of some of the solvent. The sample components are separated, identified, and measured by injecting an aliquot of the concentrated methylene chloride extract into a high resolution fused silica capillary column of a gas chromatography/mass spectrometry (GC/MS) system. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a data base. Reference spectra and

retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure.

### 3. DEFINITIONS

- 3.1 Internal standard -- A pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.
- 3.2 Surrogate analyte -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of a surrogate analyte is to monitor method performance with each sample.
- 3.3 Laboratory duplicates (LD1 and LD2) -- Two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 3.4 Field duplicates (FD1 and FD2) -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 3.5 Laboratory reagent blank (LRB) -- An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.6 Field reagent blank (FRB) -- Reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.
- 3.7 Laboratory performance check solution (LPC) -- A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

- 3.8 Laboratory fortified blank (LFB) -- An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit.
- 3.9 Laboratory fortified sample matrix (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 3.10 Stock standard solution -- A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assayed reference compound. Stock standard solutions are used to prepare primary dilution standards.
- 3.11 Primary dilution standard solution -- A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 3.12 Calibration standard (CAL) -- a solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.13 Quality control sample (QCS) -- a sample matrix containing method analytes or a solution of method analytes in a water miscible solvent which is used to fortify reagent water or environmental samples. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance with externally prepared test materials.

#### 4. INTERFERENCES

- 4.1 During analysis, major contaminant sources are reagents and liquid-solid extraction columns. Analyses of field and laboratory reagent blanks provide information about the presence of contaminants.
- 4.2 Interfering contamination may occur when a sample containing low concentrations of compounds is analyzed immediately after a sample containing relatively high concentrations of compounds. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample containing high concentrations

of compounds, a laboratory reagent blank should be analyzed to ensure that accurate values are obtained for the next sample.

## 5. SAFETY

- 5.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are cited (2-4).
- 5.2 Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin, eyes, etc.

## 6. Apparatus and Equipment

- 6.1 All glassware must be meticulously cleaned. This may be accomplished by washing with detergent and water, rinsing with water, distilled water, or solvents, air-drying, and heating (where appropriate) in an oven. Volumetric glassware is never heated.
- 6.2 Sample containers. 1-liter or 1-quart amber glass bottles fitted with a Teflon-lined screw cap. (Bottles in which high purity solvents were received can be used as sample containers without additional cleaning if they have been handled carefully to avoid contamination during use and after use of original contents.)
- 6.3 Separatory funnels. 2-liter and 100-mL with a Teflon stopcock.
- 6.4 Liquid chromatography column reservoirs. Pear-shaped 100- or 125-mL vessels without a stopcock but with a ground glass outlet joint sized to fit the liquid-solid extraction column. (Lab Glass, Inc. part no. ML-700-706S, with a 24/40 top outer joint and a 14/35 bottom inner joint, or equivalent). A 14/35 outlet joint fits some commercial cartridges.
- 6.5 Syringe needles. No. 18 or 20 stainless steel.
- 6.6 Vacuum flasks. 1- or 2-liter with solid rubber stoppers.
- 6.7 Volumetric flasks, various sizes.
- 6.8 Laboratory or aspirator vacuum system. Sufficient capacity to maintain a slight vacuum of 13 cm (5 in.) of mercury in the vacuum flask.
- 6.9 Micro syringes, various sizes.
- 6.10 Vials. Various sizes of amber vials with Teflon-lined screw caps.

- 6.11 Drying column. Approximately 1.2 cm x 40 cm with 10 mL graduated collection vial.
- 6.12 Analytical balance. Capable of weighing 0.0001 g accurately.
- 6.13 Fused silica capillary gas chromatography column. Any capillary column that provides adequate resolution, capacity, accuracy, and precision (Sect. 10) can be used. A 30 m X 0.25 mm id fused silica capillary column coated with a 0.25  $\mu$ m bonded film of polyphenylmethylsilicone is recommended (J&W DB-5 or equivalent).
- 6.14 Gas chromatograph/mass spectrometer/data system (GC/MS/DS)
- 6.14.1 The GC must be capable of temperature programming and be equipped for splitless/split injection. The injection tube liner should be quartz and about 3 mm in diameter. The injection system must not allow the analytes to contact hot stainless steel or other metal surfaces that promote decomposition.
- 6.14.2 The GC/MS interface should allow the capillary column or transfer line exit to be placed within a few mm of the ion source. Other interfaces, for example the open split interface, are acceptable as long as the system has adequate sensitivity (see Sect. 9 for calibration requirements).
- 6.14.3 The mass spectrometer must be capable of electron ionization at a nominal electron energy of 70 eV. The spectrometer must be capable of scanning from 45 to 450 amu with a complete scan cycle time (including scan overhead) of 1.5 sec or less. (Scan cycle time = Total MS data acquisition time in sec divided by number of scans in the chromatogram). The spectrometer must produce a mass spectrum that meets all criteria in Table 1 when 5 ng or less of DFTPP is introduced into the GC. An average spectrum across the DFTPP GC peak may be used to test instrument performance.
- 6.14.4 An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software must have the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectra from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software must also allow integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as defined in Sect. 9.2.6 (or construction of a second or third order regression calibration curve), calculation of response factor statistics (mean and standard deviation), and calculation of

concentrations of analytes using either the calibration curve or the equation in Sect. 12.

## 7. REAGENTS AND CONSUMABLE MATERIALS

- 7.1 Helium carrier gas, as contaminant free as possible.
- 7.2 Liquid-solid extraction (LSE) cartridges. Cartridges are inert non-leaching plastic, for example polypropylene, or glass, and must not contain plasticizers, such as phthalate esters or adipates, that leach into methylene chloride. The cartridges are packed with about 1 gram of silica, or other inert inorganic support, whose surface is modified by chemically bonded octadecyl (C<sub>18</sub>) groups. The packing must have a narrow size distribution and must not leach organic compounds into methylene chloride. One liter of water should pass through the cartridge in about 2 hrs with the assistance of a slight vacuum of about 13 cm (5 in.) of mercury. Sect. 10 provides criteria for acceptable LSE cartridges which are available from several commercial suppliers.
- 7.3 Solvents
  - 7.3.1 Methylene chloride, acetone, toluene and methanol. High purity pesticide quality or equivalent.
  - 7.3.2 Reagent water. Water in which an interferent is not observed at the method detection limit of the compound of interest. Prepare reagent water by passing tap water through a filter bed containing about 0.5 kg of activated carbon or by using a water purification system. Store in clean, narrow-mouth bottles with Teflon-lined septa and screw caps.
- 7.4 Hydrochloric acid. 6N.
- 7.5 Sodium sulfate, anhydrous. (Soxhlet extracted with methylene chloride for a minimum of 4 hrs.)
- 7.6 Stock standard solutions. Individual solutions of analytes, surrogates, and internal standards may be purchased as certified solutions or prepared from pure materials. To prepare, add 10 mg (weighed on an analytical balance to 0.1 mg) of the pure material to 1.9 mL of methanol or acetone in a 2-mL volumetric flask, dilute to the mark, and transfer the solution to an amber glass vial. If the analytical standard is available only in quantities smaller than 10 mg, reduce the volume of solvent accordingly. Some polycyclic aromatic hydrocarbons are not soluble in methanol or acetone, and their stock standard solutions are prepared in toluene. Methylene chloride should be avoided as a solvent for standards because its high vapor pressure leads to rapid evaporation and concentration changes. Methanol and acetone are not as volatile as methylene chloride, but their solutions must also be handled with care to avoid evaporation. Compounds 10, 11, and 35 in Table 2 are soluble in acetone. Compounds 12, 13, and 20 in

Table 2 are soluble in toluene. If compound purity is certified by the supplier at >96%, the weighed amount can be used without correction to calculate the concentration of the solution (5  $\mu\text{g}/\mu\text{L}$ ). Store the amber vials in a dark cool place.

- 7.7 Primary dilution standard solution. The stock standard solutions are used to prepare a primary dilution standard solution that contains multiple analytes. The recommended solvent for this dilution is acetone. Aliquots of each of the stock standard solutions are combined to produce the primary dilution in which the concentration of the analytes is at least equal to the concentration of the most concentrated calibration solution, that is, 10  $\text{ng}/\mu\text{L}$ . Store the primary dilution standard solution in an amber vial in a dark cool place, and check frequently for signs of deterioration or evaporation, especially just before preparing calibration solutions.
- 7.8 Fortification solution of internal standards and surrogates. Prepare a solution of acenaphthene-D<sub>10</sub>, phenanthrene-D<sub>10</sub>, chrysene-D<sub>12</sub>, and perylene-D<sub>12</sub> in methanol or acetone at a concentration of 500  $\mu\text{g}/\text{mL}$  of each. This solution is used in the preparation of the calibration solutions. Dilute a portion of this solution by 10 to 50  $\mu\text{g}/\text{mL}$  and use this solution to fortify the actual water samples (see Sect. 11.2). Other surrogates, for example, caffeine-<sup>15</sup>N<sub>2</sub> and pyrene-D<sub>10</sub> may be included in this solution as needed (a 100- $\mu\text{L}$  aliquot of this 50  $\mu\text{g}/\text{mL}$  solution added to 1 liter of water gives a concentration of 5  $\mu\text{g}/\text{L}$  of each internal standard or surrogate). Store this solution in an amber vial in a dark cool place.
- 7.9 MS performance check solution. Prepare a 5  $\text{ng}/\mu\text{L}$  solution of DFTPP in methylene chloride. Store this solution in an amber vial in a dark cool place.
- 7.10 Calibration solutions (CAL1 through CAL6). Prepare a series of six concentration calibration solutions in acetone which contain all analytes except pentachlorophenol and toxaphene at concentrations of 10, 5, 2, 1, 0.5, and 0.1  $\text{ng}/\mu\text{L}$ , with a constant concentration of 5  $\text{ng}/\mu\text{L}$  of each internal standard and surrogate in each CAL solution. CAL1 through CAL6 are prepared by combining appropriate aliquots of the primary dilution standard solution (7.7) and the fortification solution (500  $\mu\text{g}/\text{mL}$ ) of internal standards and surrogates (7.8). Pentachlorophenol is included in this solution at a concentration four times the other analytes. Toxaphene CAL solutions should be prepared as separate solutions at concentrations of 250, 200, 100, 50, 25, and 10  $\text{ng}/\mu\text{L}$ . Store these solutions in amber vials in a dark cool place. Check these solutions regularly for signs of deterioration, for example, the appearance of anthraquinone from the oxidation of anthracene.
- 7.11 Reducing agents. Sodium sulfite or sodium arsenite. Sodium thio-sulfate is not recommended as it may produce a residue of elemental sulfur that can interfere with some analytes.

7.12 Fortification solution for optional recovery standard. Prepare a solution of terphenyl-D<sub>14</sub> in methylene chloride at a concentration of 500 µg/mL. An aliquot of this solution may be added (optional) to the extract of the LSE cartridge to check on the recovery of the internal standards in the extraction process.

## 8. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Sample collection. When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 2-5 min). Adjust the flow to about 500 mL/min and collect samples from the flowing stream. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach analytes into water. Automatic samplers that composite samples over time must use refrigerated glass sample containers.

8.2 Sample dechlorination and preservation. All samples should be iced or refrigerated at 4°C from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of a reducing agent. Add 40-50 mg of sodium sulfite or sodium arsenite (these may be added as solids with stirring until dissolved) to each liter of water. Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in unchlorinated water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of pentachlorophenol.

8.3 Holding time. Samples must be extracted within 7 days and the extracts analyzed within 30 days of sample collection.

8.4 Field blanks.

8.4.1 Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sampling site along with the empty sample containers. Return the FRB to the laboratory with filled sample bottles.

8.4.2 When hydrochloric acid is added to samples, use the same procedures to add the same amount to the FRB.

## 9. CALIBRATION

9.1 Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successful, a

continuing calibration check is required at the beginning of each 8 hr. period during which analyses are performed. Additional periodic calibration checks are good laboratory practice.

## 9.2 Initial calibration

- 9.2.1 Calibrate the mass and abundance scales of the MS with calibration compounds and procedures prescribed by the manufacturer with any modifications necessary to meet the requirements in Sect. 9.2.2.
- 9.2.2 Inject into the GC a 1- $\mu$ L aliquot of the 5 ng/ $\mu$ L DFTPP solution and acquire a mass spectrum that includes data for m/z 45-450. Use GC conditions that produce a narrow (at least five scans per peak) symmetrical peak. If the spectrum does not meet all criteria (Table 1), the MS must be retuned and adjusted to meet all criteria before proceeding with calibration. An average spectrum across the GC peak may be used to evaluate the performance of the system.
- 9.2.3 Inject a 1- $\mu$ L aliquot of a medium concentration calibration solution, for example 0.5-2  $\mu$ g/L, and acquire and store data from m/z 45-450 with a total cycle time (including scan overhead time) of 1.5 sec or less. Cycle time should be adjusted to measure at least five or more spectra during the elution of each GC peak.
- 9.2.3.1 Multi-ramp temperature program GC conditions. Adjust the helium carrier gas flow rate to about 33 cm/sec. Inject at 45 $^{\circ}$ C and hold in splitless mode for 1 min. Heat rapidly to 130 $^{\circ}$ C. At 3 min start the temperature program: 130-180 $^{\circ}$ C at 12 $^{\circ}$ /min; 180-240 $^{\circ}$ C at 7 $^{\circ}$ /min; 240-320 $^{\circ}$ C at 12 $^{\circ}$ /min. Start data acquisition at 5 min.
- 9.2.3.2 Single ramp linear temperature program. Adjust the helium carrier gas flow rate to about 33 cm/sec. Inject at 40 $^{\circ}$ C and hold in splitless mode for 1 min. Heat rapidly to 160 $^{\circ}$ C. At 3 min start the temperature program: 160-320 $^{\circ}$ C at 6 $^{\circ}$ /min; hold at 320 $^{\circ}$  for 2 min. Start data acquisition at 3 min.
- 9.2.4 Performance criteria for the medium calibration. Examine the stored GC/MS data with the data system software. Figure 1 shows an acceptable total ion chromatogram.
- 9.2.4.1 GC performance. Anthracene and phenanthrene should be separated by baseline. Benz[a]anthracene and chrysene should be separated by a valley whose height is less than 25% of the average peak height of these two compounds. If the valley between benz[a]anthracene and chrysene exceeds 25%, the GC column requires maintenance. See Sect. 9.3.6.

- 9.2.4.2 MS sensitivity. The GC/MS/DS peak identification software should be able to recognize a GC peak in the appropriate retention time window for each of the compounds in calibration solution, and make correct tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required. See Sect. 9.3.6.
- 9.2.4.3 Lack of degradation of endrin. Examine a plot of the abundance of m/z 67 in the region of 1.05-1.3 of the retention time of endrin. This is the region of elution of endrin aldehyde, a product of the thermal isomerization of endrin. Confirm that the abundance of m/z 67 at the retention time of endrin aldehyde is <10% of the abundance of m/z 67 produced by endrin. If more than 10% endrin aldehyde is observed, system maintenance is required to correct the problem. See Sect. 9.3.6.
- 9.2.5 If all performance criteria are met, inject a 1- $\mu$ L aliquot of each of the other CAL solutions using the same GC/MS conditions.
- 9.2.6 Calculate a response factor (RF) for each analyte and surrogate for each CAL solution using the internal standard whose retention time is nearest the retention time of the analyte or surrogate. Table 2 contains suggested internal standards for each analyte and surrogate, and quantitation ions for all compounds. This calculation is supported in acceptable GC/MS data system software (Sect. 6.14.4), and many other software programs. RF is a unitless number, but units used to express quantities of analyte and internal standard must be equivalent.

$$RF = \frac{(A_X)(Q_{IS})}{(A_{IS})(Q_X)}$$

where:

- $A_X$  = integrated abundance of the quantitation ion of the analyte.  
 $A_{IS}$  = integrated abundance of the quantitation ion internal standard.  
 $Q_X$  = quantity of analyte injected in ng or concentration units.  
 $Q_{IS}$  = quantity of internal standard injected in ng or concentration units.

- 9.2.6.1 For each analyte and surrogate, calculate the mean RF from the analyses of the six CAL solutions. Calculate

the standard deviation (SD) and the relative standard deviation (RSD) from each mean:  $RSD = 100 (SD/M)$ . If the RSD of any analyte or surrogate mean RF exceeds 30%, either analyze additional aliquots of appropriate CAL solutions to obtain an acceptable RSD of RFs over the entire concentration range, or take action to improve GC/MS performance. See Sect. 9.2.7.

- 9.2.7 As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear, second, or third order regression calibration curve.
- 9.3 Continuing calibration check. Verify the MS tune and initial calibration at the beginning of each 8 hr. work shift during which analyses are performed using the following procedure.
- 9.3.1 Inject a 1- $\mu$ L aliquot of the 5ng/ $\mu$ L DFTPP solution and acquire a mass spectrum that includes data for m/z 45-450. If the spectrum does not meet all criteria (Table 1), the MS must be retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.
- 9.3.2 Inject a 1- $\mu$ L aliquot of a medium concentration calibration solution and analyze with the same conditions used during the initial calibration.
- 9.3.3 Demonstrate acceptable performance for the criteria shown in Sect. 9.2.4.
- 9.3.4 Determine that the absolute areas of the quantitation ions of the internal standards and surrogate(s) have not decreased by more than 30% from the areas measured in the most recent continuing calibration check, or by more than 50% from the areas measured during initial calibration. If these areas have decreased by more than these amounts, adjustments must be made to restore system sensitivity. These adjustments may require cleaning of the MS ion source, or other maintenance as indicated in Sect. 9.3.6, and recalibration. Control charts are useful aids in documenting system sensitivity changes.
- 9.3.5 Calculate the RF for each analyte and surrogate from the data measured in the continuing calibration check. The RF for each analyte and surrogate must be within 30% of the mean value measured in the initial calibration. Alternatively, if a second or third order regression is used, the point from the continuing calibration check for each analyte and surrogate must fall, within the analyst's judgement, on the curve from the initial calibration. If these conditions do not exist, remedial action must be taken which may require reinitial calibration.

- 9.3.6 Some possible remedial actions. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.
- 9.3.6.1 Check and adjust GC and/or MS operating conditions; check the MS resolution, and calibrate the mass scale.
  - 9.3.6.2 Clean or replace the splitless injection liner; silanize a new injection liner.
  - 9.3.6.3 Flush the GC column with solvent according to manufacturer's instructions.
  - 9.3.6.4 Break off a short portion (about 1 meter) of the column from the end near the injector; or replace GC column. This action will cause a change in retention times.
  - 9.3.6.5 Prepare fresh CAL solutions, and repeat the initial calibration step.
  - 9.3.6.6 Clean the MS ion source and rods (if a quadrupole).
  - 9.3.6.7 Replace any components that allow analytes to come into contact with hot metal surfaces.
  - 9.3.6.8 Replace the MS electron multiplier, or any other faulty components.

## 10. QUALITY CONTROL

- 10.1 Quality control (QC) requirements are the initial demonstration of laboratory capability followed by regular analyses of laboratory reagent blanks, laboratory fortified blanks, and laboratory fortified matrix samples. The laboratory must maintain records to document the quality of the data generated. Additional quality control practices are recommended.
- 10.2 Initial demonstration of low system background and acceptable particle size and packing. Before any samples are analyzed, or any time a new supply of cartridges is received from a supplier, it must be demonstrated that a laboratory reagent blank (LRB) is reasonably free of contamination that would prevent the determination of any analyte of concern. In this same experiment, it must be demonstrated that the particle size and packing of the LSE cartridge are acceptable. Consistent flow rate is an indication of acceptable particle size distribution and packing.
- 10.2.1 A major source of potential contamination is the liquid-solid extraction (LSE) cartridges which very likely will contain phthalate esters, silicon compounds, and other contaminants that could prevent the determination of method analytes (5).

Generally, phthalate esters will be leached from the cartridges into methylene chloride and produce a variable background that is equivalent to  $<2 \mu\text{g/L}$  in the water sample. If the background contamination is sufficient to prevent accurate and precise analyses, the condition must be corrected before proceeding with the initial demonstration. Figure 2 shows unacceptable background contamination from a poor quality commercial LSE cartridge. The background contamination is the large broad peak, and the small peaks are method analytes present at a concentration equivalent to  $2 \mu\text{g/L}$ . Several sources of LSE cartridges may be evaluated before an acceptable supply is identified.

- 10.2.2 Other sources of background contamination are solvents, reagents, and glassware. Background contamination must be reduced to an acceptable level before proceeding with the next section. In general, background from method analytes should be below the method detection limit.
  - 10.2.3 One liter of water should pass through the cartridge in about 2 hrs with a partial vacuum of about 13 cm (5 in.) of mercury. The extraction time should not vary unreasonably among LSE cartridges.
- 10.3 Initial demonstration of laboratory accuracy and precision. Analyze four to seven replicates of a laboratory fortified blank containing each analyte of concern at a concentration in the range of  $2\text{-}5 \mu\text{g/L}$  (see regulations and maximum contaminant levels for guidance on appropriate concentrations).
- 10.3.1 Prepare each replicate by adding an appropriate aliquot of the primary dilution standard solution, or another certified quality control sample, to reagent water. Analyze each replicate according to the procedures described in Sect. 11 and on a schedule that results in the analyses of all replicates over a period of several days.
  - 10.3.2 Calculate the measured concentration of each analyte in each replicate, the mean concentration of each analyte in all replicates, and mean accuracy (as mean percentage of true value) for each analyte, and the precision (as relative standard deviation, RSD) of the measurements for each analyte. Calculate the MDL of each analyte using the procedures described in Sect. 13.1.2 (1).
  - 10.3.3 For each analyte and surrogate, the mean accuracy, expressed as a percentage of the true value, should be 70-130% and the RSD should be  $<30\%$ . Some analytes, particularly the polycyclic aromatic hydrocarbons with molecular weights  $>250$ , are measured at concentrations below  $2 \mu\text{g/L}$ , with a mean accuracy of 35-130% of true value. The MDLs should be sufficient to detect analytes at the regulatory levels. If these criteria are not

met for an analyte, take remedial action and repeat the measurements for that analyte to demonstrate acceptable performance before samples are analyzed.

- 10.3.4 Develop and maintain a system of control charts to plot the precision and accuracy of analyte and surrogate measurements as a function of time. Charting of surrogate recoveries is an especially valuable activity since these are present in every sample and the analytical results will form a significant record of data quality.
- 10.4 Monitor the integrated areas of the quantitation ions of the internal standards and surrogates in continuing calibration checks (see Sect. 9.3.4). In laboratory fortified blanks or samples, the integrated areas of internal standards and surrogates will not be constant because the volume of the extract will vary (and is difficult to keep constant). But the ratios of the areas should be reasonably constant in laboratory fortified blanks and samples. The addition of 10  $\mu\text{L}$  of the recovery standard, terphenyl- $\text{D}_{14}$  (500  $\mu\text{g}/\text{mL}$ ), to the extract is optional, and may be used to monitor the recovery of internal standards and surrogates in laboratory fortified blanks and samples. Internal standard recovery should be in excess of 70%.
- 10.5 Laboratory reagent blanks. With each batch of samples processed as a group within a work shift, analyze a laboratory reagent blank to determine the background system contamination. Any time a new batch of LSE cartridges is received, or new supplies of other reagents are used, repeat the demonstration of low background described in 10.2.
- 10.6 With each batch of samples processed as a group within a work shift, analyze a single laboratory fortified blank (LFB) containing each analyte of concern at a concentration as determined in 10.3. If more than 20 samples are included in a batch, analyze a LFB for every 20 samples. Use the procedures described in 10.3.3 to evaluate the accuracy of the measurements, and to estimate whether the method detection limits can be obtained. If acceptable accuracy and method detection limits cannot be achieved, the problem must be located and corrected before further samples are analyzed. Add these results to the on-going control charts to document data quality.
- 10.7 Determine that the sample matrix does not contain materials that adversely affect method performance. This is accomplished by analyzing replicates of laboratory fortified matrix samples and ascertaining that the precision, accuracy, and method detection limits of analytes are in the same range as obtained with laboratory fortified blanks. If a variety of different sample matrices are analyzed regularly, for example, drinking water from groundwater and surface water sources, matrix independence should be established for each. A laboratory fortified sample matrix should be analyzed for every 20 samples processed in the same batch.

- 10.8 With each set of field samples a field reagent blank (FRB) should be analyzed. The results of these analyses will help define contamination resulting from field sampling and transportation activities.
- 10.9 At least quarterly, replicates of laboratory fortified blanks should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality (as in Sect. 10.3).
- 10.10 At least quarterly, analyze a quality control sample from an external source. If measured analyte concentrations are not of acceptable accuracy (Sect. 10.3.3), check the entire analytical procedure to locate and correct the problem source.
- 10.11 Numerous other quality control measures are incorporated into other parts of this procedure, and serve to alert the analyst to potential problems.

## 11. PROCEDURE

- 11.1 Setup the extraction apparatus shown in Figure 3A. The reservoir is not required, but recommended for convenient operation. Water drains from the reservoir through the LSE cartridge and into a syringe needle which is inserted through a rubber stopper into the suction flask. A slight vacuum of 13 cm (5 in.) of mercury is used during all operations with the apparatus. The pressure used is critical as a vacuum > than 13 cm may result in poor precision. About 2 hrs is required to draw a liter of water through the system.
- 11.2 Pour the water sample into the 2-L separatory funnel with the stopcock closed. Residual chlorine should not be present as a reducing agent should have been added at the time of sampling. Also the pH of the sample should be about 2. If residual chlorine is present and/or the pH is >2, the sample may be invalid. Add a 100- $\mu$ L aliquot of the fortification solution (50  $\mu$ g/mL) for internal standards and surrogates, and mix immediately until homogeneous. The concentration of these compounds in the water should be 5  $\mu$ g/L.
- 11.3 Flush each cartridge with two 10 mL aliquots of methylene chloride, followed by two 10 mL aliquots of methanol, letting the cartridge drain dry after each flush. These solvent flushes may be accomplished by adding the solvents directly to the solvent reservoir in Figure 3A. Add 10 mL of reagent water to the solvent reservoir, but before the reagent water level drops below the top edge of the packing in the LSE cartridge, open the stopcock of the separatory funnel and begin adding sample water to the solvent reservoir. Close the stopcock when an adequate amount of sample is in the reservoir.
- 11.4 Periodically open the stopcock and drain a portion of the sample water into the solvent reservoir. The water sample will drain into the cartridge, and from the exit into the suction flask. Maintain the packing material in the cartridge immersed in water at all times.

After all of the sample has passed through the LSE cartridge, wash the separatory funnel and cartridge with 10 mL of reagent water, and draw air through the cartridge for 10 min.

- 11.5 Transfer the 125-mL solvent reservoir and LSE cartridge (from Figure 3A) to the elution apparatus (Figure 3B). The same 125-mL solvent reservoir is used for both apparatus. Wash the 2-liter separatory funnel with 5 mL of methylene chloride and collect the washings. Close the stopcock on the 100-mL separatory funnel of the elution apparatus, add the washings to the reservoir and enough additional methylene chloride to bring the volume back up to 5 mL and elute the LSE cartridge. Elute the LSE cartridge with an additional 5 mL of methylene chloride (10-mL total). A small amount of nitrogen positive pressure may be used to elute the cartridge. Small amounts of residual water from the LSE cartridge will form an immiscible layer with the methylene chloride in the 100-mL separatory funnel. Open the stopcock and allow the methylene chloride to pass through the drying column packed with anhydrous sodium sulfate (1-in) and into the collection vial. Do not allow the water layer to enter the drying column. Remove the 100 mL separatory funnel and wash the drying column with 2 mL of methylene chloride. Add this to the extract. Concentrate the extract to 1 mL under a gentle stream of nitrogen. If desired, gently warm the extract in a water bath to evaporate to between 0.5 - 1.0 mL (without gas flow). Do not concentrate the extract to less than 0.5 mL (or dryness) as this will result in losses of analytes. If desired, add an aliquot of the recovery standard to the concentrated extract to check the recovery of the internal standards (see Sect. 10.4).
- 11.6 Analyze a 1-2  $\mu$ L aliquot with the GC/MS system under the same conditions used for the initial and continuing calibrations (Sect. 9.2.3).
- 11.7 At the conclusion of data acquisition, use the same software that was used in the calibration procedure to tentatively identify peaks in retention time windows of interest. Use the data system software to examine the ion abundances of components of the chromatogram. If any ion abundance exceeds the system working range, dilute the sample aliquot and analyze the diluted aliquot.
- 11.8 Identification of analytes. Identify a sample component by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time of the sample component should be within 10 sec of the time observed for that same compound when a calibration solution was analyzed.
  - 11.8.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10

to 50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.

- 11.8.2 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.
- 11.8.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. See Sect. 9.2.4.1. Acceptable resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the average height of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Benzo[b] and benzo[k]fluoranthene are measured as an isomeric pair.
- 11.8.4 Phthalate esters and other background components appear in variable quantities in laboratory and field reagent blanks, and generally cannot be accurately measured at levels below about 2 µg/L. Subtraction of the concentration in the blank from the concentration in the sample at or below the 2 µg/L level is not recommended because the concentration of the background in the blank is highly variable.

## 12. CALCULATIONS

12.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations if unique ions with adequate intensities are available for quantitation. For example, although two listed analytes, dibenz[a,h]anthracene and indeno-[1,2,3,c,d]pyrene, were not resolved with the GC conditions used, and produced mass spectra containing common ions, concentrations (Tables 3-6) were calculated by measuring appropriate characteristic ions.

12.1.1 Calculate analyte and surrogate concentrations.

$$C_x = \frac{(A_x)(Q_{is})}{(A_{is}) RF V}$$

where:

$C_x$  = concentration of analyte or surrogate in µg/L in the water sample.

- $A_x$  = integrated abundance of the quantitation ion of the analyte in the sample.
- $A_{iS}$  = integrated abundance of the quantitation ion of the internal standard in the sample.
- $Q_{iS}$  = total quantity (in micrograms) of internal standard added to the water sample.
- $V$  = original water sample volume in liters.
- $RF$  = mean response factor of analyte from the initial calibration.

12.1.2 Alternatively, use the GC/MS system software or other available proven software to compute the concentrations of the analytes and surrogates from the second or third order regression curves.

12.1.3 Calculations should utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty). Experience indicates that three significant figures may be used for concentrations above 99  $\mu\text{g/L}$ , two significant figures for concentrations between 1-99  $\mu\text{g/L}$ , and one significant figure for lower concentrations.

### 13. METHOD PERFORMANCE

13.1 Single laboratory accuracy and precision data (Tables 3-7) for each listed analyte was obtained at two concentrations with the same extracts analyzed on two different instrument systems. Seven 1-liter aliquots of reagent water containing 2  $\mu\text{g/L}$  of each analyte, and five to seven 1-liter aliquots of reagent water containing 0.2  $\mu\text{g/L}$  of each analyte were analyzed with this procedure.

13.1.2 With these data, method detection limits (MDL) were calculated using the formula:

$$\text{MDL} = S t_{(n-1, 1-\alpha = 0.99)}$$

where:

$t_{(n-1, 1-\alpha = 0.99)}$  = Student's t value for the 99% confidence level with n-1 degrees of freedom

n = number of replicates

S = standard deviation of replicate analyses.

#### 13.2 Problem compounds

13.2.1 The common phthalate and adipate esters (compounds 14, 21, and 23-26), which are widely used commercially, appear in variable quantities in laboratory and field reagent blanks, and generally cannot be accurately or precisely measured at levels below about 2  $\mu\text{g/L}$ . Subtraction of the concentration in the blank from the concentration in the sample at or below the

2 µg/L level is not recommended because the concentrations of the background in blanks is highly variable.

- 13.2.2 Some polycyclic aromatic hydrocarbons are rapidly oxidized and/or chlorinated in water containing residual chlorine. Therefore residual chlorine must be reduced before analysis.
- 13.2.3 In water free of residual chlorine, some polycyclic aromatic hydrocarbons (for example, compounds 9, 12, 13, 20, and 35) are not accurately measured because of low recoveries in the extraction process.
- 13.2.4 Pentachlorophenol No. 40 and hexachlorocyclopentadiene No. 34 may not be accurately measured. Pentachlorophenol is a strong acid and elutes as a broad weak peak. Hexachlorocyclopentadiene is susceptible to photochemical and thermal decomposition.

#### 14. REFERENCES

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TABLE 1. ION ABUNDANCE CRITERIA FOR BIS(PERFLUOROPHENYL)PHENYL PHOSPHINE (DECAFLUOROTRIPHENYLPHOSPHINE, DFTPP)

Mass (M/z)	Relative Abundance Criteria	Purpose of Checkpoint <sup>1</sup>
51	10-80% of the base peak	low mass sensitivity
68	<2% of mass 69	low mass resolution
70	<2% of mass 69	low mass resolution
127	10-80% of the base peak	low-mid mass sensitivity
197	<2% of mass 198	mid-mass resolution
198	base peak or >50% of 442	mid-mass resolution and sensitivity
199	5-9% of mass 198	mid-mass resolution and isotope ratio
275	10-60% of the base peak	mid-high mass sensitivity
365	>1% of the base peak	baseline threshold
441	Present and < mass 443	high mass resolution
442	base peak or >50% of 198	high mass resolution and sensitivity
443	15-24% of mass 442	high mass resolution and isotope ratio

<sup>1</sup>All ions are used primarily to check the mass measuring accuracy of the mass spectrometer and data system, and this is the most important part of the performance test. The three resolution checks, which include natural abundance isotope ratios, constitute the next most important part of the performance test. The correct setting of the baseline threshold, as indicated by the presence of low intensity ions, is the next most important part of the performance test. Finally, the ion abundance ranges are designed to encourage some standardization to fragmentation patterns.

TABLE 2. RETENTION TIME DATA, QUANTITATION IONS, AND INTERNAL STANDARD REFERENCES FOR METHOD ANALYTES.

Compound	Compound Number	Retention Time(min:sec)		Quantitation Ion (m/z)	Internal Standard Reference
		A <sup>a</sup>	B <sup>b</sup>		
<u>Internal standards</u>					
acenaphthene-D <sub>10</sub>	1	4:49	7:45	164	-
phenanthrene-D <sub>10</sub>	2	8:26	11:08	188	-
chrysene-D <sub>12</sub>	3	18:14	19:20	240	-
<u>Surrogate</u>					
perylene-D <sub>12</sub>	4	23:37	22:55	264	3
<u>Target analytes</u>					
acenaphthylene	5	4:37	7:25	152	1
aldrin	6	11:21	13:36	66	2
anthracene	7	8:44	11:20	178	2
atrazine	8	7:56	10:42	200/215	1/2
benz[a]anthracene	9	18:06	19:14	228	3
benzo[b]fluoranthene	10	22:23	22:07	252	3
benzo[k]fluoranthene	11	22:28	22:07	252	3
benzo[a]pyrene	12	23:28	22:47	252	3
benzo[g,h,i]perylene	13	27:56	26:44	276	3
butylbenzylphthalate	14	16:40	18:09	149	2/3
chlordane components					
alpha-chlordane	15	13:44	15:42	375	2/3
gamma-chlordane	16	13:16	15:18	375	2/3
trans nonachlor	17	13:54	15:50	409	2/3
2-chlorobiphenyl	18	4:56	7:55	188	1
chrysene	19	18:24	19:23	228	3
dibenz[a,h]anthracene	20	27:15	25:57	278	3
di-n-butylphthalate	21	10:58	13:20	149	2
2,3-dichlorobiphenyl	22	7:20	10:12	222	1
diethylphthalate	23	5:52	8:50	149	1
di(2-ethylhexyl)phthalate	24	19:19	20:01	149	2/3
di(2-ethylhexyl)adipate	25	17:17	18:33	129	2/3
dimethylphthalate	26	4:26	7:21	163	1
endrin	27	15:52	16:53	81	2/3
fluorene	28	6:00	8:53	166	1
heptachlor	29	10:20	12:45	100/160	2
heptachlor epoxide	30	12:33	14:40	81/353	2

TABLE 2. (Continued)

Compound	Compound Number	Retention Time (min:sec)		Quantitation Ion (m/z)	Internal Standard Reference
		A <sup>a</sup>	B <sup>b</sup>		
2,2',3,3',4,4',6-hepta-chlorobiphenyl	31	18:25	19:25	394/396	3
hexachlorobenzene	32	7:37	10:20	284/286	1/2
2,2',4,4',5,6'-hexa-chlorobiphenyl	33	14:34	16:30	360	2
hexachlorocyclo-pentadiene	34	3:36	6:15	237	1
indeno[1,2,3,c,d]pyrene	35	27:09	25:50	276	3
lindane	36	8:17	10:57	181/183	1/2
methoxychlor	37	18:34	19:30	227	3
2,2',3,3',4,5',6,6'-octachlorobiphenyl	38	18:38	19:33	430	3
2,2',3',4,6-penta-chlorobiphenyl	39	12:50	15:00	326	2
pentachlorophenol	40	8:11	10:51	266	2
phenanthrene	41	8:35	11:13	178	2
pyrene	42	13:30	15:29	202	2/3
simazine	43	7:47	10:35	201	1/2
2,2',4,4'-tetrachloro-biphenyl	44	11:01	13:25	292	2
toxaphene	45	11:30-23:30	13:00-21:30	159	2
2,4,5-trichlorobiphenyl	46	9:23	11:59	256	2
alachlor	47	--	13:19	160	2

<sup>a</sup>Single ramp linear temperature program conditions (Sect. 9.2.3.2).

<sup>b</sup>Multi-ramp linear temperature program conditions (Sect. 9.2.3.1).

TABLE 3. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS OF THE METHOD ANALYTES AT 2  $\mu\text{g/L}$  WITH LIQUID-SOLID EXTRACTION AND THE ION TRAP MASS SPECTROMETER

Compound Number (Table 2)	True Conc. ( $\mu\text{g/L}$ )	Mean Observed Conc. ( $\mu\text{g/L}$ )	Std. Dev. ( $\mu\text{g/L}$ )	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) ( $\mu\text{g/L}$ )
4	5	5.0	0.3	6.0	100	a
5	2	1.9	0.2	11.	95	a
6	2	1.6	0.2	13.	80	a
7	2	1.7	0.1	5.9	85	a
8	2	2.2	0.3	14.	110	a
9	2	1.8	0.2	11.	90	a
10	2	not separated from No. 11; measured with No. 11				
11	2	4.2	0.3	7.1	105	a
12	2	0.8	0.2	25.	40	a
13	2	0.7	0.1	14.	35	a
14	2	2.0	0.3	15.	100	a
15	2	2.0	0.2	10.	100	a
16	2	2.2	0.3	14.	110	a
17	2	2.7	1.0	37.	135	a
18	2	1.9	0.1	5.2	95	a
19	2	2.2	0.1	4.5	110	a
20	2	0.3	0.3	100.	15	a
21	2	2.2	0.3	14.	110	a
22	2	2.3	0.1	4.3	115	a
23	2	2.0	0.3	15.	100	a
24	2	1.9	0.2	11.	95	a
25	2	1.6	0.3	19.	80	a
26	2	1.9	0.2	11.	95	a
27	2	1.8	0.1	5.5	90	a
28	2	2.2	0.2	9.1	110	a
29	2	2.2	0.3	14.	110	a
30	2	2.3	0.2	8.7	115	a
31	2	1.4	0.2	14.	70	a
32	2	1.7	0.2	12.	85	a
33	2	1.6	0.4	25.	80	a
34	2	1.1	0.1	9.1	55	a
35	2	0.4	0.2	50.	20	a
36	2	2.1	0.2	9.5	105	a
37	2	1.8	0.2	11.	90	a
38	2	1.8	0.2	11.	90	a
39	2	1.9	0.1	5.3	95	a
40	8	8.2	1.2	15.	102	a
41	2	2.4	0.1	4.2	120	a
42	2	1.9	0.1	5.3	95	a
43	2	2.1	0.2	9.5	105	a
44	2	1.5	0.1	6.7	75	a
45	25	28.	4.7	17.	112	15.
46	2	1.7	0.1	5.9	85	a
Mean <sup>b</sup>	2	1.8	0.2	15.	91	0.6

<sup>a</sup>See Table 4. <sup>b</sup>Compounds 4, 40, and 45 excluded from the means.

**TABLE 4. ACCURACY AND PRECISION DATA FROM FIVE TO SEVEN DETERMINATIONS OF THE METHOD ANALYTES AT 0.2  $\mu\text{g/L}$  WITH LIQUID-SOLID EXTRACTION AND THE ION TRAP MASS SPECTROMETER.**

Compound Number (Table 2)	True Conc. ( $\mu\text{g/L}$ )	Mean Observed Conc. ( $\mu\text{g/L}$ )	Std. Dev. ( $\mu\text{g/L}$ )	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) ( $\mu\text{g/L}$ )
4	0.5	0.45	0.6	13.	90	0.1
5	0.2	0.13	0.03	23.	65	0.1
6	0.2	0.13	0.03	23.	65	0.1
7	0.2	0.13	0.01	7.7	65	0.04
8	0.2	0.24	0.03	13.	120	0.1
9	0.2	0.14	0.01	7.1	70	0.04
10	0.2	not separated from No. 11; measured with No. 11				
11	0.2	0.25	0.04	16.	62	0.2
12	0.2	0.03	0.01	33.	15	0.04
13	0.2	0.03	0.02	67.	15	0.1
14	0.2	0.32	0.07	22.	160	0.3
15	0.2	0.17	0.04	24.	85	0.2
16	0.2	0.19	0.03	16.	95	0.1
17	0.2	0.17	0.08	47.	85	0.3
18	0.2	0.19	0.03	16.	95	0.1
19	0.2	0.21	0.01	4.8	105	0.04
20	0.2	0.03	0.02	67.	150	0.1
21	0.2	0.48	0.09	19.	240	0.3
22	0.2	0.20	0.03	15.	100	0.1
23	0.2	0.45	0.21	47.	225	0.8
24	0.2	0.39	0.16	41.	195	0.6
25	0.2	0.31	0.16	52.	155	0.6
26	0.2	0.21	0.01	4.8	105	0.04
27	0.2	0.12	0.12	100.	60	0.5
28	0.2	0.21	0.05	24.	105	0.2
29	0.2	0.22	0.01	4.5	110	0.04
30	0.2	0.19	0.04	21.	95	0.2
31	0.2	0.19	0.03	16.	95	0.1
32	0.2	0.16	0.04	25.	80	0.1
33	0.2	0.19	0.03	16.	95	0.1
34	0.2	0.04	0.01	25.	20	0.03
35	0.2	0.04	0.03	75.	20	0.1
36	0.2	0.22	0.02	9.1	110	0.1
37	0.2	0.11	0.01	9.1	55	0.04
38	0.2	0.19	0.05	26.	95	0.2
39	0.2	0.13	0.02	15.	65	0.1
40	0.8	0.78	0.08	10.	97	0.3
41	0.2	0.20	0.004	2.0	100	0.01
42	0.2	0.18	0.005	2.8	90	0.02
43	0.2	0.25	0.04	16.	125	0.2
44	0.2	0.14	0.04	29.	70	0.1
45		not measured at this level				
46	0.2	0.13	0.02	15.	65	0.06
Mean <sup>a</sup>	0.2	0.18	0.04	25.	95	0.16

<sup>a</sup>Compounds 4, 40, and 45 excluded from the means.

TABLE 5. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS OF THE METHOD ANALYTES AT 2  $\mu\text{G/L}$  WITH LIQUID-SOLID EXTRACTION AND A MAGNETIC SECTOR MASS SPECTROMETER

Compound Number (Table 2)	True Conc. ( $\mu\text{g/L}$ )	Mean Observed Conc. ( $\mu\text{g/L}$ )	Std. Dev. ( $\mu\text{g/L}$ )	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) ( $\mu\text{g/L}$ )
4	5	5.7	0.34	6.0	114	a
5	2	1.9	0.22	12.	95	a
6	2	1.6	0.18	11.	80	a
7	2	2.2	0.67	30.	110	a
8	2	2.4	0.46	19.	120	a
9	2	2.2	0.87	40	110	a
10	2	not separated from No. 11; measured with No. 11				
11	2	4.0	0.37	9.3	100	a
12	2	0.85	0.15	18.	43	a
13	2	0.69	0.12	17.	35	a
14	2	2.0	0.20	10.	100	a
15	2	2.2	0.41	19.	110	a
16	2	2.1	0.38	18.	105	a
17	2	1.9	0.10	5.2	95	a
18	2	2.0	0.29	14.	100	a
19	2	2.1	0.32	15.	105	a
20	2	0.75	0.18	24.	38	a
21	2	2.5	0.32	13.	125	a
22	2	2.0	0.23	12.	100	a
23	2	3.5	1.8	51.	175	a
24	2	2.0	0.28	14.	100	a
25	2	1.4	0.16	11.	70	a
26	2	2.9	0.70	24.	145	a
27	2	1.7	0.45	26.	85	a
28	2	2.6	1.0	38.	130	a
29	2	1.2	0.10	8.3	60	a
30	2	2.6	0.42	16.	130	a
31	2	1.5	0.19	13.	75	a
32	2	1.5	0.35	23.	75	a
33	2	1.9	0.17	8.9	95	a
34	2	0.89	0.11	12.	45	a
35	2	0.83	0.072	8.7	42	a
36	2	2.2	0.10	4.5	110	a
37	2	2.0	0.88	44.	100	a
38	2	1.5	0.11	7.3	75	a
39	2	1.6	0.14	8.8	80	a
40	8	12.	2.6	22.	150	a
41	2	2.3	0.18	7.8	115	a
42	2	2.0	0.26	13.	100	a
43	2	2.5	0.34	14.	125	a
44	2	1.6	0.17	11.	80	a
45	25	28.	2.7	10.	112	9.
46	2	1.9	0.073	3.8	95	a
Mean <sup>b</sup>	2	1.8	0.32	16.	88	1.

<sup>a</sup>See Table 6. <sup>b</sup>Compounds 4, 40, and 45 excluded from the means.

TABLE 6. ACCURACY AND PRECISION DATA FROM SIX OR SEVEN DETERMINATIONS OF THE METHOD ANALYTES AT 0.2  $\mu\text{g/L}$  WITH LIQUID-SOLID EXTRACTION AND A MAGNETIC SECTOR MASS SPECTROMETER.

Compound Number (Table 2)	True Conc. ( $\mu\text{g/L}$ )	Mean Observed Conc. ( $\mu\text{g/L}$ )	Std. Dev. ( $\mu\text{g/L}$ )	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) ( $\mu\text{g/L}$ )
4	0.5	0.67	0.07	9.4	134	0.2
5	0.2	0.11	0.03	24.	55	0.1
6	0.2	0.11	0.02	21.	56	0.1
7	0.2	0.14	0.02	17.	70	0.1
8	0.2	0.26	0.08	31.	130	0.3
9	0.2	0.24	0.06	26.	120	0.2
10	0.2	not separated from No. 11; measured with No. 11				
11	0.2	0.40	0.10	25.	100	0.3
12	0.2	0.08	0.02	27.	38	0.1
13	0.2	0.07	0.01	22.	33	0.1
14	0.2	0.33	0.16	48.	160	0.5
15	0.2	0.19	0.02	13.	95	0.1
16	0.2	0.17	0.08	45.	85	0.3
17	0.2	0.19	0.04	18.	95	0.1
18	0.2	0.17	0.02	13.	85	0.1
19	0.2	0.27	0.08	28.	135	0.3
20	0.2	0.09	0.01	15.	46	0.1
21	0.2	1.1	1.2	109.	550	4.
22	0.2	0.18	0.05	30.	90	0.2
23	0.2	0.29	0.17	59.	145	0.6
24	0.2	0.42	0.23	55.	210	0.8
25	0.2	0.32	0.16	50.	160	0.5
26	0.2	0.20	0.09	47.	100	0.3
27	0.2	0.53	0.30	57.	265	1.
28	0.2	0.18	0.03	15.	90	0.1
29	0.2	0.11	0.05	42.	55	0.2
30	0.2	0.33	0.08	26.	165	0.3
31	0.2	0.17	0.01	7.1	85	0.04
32	0.2	0.11	0.04	40.	55	0.2
33	0.2	0.17	0.03	15.	85	0.1
34	0.2	0.05	0.02	35.	24	0.1
35	0.2	0.08	0.06	8.1	40	0.02
36	0.2	0.27	0.03	11.	135	0.1
37	0.2	0.24	0.09	39.	120	0.3
38	0.2	0.15	0.02	12.	75	0.1
39	0.2	0.13	0.02	13.	65	0.1
40	0.8	1.8	0.82	46.	225	3.
41	0.2	0.21	0.07	33.	105	0.2
42	0.2	0.19	0.04	23.	95	0.1
43	0.2	0.27	0.07	27.	135	0.2
44	0.2	0.13	0.03	22.	65	0.1
45		not measured at this level				
46	0.2	0.16	0.04	23.	80	0.12
Mean <sup>a</sup>	0.2	0.21	0.09	28.	102	0.3

<sup>a</sup>Compounds 4, 40, and 45 excluded from the means.

TABLE 7. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS AT 2  $\mu\text{G/L}$  WITH LIQUID-SOLID EXTRACTION AND A QUADRUPOLE MASS SPECTROMETER

Compound Number (Table 2)	True Conc. ( $\mu\text{g/L}$ )	Mean Observed Conc. ( $\mu\text{g/L}$ )	Std. Dev. ( $\mu\text{g/L}$ )	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) ( $\mu\text{g/L}$ )
47	2	2.4	0.4	16.	122	1.0

FIGURE 1. TOTAL ION CHROMATOGRAM OF TWO NANOGRAMS OF ANALYTES AND FIVE NANOGRAMS OF SURROGATES AND INTERNAL STANDARD

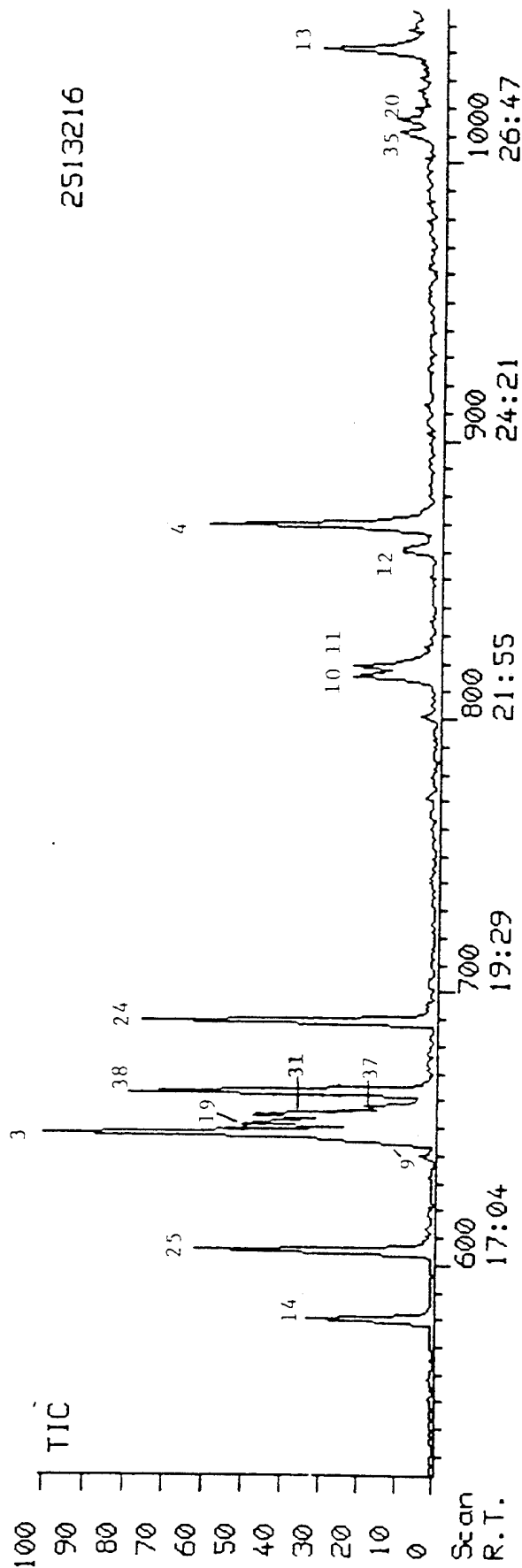
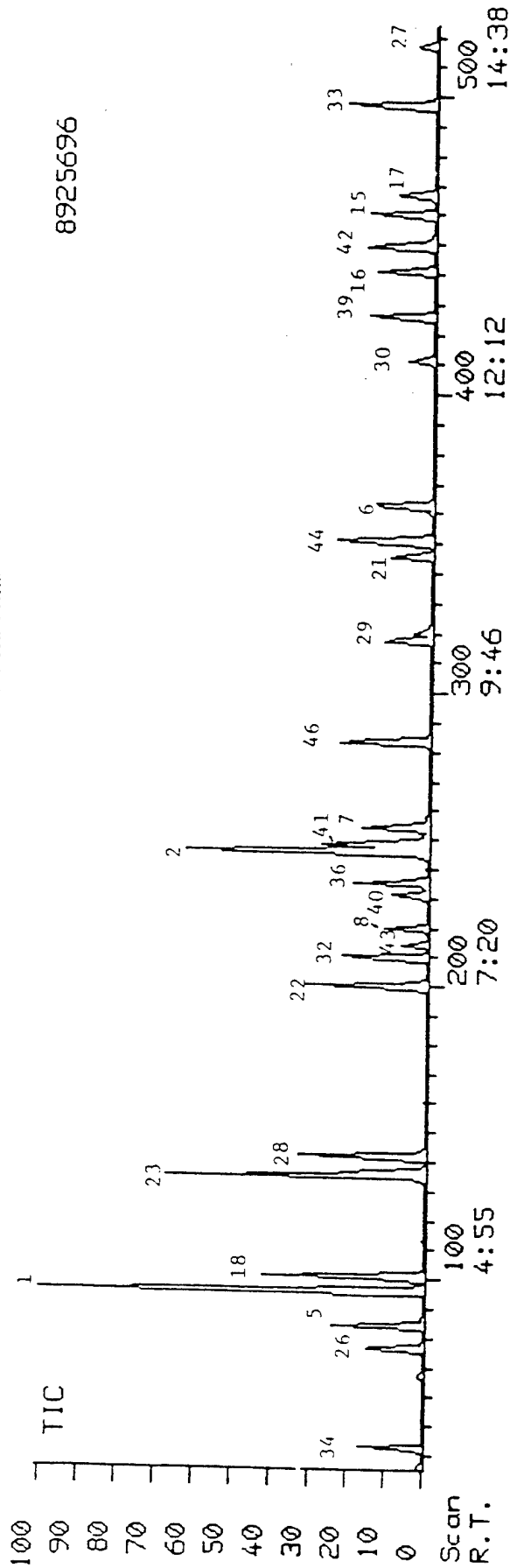
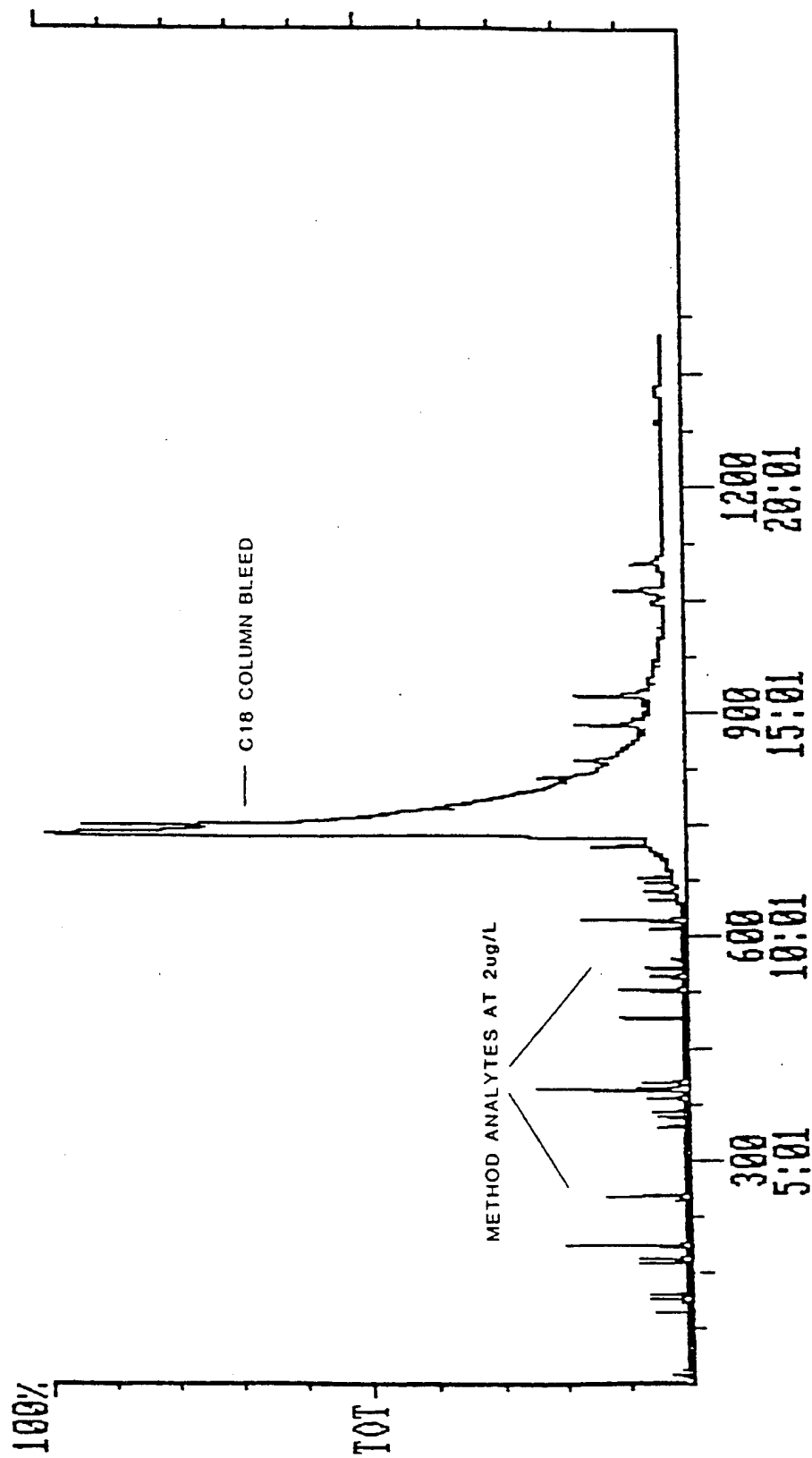
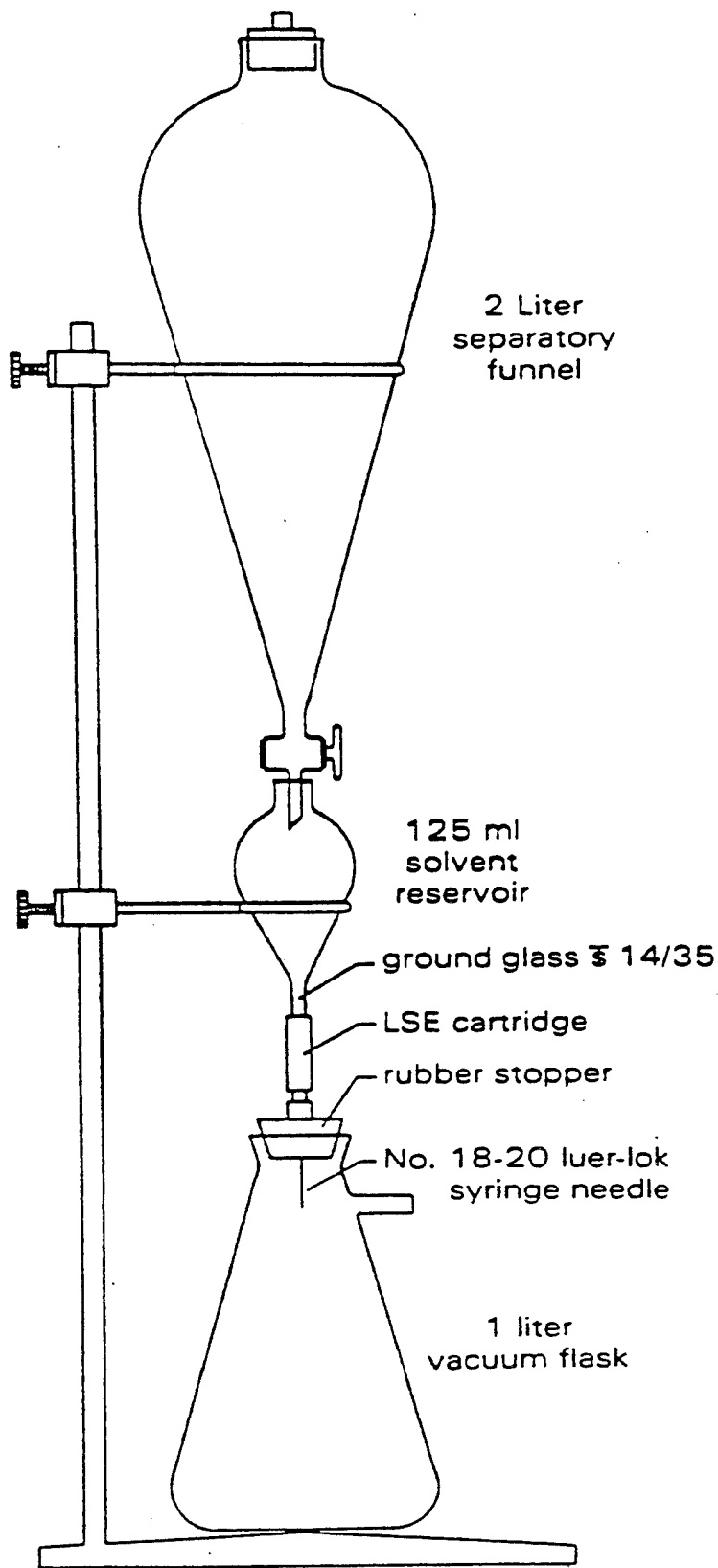
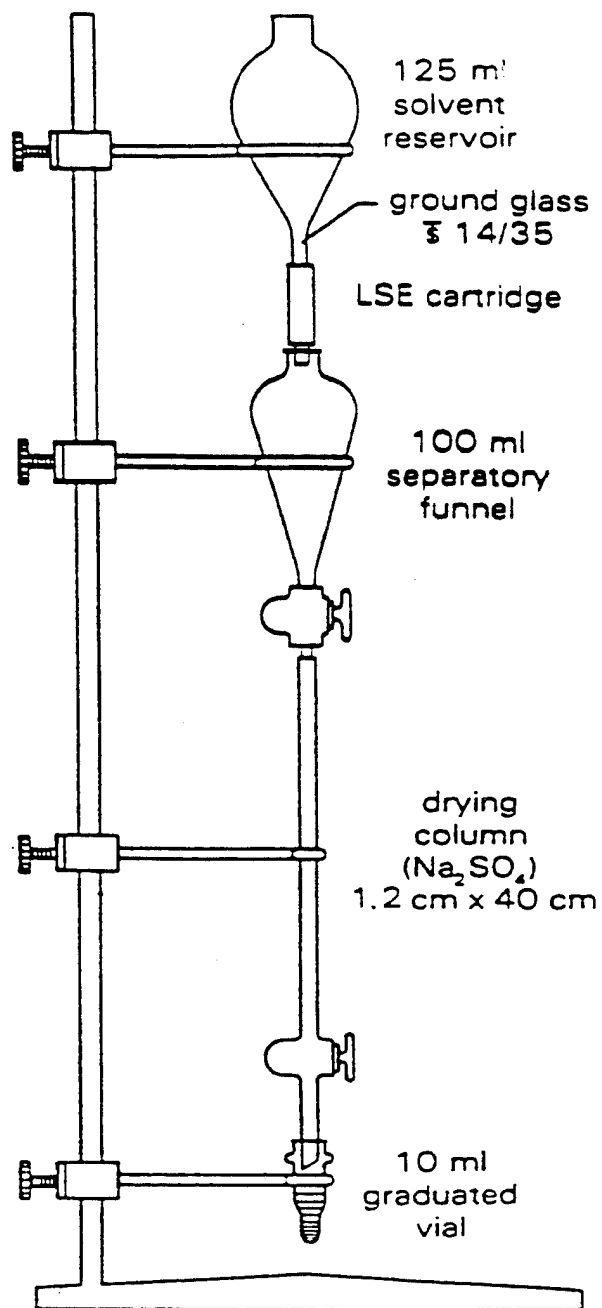


FIGURE 2. TOTAL ION CHROMATOGRAM FROM A LABORATORY BLANK WITH AN UNACCEPTABLY HIGH BACKGROUND





A. Extraction apparatus



B. Elution apparatus

FIGURE 3