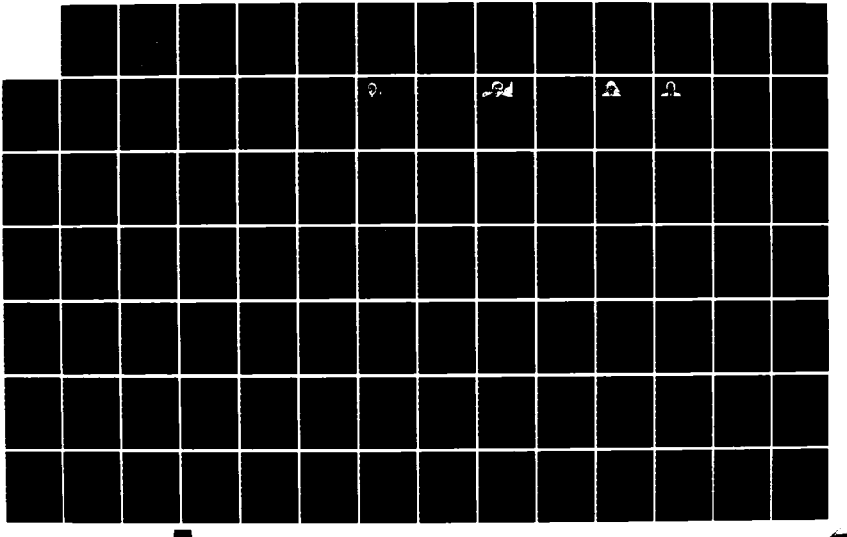
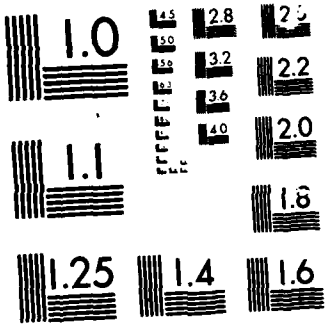
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Installation Restoration Program

Final Report
Phase II, Stage 1 - Problem Confirmation Study
Norton Air Force Base
San Bernardino, California

Volume II - Appendices

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Prepared For:

United States Air Force
Occupational and Environmental Health Laboratory (OEHL)
Brooks Air Force Base, Texas

July 1985

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WESTON

Norton AFB

PHASE II, STAGE 1 REPORT

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K. Laboratory Analytical Reports - Raw Chemical Data

L. Federal and State Drinking Water and Human Health Standards Applicable in the State of California

ADA 165514

REPORT DOCUMENTATION PAGE

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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

A Phase II Stage 1 Field evaluation was conducted at Norton Air Force Base, San Bernardino, California, under the auspices of the U.S. Air Force Installation Restoration Program (IRP). The evaluation was accomplished by Roy F. Weston, Inc. (WESTON) as authorized by Task Order 0021 of Air Force Contract No. F 33615-80-D-4006. Fifteen sites of potential environmental concern, grouped into six waste management zones, were evaluated. A total of 22 monitor wells were installed and groundwater samples were obtained from each well. Soil samples were obtained for chemical analysis from 12 soil borings. Samples of surface water, bottom sediments and fish tissues were obtained from three ponds. All chemical analyses were accomplished in accordance with Standard USEPA analytical methods. Based on the sampling and analyses performed, levels of contamination were found in soils or

20 DISTRIBUTION/AVAILABILITY OF ABSTRACT: UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT ☐ DTIC USERS ☐	21 ABSTRACT SECURITY CLASSIFICATION: Unclassified
22a NAME OF RESPONSIBLE INDIVIDUAL	22b TELEPHONE NUMBER (Include Area Code) / 22c OFFICE SYMBOL

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19. Abstract (continued)

groundwater at seven of the 15 sites evaluated which warrant further investigation and potential remedial actions. Recommendations were made as to appropriate follow-up site evaluation work at these seven sites.

APPENDIX A

ACRONYMS, DEFINITIONS, NOMENCLATURE

UNITS OF MEASUREMENT

Accession For
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AFFF	Air Force Firefighting Foam
ALC	Air Logistics Center
ASTM	American Society for Testing and Materials
Alluvium	Sedimentary materials deposited in an environment of flowing surface waters.
Aquifer	Zone beneath the earth's surface capable of producing water for a well.
Artesian	Groundwater conditions in which pressure within an aquifer causes groundwater to rise in a well above the top of the aquifer, and sometimes above ground surface.
AVGAS	Aviation gas
BEE	Bio-Environmental Engineering
CERCLA	Comprehensive Environmental Response Compensation and Liability Act of 1980
cm/s	centimeters per second
DEQPPM	Defense Environmental Quality Program Policy Memorandum
DoD	Department of Defense
ft/day	feet per day
groundwater divide	A theoretical dividing line in the water-table on each side of which the water-table slopes away, forming a boundary between separate groundwater basins
HARM	Hazard Assessment Rating Methodology
HNu	A common brand name for a volatile organic vapor detection meter
hydraulic gradient	Rate of change in pressure or head in the groundwater over a given distance of flow
IRP	Installation Restoration Program
IWTP	Industrial Waste Treatment Plant

MAC	Military Airlift Command
MAG	Military Airlift Group
MAW	Military Airlift Wing
ug/g	Micrograms per gram (equivalent to parts per million in solids)
ug/L	Micrograms per liter (equivalent to parts per billion in water)
mg/L	Milligrams per liter (equivalent to parts per million in water)
mgd	Million gallons per day
M.S.	Master of Science degree
MSL	Mean Sea Level datum
N	North
NoAFB	Norton Air Force Base
O&G	Oil and grease
OEHL	Occupational and Environmental Health Laboratory
P.G.	Registered Professional Geologist
Ph.D.	Doctor of Philosophy degree
POL	Petroleum Oil and Lubricants
ppb	Parts per billion (equivalent to ug/L in water
ppm	Parts per million (equivalent to mg/L in water
RCRA	Resource Conservation and Recovery Act of 1976

Unconsolidated Sediments	Sediments that are uncemented and thus include interconnected void space (primary porosity) that allows storage and transmission of groundwater.
USAF	United States Air Force
USEPA	United States environmental Protection Agency
VOA	Volatile Organic and Aromatic Hydrocarbon Compounds
Water-table	The level below which earth materials are saturated with water

APPENDIX B

SCOPE OF WORK - TASK ORDER 0021-01

Installation Restoration Program

Phase IIB Field Evaluation

Norton AFB, California

Revision No. 1 to Description of Work

I. Description of Work:

A. Purpose

To determine if environmental contamination has resulted from waste disposal practices at Norton AFB CA and to provide estimates of the magnitude and extent of contamination, should contamination be found.

B. Background

The presurvey report (mailed under separate cover) and Phase I IRP report (mailed under separate cover) incorporate all background, description and site numbers for this task.

C. Technical Effort

1. Golf Course Waste Management Zone. The Industrial Waste Lagoon (Site No. 1), Waste Pit No. 2 (Site No. 3), Waste Pit No. 1 (Site No. 4), Fire Protection Training Area No. 2 (Site No. 5) Landfill No. 1 (Site No. 10) and Waste Pit No. 3 (Site No. 12) shall be combined into one zone for purposes of the Confirmation Stage survey.

a. Site No. 1

(1) Peform a Ground Penetrating Radar (GPR) survey to define the areal limits of the industrial waste lagoons and to determine the presence or absence of drum-like targets in the site.

(2) Obtain one bottom sediment and one surface water sample from each of the two golf course ponds and from the golf course irrigation reservoir.

(3) Obtain three fish samples each from the three ponds listed immediately above (nine fish). The three fish from each pond will be composited to prepare one tissue sample from each pond (three total) for analysis.

b. Site No. 3

Conduct a GPR survey to define the areal limits of the waste pit.

c. Site No. 4

Conduct a GPR survey to define the areal limits of the waste pit.

Modification highlights are underscored.

d. Site No. 5

Drill six exploratory soil borings in and around the perimeter of the area. Sample the soil continuously to a depth of six feet. Retain samples as two-foot composites for analysis.

e. Site No. 10

Conduct a GPR survey to define the areal limits of the landfill.

f. Site No. 12

Conduct a GPR survey to define the areal limits of the waste pit and to determine the presence or absence of drum-like targets in the site.

g. General Zone Procedures

(1) Install a maximum of nine groundwater monitoring wells the Golf Course Waste Management Zone. Specific locations for monitoring wells shall be determined by the site-specific actions outlined above. All wells shall be downgradient of the sites being monitored. Obtain one water sample for each well.

(2) Perform the analyses shown in Table 1 on the samples obtained from the zone.

2. Landfill Waste Management Zone. Landfill No. 2 (Site No. 2) and the Fuel Sludge Disposal Area (Site No. 11) shall be combined into one zone for purposes of the Confirmation Stage Survey.

a. Install a maximum of three groundwater monitoring wells in the Landfill Waste Management Zone. The wells shall be downgradient of the site. Obtain one water sample from each well.

b. Perform the following analyses on one groundwater sample from each well: TOC, TOX, VOA; Li, Pb, Cr, Ni, Cd, As, Zn, Cu, Hg; oil and grease, specific conductance. Analysis for pH shall be performed in the field.

3. Underground Waste Oil Storage Tank (Site No. 6)

a. Install a maximum of two monitoring wells downgradient of the tank. Obtain one water sample from each well.

b. Analyze one sample from each well for the following parameters: Oil and grease, lead, VOA, TOC and TOX.

4. IWTP Waste Mangement Zone. The IWTP Sludge Drying Beds (Site No. 7), the IWTP Sludge Disposal Area (Site No. 13), Drummed Waste Storage Area No. 3 (Site No. 17), the IWTP Discharge Ditch, and the Waste Fuel and Solvent Sump shall be combined into one zone for the purposes of the Confirmation Stage Survey.

TABLE 1: SUMMARY OF ANALYSES FOR GOLF COURSE WASTE MANAGEMENT ZONE SAMPLES

SAMPLE NO.	TYPE[1]	TOC	TOX	VOA	METALS[2]	OIL & GREASE	CONDUCITIVTY	Ph[3]	PHENOL	MEK	CYANIDE
MW-1	GW	X	X	X	X	X	X	X	X	X	
MW-2	GW	X	X	X	X	X	X	X	X	X	
MW-3	GW	X	X	X	X	X	X	X	X	X	
MW-4	GW	X	X	X	X	X	X	X	X	X	
MW-5	GW	X	X	X	X	X	X	X			
MW-6	GW	X	X	X	X	X	X	X			
MW-7	GW	X	X	X	X	X	X	X			
MW-8	GW	X	X	X	X	X	X	X	X		X
MW-9	GW	X	X	X	X	X	X	X			
TB-1:1-5	SS			X							
TB-2:1-5	SS			X							
TB-3:1-5	SS			X							
TB-4:1-5	SS			X							
TB-5:1-5	SS			X							
TB-6:1-5	SS			X							
POND 1-W	SW	X	X	X	X	X	X	X	X	X	
POND 1-S	BS			X					X	X	
POND 1-F	FT				X						
POND 2-W	SW	X	X	X	X	X	X	X	X	X	
POND 2-S	BS			X					X	X	
POND 2-F	FT				X						
POND 3-W	SW	X	X	X	X	X	X	X	X	X	
POND 3-S	BS			X					X	X	
POND 3-F	FT				X						

[1]GW= Groundwater, SS=Subsurface Soil, SW= Surface Water, BS= Bottom Sediment, FT=Fish Tissue Composite

[2]Metals analyses include Pb, Cr, Ni, Cd, As, Zn, Cu, Hg

[3]Determined in the field.

MW=Monitoring Well
TB=Test Boring

a. Install six soil borings around the Waste Fuel and Solvent Sump. Sample each boring continuously to a depth of ten feet, and retain the soil samples as two-foot composites for analysis. Analyze each soil sample (composite) for VOA and Phenols.

b. Install three monitoring wells downgradient and one well upgradient of the sites. Obtain one water sample from each well.

c. Analyze one sample from each well for the following parameters: TOC, TOX, VOA; Pb, Cr, Ni, Cd, As, Zn, Cu, Hg; oil and grease; specific conductance. Analysis for pH shall be performed in the field.

5. Waste Pit No. 4 (Site No. 14)

a. Install a maximum of two monitoring wells downgradient of the site. Obtain one water sample from each well.

b. Analyze one sample from each well for the following parameters: TOC, TOX, VOA; Pb, Cr, Ni, Cd, As, Zn, Cu, Hg; oil and grease; specific conductance. Analysis for pH shall be performed in the field.

6. AAVS/DAVA Evaporation Basins (Site No. 16)

a. Install three monitoring wells downgradient and one well upgradient of the site. Obtain one groundwater sample from each well.

b. Analyze one sample from each well for the following parameters: TOC, TOX, VOA; Pb, Cr, Ni, Cd, As, Zn, Cu, Hg, CN; oil and grease. Specific conductance and pH shall be performed in the field.

7. Abandoned Wells

Any time an abandoned water supply well is discovered during the monitoring effort, the location of the well shall be accurately marked on a base map. The map shall be provided to base Civil Engineering at the end of the survey.

D. Well Installation and Cleanup

1. All wells installed during this survey shall be constructed of two-inch diameter Schedule 40 PVC pipe with 20 feet of PVC screen. Wells shall be 60 feet deep. Each well boring shall be sampled for stratigraphic purposes at five-foot intervals using a split-spoon sampler and standard penetration tests. Each pilot boring shall be logged in accordance with U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) procedures (furnished under separate cover). Logs of borings shall be included in the final report referenced in Item VI below. Location and elevation of each well shall be surveyed, and location and elevation recorded on the appropriate zone or site map.

2. A maximum of 22 wells shall be installed during the Confirmation Stage survey (this task order).

3. Each well installation shall be cleaned following the completion of the well. Drill cuttings shall be removed and the general area cleaned.

E. Sampling and Analysis

Sampling, maximum holding time and preservation of samples shall strictly comply with the following references: Examination of Water and Wastewater, 15th ed. (1980), pp. 35-42; ASTM, Part 31, pp. 72-82, (1976), Method D-3370; and Methods for Chemical Analysis of Waters and Wastes, EPA Manual 600/4-79-020, pp. xiii to xix (1979). Limits of detection are specified in Table 2.

F. Data Review

Results of sampling and analysis shall be tabulated and incorporated in the monthly R&D status report and forwarded to the USAF OEHL for review as soon as they become available as specified in Item VI below.

G. Reporting

1. A draft final report delineating all findings of this field investigations shall be prepared and forwarded to the USAF OEHL as specified in Item VI below for Air Force review and comment. This report shall include a discussion of the regional hydrogeology, well logs of projects wells, data from water level surveys, ground penetrating radar (GPR) survey results, water quality analysis results, available geohydrologic cross sections, ground water surface and gradient maps, laboratory quality assurance information and any available vertical and horizontal flow vectors. The report shall follow the USAF OEHL supplied format (mailed under separate cover).

2. Estimates shall be made of the magnitude, extent and direction of movement of contaminants discovered. Potential environmental consequences of discovered contamination must be identified. Where survey data are insufficient to properly determine or estimate the magnitude, extent and direction of movement of discovered contaminants, specific recommendations, fully justified, shall be made for additional efforts required to properly evaluate contamination migration and included in a separately bound appendix to the draft final report (see H below).

3. Specific requirements, if any, for future groundwater and surface water monitoring must be identified.

H. Cost Estimates

The contractor shall provide estimates for all additional work recommended to permit proper determination of contaminants. The recommendations provided shall include all efforts required to determine the magnitude and direction of movement of discovered contaminants along with an estimate of the time required to accomplish the proposed effort. This information shall be provided in a separately bound appendix to the draft final report.

II. Site Location and Dates:

Norton AFB CA
USAF Clinic/SGPB
Dates to be established

III. Base Support: None

IV. Government Furnished Property: None

V. Government Points of Contact:

1. Dr Dee Ann Sanders
USAF OEHL/CVT
Brooks AFB TX 72835
(512) 536-2158
AV 240-2158

2. Lt Col Dean Nelson
HQ MAC/SGPB
Scott AFB IL 62225
(618) 256-2306
AV 638-2306

3. Capt Cedric Daksla
USAF Clinic/SGPB
Norton AFB CA 92409
(714) 382-3061
AV 876-3061

VI. In addition to sequence numbers 1, 5 and 11 listed in Atch 1 to the contract, which are applicable to all orders, the sequence number listed belo are applicable to this order. Also shown are data applicable to this order.

Seq Nr	Block 10	Block 11	Block 12	Block 13	Block 14
4	One/R	84JUL10	84AUG14	84NOV13	*

*A minimum of two draft reports will be required. After incorporating Air Force comments concerning the first draft report, the contractor shall supply the USAF OEHL with a second draft report. The report will be forwarded to the applicable regulatory agencies for their comments. The contractor shall supply the USAF OEHL with 20 copies of each draft report, and 50 copies plus the original camera ready copy of the final report.

TABLE 2: DETECTION LIMITS

The limits of detection for chemical analyses are specified below.

TOC - 1 mg/L

TOX - 5 µg/L

Volatile Organic Aromatics (VOA) - Detection limits as specified for compounds listed in EPA Method 602.

Lithium - 1 mg/L

Lead - 20 µg/L water, 0.2 µg/g soil

Chromium - 50 µg/L water, 0.5 µg/g soil

Nickel - 100 µg/L water, 1.0 µg/g soil

Cadmium - 10 µg/L water, 0.1 µg/g soil

Arsenic - 10 µg/L water, 0.1 µg/g soil

Zinc - 50 µg/L water, 0.5 µg/g soil

Copper - 50 µg/L water, 0.5 µg/g soil

Mercury - 1 µg/L water, 0.01 µg/g soil

Oils and Greases (IR method) - 0.1 mg/L

Conductance - 1 µmho

Phenols in soil - 0.1 µg/g

Volatile Organic Aromatics in soils - report values down to 1 µg solvent per gram of soil.

Cyanide - 10 µg/L

APPENDIX C

BIOGRAPHIES OF KEY PERSONNEL



Frederick Bopp III, Ph.D., P.G.

Registration

Registered Professional Geologist in the State of Indiana

Fields of Competence

Groundwater resources evaluation; hydrogeologic evaluation of sanitary landfills and other waste disposal sites; detection and abatement of groundwater pollution; digital modeling of groundwater flow and solute transport; statistical analysis of geological and geochemical data; geochemical prospecting; estuarine geology and geochemistry; trace metal and aqueous geochemistry.

Experience Summary

Seven years experience in hydrogeology and geochemistry, involving such activities as: assessment of subsurface water and soil contamination; development of contamination profiles; evaluation of remediation actions for groundwater quality restoration; quantitative chemical analysis of water and soil; ore assay and ore body evaluation; drilling supervisor; hydrogeologic assessment; pollution detection and abatement; estuarine pollution analysis; application of flow and solute transport computer models; computer programming; project management; teaching environmental geology and geochemistry.

Credentials

B.A., Geology—Brown University (1966)

M.S., Geology—University of Delaware (1973)

Ph.D., Geology—University of Delaware (1979)

Sigma Xi, The Scientific Research Society of North America

Geological Society of America, Hydrology Division

National Water Well Association, Technical Division

American Association for the Advancement of Science

Estuarine Research Federation: Atlantic Estuarine Research Society

Employment History

1979-Present	WESTON
1977-1979	U.S. Army Corps of Engineers Waterways Experiment Station
1976-1977	University of South Florida Department of Geology
1970-1976	University of Delaware Department of Geology
1974-1976	Earth Quest Associates President and Principal Partner
1974 (Summer)	WESTON
1966-1970	United States Navy Commissioned Officer

Key Projects

Project manager on seven task orders for environmental assessment services at United States Air Force facilities in nine states.

Task manager for a Superfund site evaluation in Ohio.

Site manager for drum recovery operations in Pennsylvania and New Jersey.

Project manager for site assessments of oil and fuel spills in four states.

Project manager for closure plan development at a hazardous waste landfill in New Jersey.

Definition and abatement of groundwater contamination from chemical manufacturing in Delaware.

Flow and solute transport digital model of a heavily-pumped regional aquifer in southern New Jersey.

Definition and abatement of groundwater contamination from chemical manufacturing in the Denver area.

Hydrogeologic impact assessment of on-land dredge spoil disposal in coastal North Carolina.

Geochemical prospecting and ore body analysis in Arizona.

Professional Profile

Definition and abatement of groundwater contamination from a hazardous waste site in northern New England.

Definition and abatement of groundwater contamination from plating and foundry wastes in eastern Pennsylvania.

Operational test and evaluation of new naval mine ordinances in southern Florida.

Publications

"Metals in Estuarine Sediments: Factor Analysis and Its Environmental Significance". *Science*, 214 (1981): 441-443.

"The Remobilization of Trace Metals from Suspended Sediments Entering the Delaware Estuary". Presented at the 27th Annual Meeting, Southeastern Section, Geological Society of America, Chattanooga, Tennessee, April 1978.

"Trace Metals in Delaware Bay Sediments and Oysters". Presented at the International Conference on Heavy Metals in the Environment, Toronto, Canada, October 1975.



James S. Smith, Ph.D.

Fields of Competence

Analytical laboratory management; organic chemistry; mass spectrometry, GC/MS/DS, high and low resolution, chemical ionization and special techniques; gas chromatography including capillary column techniques; high performance liquid chromatography (HPLC); the uses of NMR, IR, UV, visible, inorganic analyses, electrochemical, thermal techniques and surface methodologies (SEM, ESCA, SIMS) to solve industrial problems; the development of quality control measures in analytical protocols; the testing of laboratory safety methodologies; innovation of new analytical techniques and methods to solve industrial, product liability, production and environmental problems.

Experience

Eleven years experience in the supervision of an analytical group involved in solving all types of industrial problems including environmental, product safety, production, research and development. The main emphasis was on the innovative development of analytical methods utilizing instrumental technologies. In-depth experience in the organic chemicals, inorganic chemicals polymer, fiber, tire, solvent, fluorine chemicals, coke and coal tar industries. Numerous scientific presentations. Contributor to three Chemical Manufacturers Association Task Groups: Environmental Monitoring, Groundwater, and Hazardous Waste Response Center.

Taught general chemistry, analytical chemistry, organic chemistry, and instrumental analysis for four years at Eastern Michigan University and the University of Illinois.

Credentials

B.A. Chemistry - Williams College (1960)

Ph.D. Organic Chemistry - Iowa State University (1964)

Postdoctoral Organic Chemistry - University of Illinois (1966)

Postdoctoral Mass Spectroscopy - Cornell University (1969)

American Chemical Society

American Society for Testing Materials

American Society of Mass Spectroscopists

Employment History

1981 - Present	WESTON
1969 - 1981	Allied Chemical Corporation Corporate Research Center Supervisor
1966 - 1968	Eastern Michigan University Assistant Professor of Chemistry
1965 - 1966	University of Illinois Visiting Lecturer in Chemistry

Key Projects

Directed analytical group for five years of intensive sampling and analysis of a toxic insecticide. Analyses involved soil, air, water, sludge, blood, bile, feces, urine, animal feed, and plant samples to detect the compound at the low parts-per-billion level. The project involved rapid development of new and accurate analytical methods.

Developed an industrumental analytical laboratory consisting of trace environmental analyses, gas chromatography, high performance liquid chromatography, mass spectrometry, surface analyses, X-ray photoelectron spectroscopy and nuclear magnetic resonance spectroscopy including the design and manufacture of instrument modifications, purchasing instruments, and hiring of key personnel.

Isolated, identified, and developed a method of analysis for a colored impurity on a bulk chemical product. Synthesized the colorant for proof of identification and as a standard for future analysis. Proved the mechanism of the development of the color from the packaging material. Designed new specifications eliminating the problem.

Conducted corporate plant environmental laboratory QA/QC audits including the development of a corporate QA/QC manual.

Provided an inexpensive and accurate method of analysis of lead for a manufacturing plant effluent. A published methodology in kit form was modified for plant personnel use to measure soluble and total lead in a waste stream without use of excessive manpower or capital. QA/QC procedures were included as well as the use of performance samples.

Supervision of analytical technological advances that lead to either patents and new products in the fields of coal tar chemicals, food packaging and transformer manufacturing.

Professional Profile

Publications

Smith, J., A. Weston, and C. Wezwick, "Tire Cord Emission Studies, Conclusion," The International Society of Industrial Yarn Manufacturers, Savannah, Georgia, 3-4 November 1977.

Hanrahan, J., E. McCarthy, D. Richton, J. Smith, and A. Weston, "Identification of an Interfering Compound is the Determination of Dimethylnitrosamine by Gas Chromatography-Mass Spectrometry," 26th Annual Conference on Mass Spectrometry and Allied Topics, St. Louis, Missouri, 28 May to 2 June 1978.

Brozowski, E., D. Jerolamon, D. Richton, D. Smith, J. Smith, and A. Weston, "Industrial Applications of Chemical Ionization with the Ammonium Ion," 26th Annual Conference on Mass Spectrometry and Allied Topics, St. Louis, Missouri, 28 May to 2 June 1978.

Mueller, B.W., L. Palmer, G. Rebyak, and J. Smith, "Analysis of Alpha and Beta Naphthalene Sulfonic Acids by High Performance Liquid Chromatography," North Jersey A.C.S. Chromatography Discussion Group, Nutley, New Jersey, 14 March 1979.

French, C., L. Palmer, and J. Smith, "Analysis of Polymer Oligomers by High Performance Liquid Chromatography," Middle Atlantic Regional A.C.S. Meeting, West Long Branch, New Jersey, 19-23 March 1979.

Burkitt, D. and J. Smith, "A Simple Chrcmatographic Modification Providing for Rapid Interchange of Capillary and Packed Columns," Middle Atlantic Regional A.C.S. Meeting, West Long Branch, New Jersey, 19-23 March 1979.

Brozowski, E., D. Jerolamon, D. Richton, D. Smith, and J. Smith, "A Convenient Method for the Evaporation of Solvent in the Priority Pollutant Program," Middle Atlantic Regional A.C.S. Meeting, West Long Branch, New Jersey, 19-23 March 1979.

Mady, N., D. Smith, J. Smith, and C. Wezwick, "The Analysis of Kepone in Biological Samples," Proceedings of the 9th Materials Research Symposium, Gaithersburg, Maryland, 10-12 April 1978.

Mueller, B., L. Palmer, and J. Smith, "A High Performance Liquid Chromatographic Method for the Analysis of Bis-phenol-A and Its Impurities," Middle Atlantic Regional A.C.S. Meeting, West Long Branch, New Jersey, 19-23 March 1979.

Gabriel, M., J. Hanrahan, and J. Smith, "A Sensitive Method for the Quantitative Analysis of Pyridine at the Low PPM Level," Middle Atlantic Regional A.C.S. Meeting, West Long Branch, New Jersey, 19-23 March 1979.

Burkitt, D., J. Hanrahan, and J. Smith, "Analysis of Hexachloroacetone and Hexafluoroacetone in Industrial Wastewater," Proceedings of the A.S.T.M. Committee D-19 Symposium, "The Measurement of Organic Pollutants in Water and Wastewater," Denver, Colorado, 19 to 20 June 1978.

Brozowski, E., D. Burkitt, M. Gabriel, E. McCarthy, J. Hanrahan, and J. Smith, "A Simple, Sensitive Method for the Quantitative Analysis of Carbon Tetrachloride and Chloroform in Water at the Parts Per Billion Level," Proceedings of the 9th Materials Research Symposium, Gaithersburg, Maryland, 10-12 April 1978.



John A. Williams, Jr.

Fields of Competence

Geologic and geophysical investigations; geological and groundwater sampling techniques and instrumentation technology; design, operation, and evaluation of geophysical survey, equipment, testing and analysis of aquifers, and groundwater pollution.

Experience Summary

Three years experience in geologic and geophysical investigtions including subsurface profiling using Ground Penetrating Radar (GPR), electrical resistivity and electromagnetic conductivity for numerous private and government facilities; groundwater sampling and aquifer pump tests, six years experience in bathymetric, hydrographic and biological studies.

Credentials

A. S., Marine Technology - Cape Fear Technical Institute (1975)

B. S., Earth Science (Geology) - West Chester State College (1983)

Certified Ground Penetrating Radar Operator

Certified NAUI/PADDI Scuba Diver

Geological Society of America

Employment History

1982 - Present	WESTON
1980-1982	Environmental Resources Management, Inc.
1977-1980	WESTON
1976-1977	Highway Service Marineland
1975-1976	Lawler, Matusky, Skelly Engineers

Key Projects

Coordinated and supervised geophysical investigations to locate buried drums and to delineate the boundaries of a buried waste lagoon for a scrap recovery plant in Rhode Island.

Geophysical field investigation to locate buried trenches and waste lagoons for a government facility in California.

Geophysical field investigation, well installation and sample collection to determine the distribution of leachate, and the extent of contamination in a heavily-used aquifer in New York.

Geophysical investigation to define the lateral and vertical effect of fill deposition for a facility in Massachusetts.

Soils investigation to determine the extent of contamination from old waste lagoons and fire training areas for a government facility in Arizona.

Hydrogeologic investigation for a scrap recovery facility in western Pennsylvania.

Responsible for deploying benthic and water quality sampling gear and an electronic navigation system for a dredge spoils disposal study in Lake Erie.

Geophysical investigation (ground penetrating radar and electrical resistivity) to locate buried drums and delineate trench boundaries for a government facility in Ohio.

Professional Profile



Thomas A. Drew

Fields of Competence

Hydrogeologic investigation and evaluation of sanitary and hazardous waste disposal sites; design and installation of groundwater monitoring systems; subsurface sampling for soil and groundwater quality investigations; testing and analysis of aquifers; geophysical surveys.

Experience Summary

Three years experience in hydrogeologic evaluation of groundwater contamination cases and proposed waste disposal sites; design and installation of monitor wells; collection and interpretation of groundwater quality data, downhole geophysical logging and earth resistivity surveys; collections and analysis of well pumping test data and well field evaluation.

Credentials

B.A., Geology—Augustana College (1979)

M.A., Geology/Hydrogeology—University of Missouri (1981)

National Water Well Association, Technical Division

Employment History

1982-Present	WESTON
1981-1982	Woodward-Clyde Consultants
1981	Department of Natural Resources Jefferson City, Missouri
1979-1981	University of Missouri Department of Geological Science

Key Projects

Field Team Leader for the hydrogeologic evaluation of the Chem-Dyne site, a Superfund hazardous waste disposal facility in Ohio. Directly responsible for proper construction and development of monitor wells, supervision of subcontracted drilling services and on-scene subcontract administration, acquisition of groundwater samples from monitor wells for analysis of U.S. EPA priority pollutants, establishing chain-of-custody documentation for the samples, surveying wells for location and elevation, and analyzing hydrogeologic data.

Field Supervisor for the evaluation of a municipal county landfill potentially contaminating groundwater resources in southern Maryland. Directly responsible for proper monitor well construction and development, downhole geophysical logging of deep boreholes, earth resistivity surveys for plume location, surveying of monitor wells and sampling points, sampling of groundwater and surface water for analysis of a broad spectrum of potential analytes, conducting pump tests on installed monitor wells, and collecting and analyzing a wide range of hydrogeologic data.

Conducted investigations at an industrial site in Minnesota to determine the magnitude and extent of pesticide contamination in the soil and groundwater. Supervised monitor well installation, soil and groundwater sampling.

Participated in geologic and hydrogeologic studies for proposed hazardous waste disposal site in Kansas. Investigations at the waste site included soil and rock classification, piezometer installation, field permeability testing, and ground and surface water sampling.

Performed a hydrogeologic study in Monroe and Ralls Counties, Missouri involving the analysis of groundwater from glacial till and bedrock aquifers, permeability evaluations of various soil associations, and the preparation of geologic and structural maps. Also involved in hydrogeologic investigations of current landfill sites in Missouri for the purpose of developing hydrogeologic criteria for the siting of future waste disposal facilities.

Professional Profile

Bruce W. Benyish

Fields of Competence

Broad range of experience involving subsurface exploration programs, supervising the construction of monitoring and production wells, conducting sustained pump tests, hydrogeologic data analysis, and technical report preparation.

Credentials

B.S., Earth Science—Pennsylvania State University (1979)

National Water Well Association, Technical Division

Employment History

1983-Present	WESTON
1983	Suburban Water Testing Labs, Inc.
1980-1982	Gilbert/Commonwealth
1980	General Battery Corporation

Key Projects

Served as a field geologist at hazardous waste sites during the USAF Installation Restoration Programs. Responsibilities included supervision of the installation of monitoring wells, procurement of representative soil samples for documentation, and collection of groundwater samples for analysis of various organic and inorganic chemical constituents. Participated in the preparation of Installation Restoration Program Reports.

Participated in the development of water well fields for municipal water supplies. Performed aerial photograph fracture trace analysis to assist in selecting optimum water well sites. Supervised sustained well pumping tests and analyzed data to determine safe yields. Prepared hydrogeologic reports incorporating pumping test data and geologic literature. Submitted reports to regulatory agencies to obtain groundwater withdrawal permits.

Supervised the drilling of foundation test borings and monitoring well installations pertaining to an Environmental Impact Assessment (EIA) feasibility study for a coal-fired power plant. Participated in the preparation of EIA Report.

Participated in water table aquifer decontamination programs. Scope of involvement included supervising the withdrawal of hazardous sludges from pre-existing wells, pumping, sampling, and treating contaminated groundwater, and scheduling shipment of non-treatable groundwater to certified waste disposal sites.

APPENDIX D

SOIL BORING LOGS

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: B-1/B-1A
OWNER: USAF
LOCATION: Fire Training Area No. 2
ADDRESS: Norton AFB
TOTAL DEPTH 6.0'
SURFACE ELEVATION:
WATER LEVEL:
DRILLING COMPANY: Stang Hydronics
DRILLING METHOD: Auger
DATE DRILLED 10/4/83
DRILLER: DS
HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	6	0-2	Black ASH, fine sand, silt sized	50
				7		some gravel, damp	
				24		Auger drilled up insulator pieces	
				13		HNU down hole - 15 ppm rec. 1.4	
						hit cobbles/off set 2' B-1A	
		2	SS	13	2-4	Black ASH, fine sand, silt sized	130
				21		some gravel, damp rec. 1.1'	
				22		HNU down hole 7 ppm	
				12		No explosimeter readings detected	
		3	SS	21	4-6	Black SAND, fine to medium,	50
				16		tr. silt, damp rec. 0.7'	
				16		HNU down hole-50 ppm	
				17		No explosimeter reading detected.	

* A.S.T.M. D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: B-2/B-2A
LOCATION: Fire Training Area No. 2
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang
DRILLER: DS
LOG BY: BWB/TD

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH 6.0'
WATER LEVEL: _____
DRILLING METHOD: Auger
HELPER: HM
DATE DRILLED: 11/4/83

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	2	0-2	Black ASH, fine sand, silt, sized	60
				10		damp rec. 0.8'	
				14		HNU down hole - 180 ppm	
				16		No explosimeter reading detected.	
		2	SS	17	2-4	Black ASH, fine sand, silt sized	150
				22		damp; Pushed gravel; poor recovery	
				25		rec. 0.4'	
				25		HNU downhole - 75 ppm	
		3	SS	15	4-6	Grayish brown SAND, fine to coarse,	60
				16		damp	
				40		rec 1.0'	
				21		HNU down hole-0 ppm	
						No explosimeter reading detected.	

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: B-3/B-3A OWNER: USAF
LOCATION: Fire Training Area No. 2 ADDRESS: Norton AFB
TOTAL DEPTH 6.0'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/5/84
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	8	0-1.5	Black ASH, fine sand, silt sized	30
				23		with pieces of insulators	
				23		HNU downhole - 0 ppm	
		2	SS	22	1.5-3	Black ASH, fine to medium sand, silt	130
				19		sized, some fine to coarse gravel,	
				27		damp,	
						HNU down hole-3ppm	
		3	SS	30	3-3.7	Black SAND, medium to coarse,	130
					3.7-4.5	Some fine to coarse gravel, damp	
						hammer pushing gravel, rec. 0.7	
						hit cobbles @ 3.7', offset	
						3' and augered to 4.5' HNU down hole 3 ppm	
		4	SS	17	B-3A		
				33	4.5-6.0	Black SAND, medium to coarse, some fine gravel,	130
				50		tr silt, damp	
						HNU down hole 5 ppm	

* A.S.T.M. D1586

WESTON
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DRILLING LOG

WELL NUMBER: B-4 OWNER: USAF
LOCATION: Fire Training Area No. 2 ADDRESS: Norton AFB
TOTAL DEPTH 6.5'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/5/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	2	0-1.5	Black ASH, fine to medium sand,	150
				17		silt sized, tr. coarse gravel, damp,	rec. 1.0'
				16		HNU downhole - 100 ppm	
						No explosimeter reading detected	
		2	SS	7	1.5-3	Black ASH, fine to medium sand, silt	150
				11		sized, damp; Grayish brown SAND,	
				16		medium to coarse, tr. fine sand, tr. silt	
						damp,	rec. 1.3'
						HNU down hole - 30 ppm	
						No explosimeter reading detected	
		3	SS	13	3.5-5	Grayish brown SAND, medium to coarse,	140
				17		some coarse gravel, tr. fine sand	
				14		tr. silt, damp	rec. 0.8'
						HNU down hole 20 ppm	
						No explosimeter reading detected	
		4	SS	8	5-6.5	Grayish brown SAND, medium	200
				15		to coarse, tr. fine sand, moist	
				26			rec. 1.3

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: B-5 OWNER: USAF
LOCATION: Fire Training Area No. 2 ADDRESS: Norton AFB
TOTAL DEPTH 6.0'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/5/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	3	0-1.5 Black ASH, fine to coarse sand,	70
				11	silt, sized, damp rec. 1.1'	
				16	HNU downhole - 5 ppm	
					No explosimeter reading detected	
		2	SS	10	1.5-3 Black ASH, fine to coarse	130
				12	sand, silt sized, damp rec. 1.1'	
				8	HNU downhole - 8 ppm	
					No explosimeter reading detected	
		3	SS	7	3-4.5 Black ASH, fine to coarse	180
				11	sand, silt sized, damp poor	
				12	recovery pushed gravel rec. 0.5'	
					HNU downhole - 200 ppm	
					Explosimeter 20% LEL 18% oxygen	
		4	SS	4	4.5-6 Black ASH, fine to coarse sand,	150
				2	silt sized, wet, has insulators	
				3	pieces; Grayish brown SAND,	
					medium, some fine, moist	
					HNU downhole - 200 ppm	
					Explosimeter 18% LEL 21% oxygen	

* A.S.T.M. D1586

WESTON
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DRILLING LOG

WELL NUMBER: B-6 OWNER: USAF
LOCATION: Fire Training Area No. 2 ADDRESS: Norton AFB
TOTAL DEPTH 6.0'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/5/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	3	0-1.5 Dr. brown ASH, fine to medium	60
				20	sand, silt size, tr. fine to coarse	
				5	gravel, damp rec. 1.1	
					HNU downhole - 10 ppm No explosimeter reading detected	
		2	SS	5	1.5-3 Black ASH, fine to medium	120
				4	sand, silt sized, tr. fine to coarse	
				1	gravel, moist rec. 1.3	
					HNU downhole - 20 ppm No explosimeter reading detected	
		3	SS	3	3-4.5 Black ASH, fine to medium sand	100
				3	silt sized; Grayish brown SAND,	
				4	fine to medium, some silt, moist rec. 1.4	
					HNU downhole - 200 ppm	
					Explosimeter 50% LEL 15% oxygen	
		4	SS		4.5-6 Black ASH, fine to medium sand	100
					silt, sized, moist, with fibrous	
					material	
					HNU downhole - 150 ppm	
					Explosimeter 10% LEL 20% oxygen	
					HNU and Explosimeter measurements obtained by	
					placing probes in boring hole	

* ASTM D1586

WESTON
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DRILLING LOG

WELL NUMBER: BB-1 OWNER: USAF
LOCATION: IWTP Waste Fuel and Solvent Sump ADDRESS: Norton AFB
TOTAL DEPTH 5.5'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/3/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	6	0-2	Grayish brown SAND, fine, damp	-
				7		micaceous rec. 1.3'	
				10			
				17			
		2	SS	14	2-4	Grayish brown SAND, fine to	3
				23		coarse, tr. fine gravel, tr. silt,	
				26		damp, micaceous rec. 0.3'	
		3	SS		4-5.5	Grayish brown SAND, medium	3
						to coarse, tr. fine gravel, tr. fine	
						sand, damp, micaceous rec. 1.0'	

* ASTM D1586

WESTON

DRILLING LOG

WELL NUMBER: BB-1A
OWNER: USAF
LOCATION: Waste Fuel and Solvent Sump
ADDRESS: Norton AFB
TOTAL DEPTH 9.5'
SURFACE ELEVATION:
WATER LEVEL:
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 11/3/83
DRILLER: DS
HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1		5	2-3.5	Grayish brown SAND, medium	1
				7		to coarse, tr. fine gravel, damp	
				10		rec. 1.1	
		2	SS	7	3.5-5	Grayish brown SAND, medium	1
				12		to coarse, some fine gravel,	
				21		tr. fine sand, damp to moist	
		3	SS	9	5-6.5	Grayish brown SAND, medium	2
				22		to coarse, tr. fine sand, moist	
				19		rec. 0.6'	
		4	SS	4	6.5-8	Grayish brown SAND, medium, tr.	0
				7		fine gravel, moist, grades more	
				4		fine sand on bottom rec. 1.1'	
		5	SS	6			0
				4			
				3			

* A.S.T.M. D1586

SHEET 1 OF 1

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: BB-2 OWNER: USAF
LOCATION: IWTP Waste Fuel and Solvent Sump ADDRESS: Norton AFB
TOTAL DEPTH 10.5'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/4/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	2/3	0-1.5	Grayish brown SAND, tr. medium	0
				5		sand, damp, micaceous, rec. 1.4'	
		2	SS	10/10	1.5-3	Grayish brown SAND, medium to coarse,	0
				12		tr. fine gravel, tr. fine sand, damp,	
						micaceous	
		3	SS	16/-	3-4.5	Grayish brown SAND, medium to coarse,	0
				-		tr. gravel, tr. fine sand, micaceous,	
						hammer hitting auger blow count void	
		4	SS	10/21	4.5-6	Grayish brown sandy fine, some fine to	0
				30		medium sand, damp rec. 0.8'	
					6-7.5	Grayish brown SAND, medium to coarse, tr.	2
		5	SS	7/10		fine gravel, tr. fine sand moist,	
				5		Fe stains rec 1.1'	
					7.5-9	Grayish brwon SAND, medium to coarse,	2
		6	SS	3/7		tr. fine gravel, tr. fine sand, moist,	
				12		Fe stains, grades to dr. gray	
						silty sand rec 1.3'	
		7	SS	2/7	9-10.5	Dr. gray silty SAND, moist, micaceous;	3
				14		Olive gray SAND, fine to medium, tr. coarse	
						sand, tr. silt moist.	

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: BB-3
LOCATION: IWTP Waste Fuel and Solvent Sump
SURFACE ELEVATION: _____
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 11.0'
WATER LEVEL: _____
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 11/3/83
DRILLER: DS
HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	4/4	0-1.5	Grayish brown SAND, fine, damp	1
				7		rec. 1.4'	
		2	SS	5/8	1.5-3	Grayish brown SAND, fine to coarse,	1
				13		tr. fine to coarse gravel, damp	
						rec. 1.1'	
		3	SS	9/25	3-4.5	Gray GRAVEL, fine to coarse,	1
				27		some cobbles (broken) rec. 0.5'	
		4	SS	11/9	4.5-6	Grayish brown SAND, medium, tr. gravel,	1
				7		tr. coarse sand, damp	
						Fe stains rec. 1.2'	
		5	SS	9/5	8-9.5	Grayish brown SAND, medium to coarse,	2
				5		tr. fine gravel, tr. fine sand, damp,	
						fe stains, with olive brown silty sand lenses	
		6	SS	-/-	9.5-11	Grades from Grayish brown SAND, fine to	2
				-		medium, to Dr. olive brown SAND, fine,	
						some silt, moist	
						Hammer hitting auger, blow count void, rec. 1.1'	
						Offset 3' to obtained missed 6-8'	
		7	SS	9/11	6-8	Grayish brown SAND, fine to medium moist	1
				14		Fe stains	

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: BB-4 OWNER: USAF
LOCATION: IWTP Waste Fuel and Solvent Sump ADDRESS: Norton AFB
TOTAL DEPTH 10.5'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/3/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	5/7	0-1.5 Olive brown, gray brown SAND, fine,	1
				9	tr. medium sand, tr. silt, damp rec. 1.4'	
		2	SS	5/15	1.5-3 Gray brown SAND, fine to mediu,	1
				18	tr. coarse sand, damp, Fe stains rec. 1.3'	
		3	SS	15/14	3-4.5 Grayish brown SAND, fine to coarse, some	1
				20	fine gravel, damp Fe stains, rec. 1.1'	
		4	SS	10/12	4.5-6 Grayish brown, SAND, fine to coarse, damp	1
				20	Fe stains rec. 1.3'	
		5	SS	13/21	6-7.5 Grayish brown SAND, fine to coarse,	1
				23	some broken gravel and cobbles,	
					damp rec. 1.0'	
		6	SS	6/10	7.5-9 Grayish brown SAND, fine to coarse,	2
				9	some broken gravel and cobbles,	
					damp; Dr. olive gray silty SAND,	
					fine, moist, micaceous	
		7	SS	8/15	9-10.5 Grayish brown SAND, fine to medium,	4
				20	moist; Dr. gray sand SILT, tr. clay,	
					low plastic moist, micaceous	

* A.S.T.M D1586

WESTON

DRILLING LOG

WELL NUMBER: BB-5 OWNER: USAF
LOCATION: IWTP Waste Fuel and Solvent Sump ADDRESS: Norton AFB
TOTAL DEPTH 11.0'
SURFACE ELEVATION: WATER LEVEL:
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/4/83
DRILLER: DS HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
		1	SS	7/13	0.5-2 Grayish brown SAND, fine, tr.	-
				10	medium to coarse sand, damp rec. 1.1'	
		2	SS	7/6	2-3.5 Grayish brown SAND, fine, tr.	4
				14	medium to coarse sand, damp, rec. 1.2'	
		3	SS	7/9	3.5-5 Lt. gray SAND, medium to coarse,	125
				16	tr. fine gravel, damp rec. 1.3'	
		4	SS	9/16	5-6.5 Olive brown SAND, fine to medium	75
				20	tr. silt, moist, very strong solvent	
					smell, using respirator rec. 1.2'	
		5	SS	7/15	6.5-8 Olive brown SAND, coarse, tr. fine	100
				16	gravel, tr. medium sand, moist rec. 1.0'	
		6	SS	5	8-9.5 Olive brown SAND, coarse, moist;	130
				11/15	Dr. olive gray silty SAND, fine, moist	
					micaceous, Fe stains rec. 1.2'	
		7	SS	7/9	9.5-11 Olive gray SAND, fine to medium, tr. silt,	90
				11	wet, micaceous, rec. 0.8'	
					at 11' HNU - 150 ppm	
					at 11' explosimeter - None detected	
					Above measurements for HNU and explosimeter	
					obtained by placing probes in boring hole	

* ASTM D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

BB-6/BB-6A

WELL NUMBER: BB-6B
LOCATION: IWTP Waste Fuel and Solvent Sump
SURFACE ELEVATION: _____
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 11.5'
WATER LEVEL: _____
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 10/4/83
DRILLER: DS
HELPER: HM
LOG BY: BWB/TD

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S HNU (ppm)
-6		1	SS	7	0-1.5 Grayish brown SAND, fine,	1
				7	tr. medium sand, tr. silt, damp	
				6	@ 1.5' hitting rock, cement	
					offset	
					B6A	
B-6A		1	SS	11	2-2.5 Grayish brown SAND, fine	1
				-	to coarse, tr. silt, damp rec. 0.5'	
				-	split spoon refusal @ 2.5	
					Augered to 3.5' attempted	
					split spoon - refused	
					Augered to 4.5' - still hitting cobbles	
					OFFSET 4' E drilled to	
					2.5' B-6B	
B-6B		1	SS	17	2.5-4 Grayish brown SAND, medium	1
				24	to coarse, some fine to coarse	
				27	gravel, moist rec. 1.3'	

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

BB-6/BB-6A

WELL NUMBER: BB-6B OWNER: USAF

LOCATION: IWTP Waste Fuel and Solvent Sump ADDRESS: Norton AFB

TOTAL DEPTH 10.5'

SURFACE ELEVATION: WATER LEVEL:

DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 10/4/83

DRILLER: DS HELPER: HM

LOG BY: BWB/TD

SKETCH MAP

NOTES:

	DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	S/S	HNU (ppm)
							Augered to 5' hit cobbles		
							Augered to 6'		
B-6B			2	SS	9	6-7.5	Grayish brown SAND, medium		0
					21		to coarse, tr. fine gravel, tr. fine		
					22		sand, damp, micaceous rec. 1.0		
			3	SS	7/11	7.5-9	Grayish brown SAND, medium		0
					16		to coarse, tr. fine sand, damp		
							rec. 1.1		
			4	SS	7	9-10.5	Grayish brown SAND, medium		0
					20		some gravel, tr. fine sand, moist;		
					18		Olive gray silty SAND, fine moist		
							to wet, micaceous; Olive gray		
							SAND, medium, tr. fine sand,		
							tr. silt, moist		

* A.S.T.M. D1586

APPENDIX E

MONITOR WELL LOGS AND

COMPLETION SUMMARIES

WESTON DESIGNERS & CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-1
LOCATION: Golf Course Site 1 Ind. Waste Lagoon
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang Hydronics
DRILLER: D.S.
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 46.0'
WATER LEVEL: 26.8'
DRILLING METHOD: Auger
DATE DRILLED: 12/2/83
HELPER: DCL

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2 Dk olive brown SAND, fine, tr. medium sand, some silt, damp to moist.
5		2	SS	13/16 13	4-5.5 Grayish brown SAND, fine, tr. medium sand, some silt, damp to moist rec. 1.3'
10		3	SS	12/13 14	9-10.5 Grayish brown SAND, fine, tr. medium sand, tr. silt, damp with some iron stains rec. 1.5'
					12.5-14.5 drilling hard
15		4	SS	15 50/2"	15-16.5 Lt. gray fine gravelly SAND, fine, tr. medium to coarse sand, damp rec. 0.8'

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-1 OWNER: USAF
LOCATION: Golf Course Site 1 Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH 46.0'
SURFACE ELEVATION: WATER LEVEL: 26.8'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/2/83
DRILLER: DS HELPER: DCL

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	20/40 23	19-20.5 Lt. gray SAND, fine to medium, some fine gravel, damp rec. 1.2'
		6	SS	24/27 36	24-25.5 Reddish brown SAND, fine to coarse, tr. fine gravel, damp to moist rec. 1.4' water @ 28.4'
25					
		7	SS	13/20 25	29-30.5 Olive gray SAND, fine to coarse, some silt, saturated; Dr. gray silty SAND, fine, damp to moist, micaceous rec. 1.2'
30					
		8	SS	5/11 30	34-35.5 Olive gray SAND, fine to medium, tr. coarse, tr. silt, saturated; Black SAND, fine, tr. medium sand, tr. to some silt, wet, black silt layer @ 34.5 to 34.6' rec. 1.4
35					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-1 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 1
Ind. Waste Lagoon TOTAL DEPTH 46.0'
SURFACE ELEVATION: _____ WATER LEVEL: 26.8'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/3/83
DRILLER: HM HELPER: WH

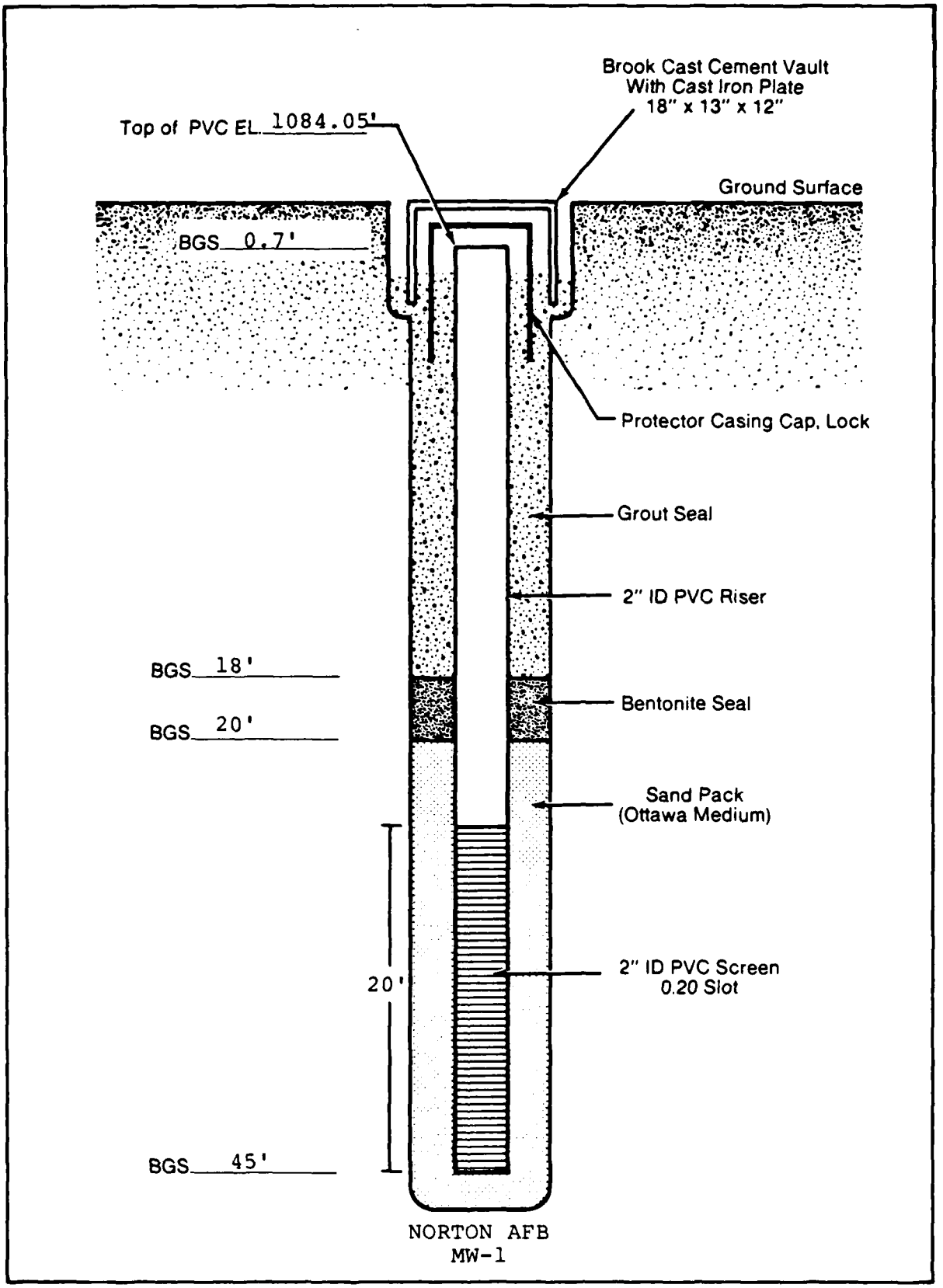
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	3/12/38	39-40.5 Gray SAND, fine to coarse, tr. fine gravel, tr. silt, saturated rec. 1.2
		10	SS	3/3/10	44-45.5 Gray SAND, fine, tr. medium sand, tr. silt, saturated, advanced auger to 46' Dr. gray silt was on bottom of plug
45					Note: No detected HNU readings in any split spoon samples

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1084.05'
Ground Surface
BGS 0.7'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 18'
Bentonite Seal
BGS 20'
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.20 Slot
BGS 45'
NORTON AFB
MW-1

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-2 OWNER: USAF
LOCATION: Golf Course Site 1 Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH 90.5'
SURFACE ELEVATION: WATER LEVEL: 30'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/7/83
DRILLER: DS HELPER: HM

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-0.5 Grayish brown SAND, fine tr. medium sand, tr. silt, damp
		2	SS	16/15 22	4-5.5 Grayish brown SAND, fine tr. medium sand, tr. silt, damp rec. 1.2'
5					
		3	SS	21/27 50/3"	9-10.5 Grayish brown SAND, medium to coarse, some cobbles, some fine to coarse gravel, tr. fine sand, damp to moist rec. 1.2'
10					
		4	SS	11/17 26	14-15.5 Grayish brown SAND, medium, tr. fine gravel, tr. coarse sand, some fine sand, damp
15					
20					

* ASTM D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-2 OWNER: USAF
LOCATION: Golf Course, Site 1, Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH: 90.5'
SURFACE ELEVATION: WATER LEVEL: 30'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/7/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	15/22/43	19-20.5 Grayish brown SAND, medium to coarse, tr. fine gravel, tr. fine sand, damp rec. 1.2
		6	SS	12/20/30	24-25.5 Olive gray SAND, fine, some silt, damp micaceous rec. 1.1
25					bit dry @ 29'
					water @ 30'
		7	SS	6/11/34	29-30.5 Olive gray silty SAND, fine, tr. clay, wet to saturated, micaceous
30					
		8	SS	4/10/11	34-35.5 Dr. gray SAND, fine to medium, tr. coarse sand, tr. silt, saturated, micaceous
35					
40					

* ASTM D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-2 OWNER: USAF
LOCATION: Golf Course Site 1 Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH 90.5'
SURFACE ELEVATION: WATER LEVEL: 30'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/7/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	5/10/27	39-40.5 Dr. gray clayey SILT, some fine sand, low plastic, saturated, micaceous; Dr. gray silty SAND, tr. clay, saturated, micaceous rec. 1.2
45		10	SS	6/16/72/2"	44-45.5 Olive gray silty SAND, fine, damp, micaceous sand is very dense rec. 1.0'
50		11	SS	12/75	49-50.5 Olive gray silty SAND, fine, saturated, micaceous rec. 0.9'
					@ 54' running sand in augers to 42': putting water down hole
55					While attempting to pull out in-hole hammer, the cable snapped; hammer lost, must offset and redrill
60					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-2 OWNER: USAF
LOCATION: Golf Course Site 1 Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH 90.5'
SURFACE ELEVATION: WATER LEVEL: 30'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/22/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
50					Offset 2' redrilled to 52', sampled
		12	SS	3/2	52-53.5 Grayish brown SAND, medium to coarse,
				12	tr. fine gravel, tr. fine sand
					tr. silt, saturated rec. 1.2
		13	SS	6/14	54-55.5 Olive gray SAND, fine to medium
55				13	tr. fine gravel, tr. coarse sand,
					saturated, rec. 1.3
					56-57' drilling hard
		14	SS	7/10	59-60.5 Dr. olive gray fine sandy SILT,
				55	tr. clay, low plastic, saturated
60					rec. 0.6
		15	SS	26/60	64-65.5 Dr. gray silty SAND, fine,
				28/1"	moist to wet, micaceous
65					
70					

* A.S.T.M. D1586

WESTON

DRILLING LOG

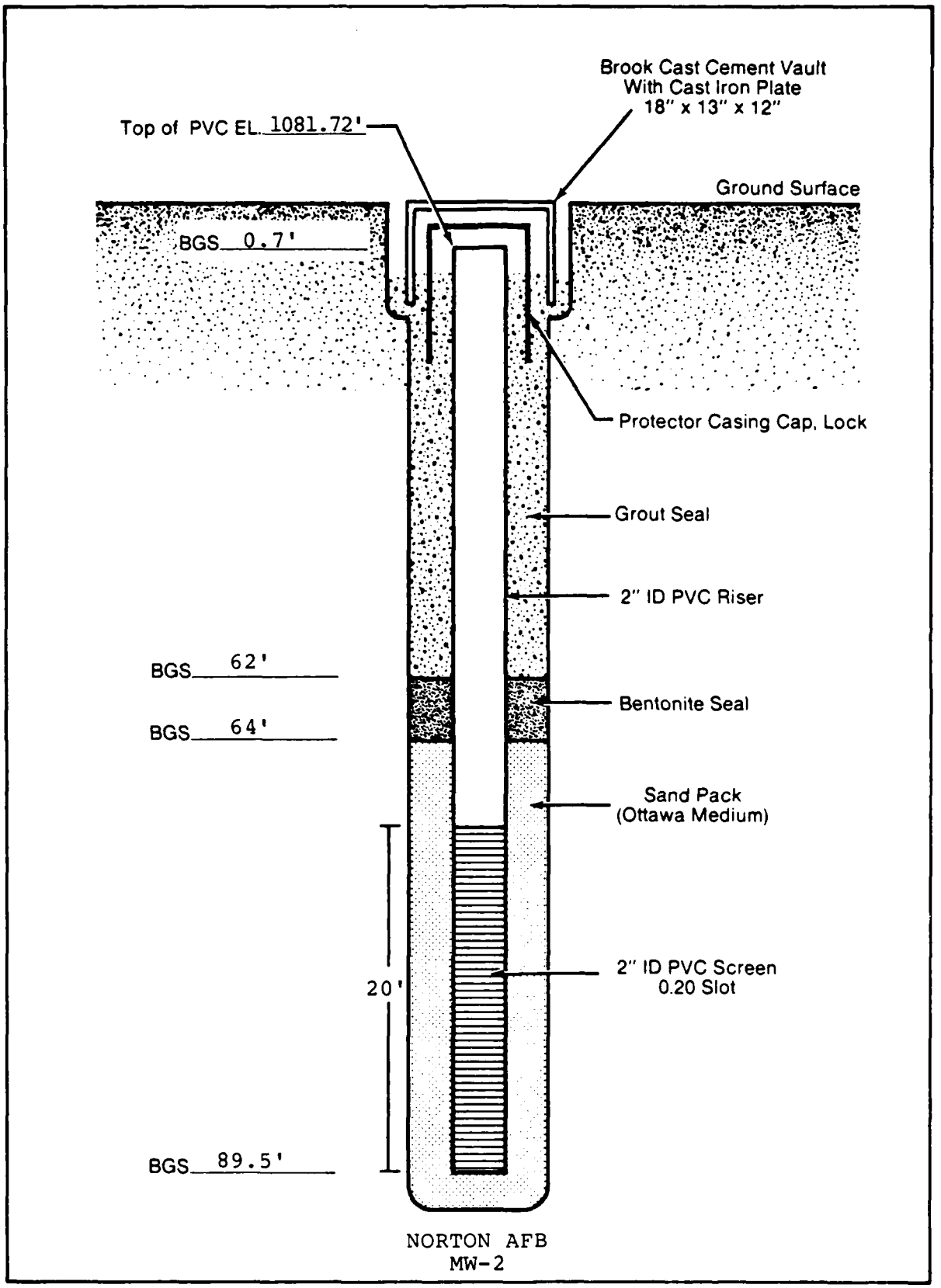
WELL NUMBER: MW-2 OWNER: USAF
LOCATION: Golf Course Site 1 Ind. Waste Lagoon ADDRESS: Norton AFB
TOTAL DEPTH 90.5'
SURFACE ELEVATION: WATER LEVEL: 30'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/22/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
70		16	SS	25/55	69-70.5 Dr. gray fine sandy SILT, wet to
				50	saturated, micaceous rec. 1.5
		17	SS	5/10	75-76.5 Grayish brown SAND, medium
75				18	to coarse, tr. fine gravel, saturated
					gravel on bottom of spoon
		18	SS	- -	79-80.5 Olive gray SAND, fine to medium,
					tr. fine gravel, some coarse sand,
80					some silt, wet, Note: Sand and gravels
					kept heaving in augers; thus split
					spoon sampled inside augers, blow counts
					not valid, sample is rep
		19	SS	5/8	84-85.5 Olive gray SAND, fine to
85				22	coarse, tr. fine gravel, some silt
					wet
		20	SS	7/8	89-90.5 Olive gray SAND, fine to medium, tr. fine gravel,
				19	some silt, wet.
90					No HNU readings in any split spoon samples.

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1081.72'
Ground Surface
BGS 0.7'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 62'
BGS 64'
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.20 Slot
BGS 89.5'
NORTON AFB
MW-2

WESTON DESIGNERS & CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-3 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 3
Waste Pit No. 2 TOTAL DEPTH 32.5'
SURFACE ELEVATION: _____ WATER LEVEL: 12.9'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/1/83
DRILLER: DCL HELPER: HM

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0					0-1.5 Black ASPHALT; Olive brown
		1	Aug		SAND, fine, some fine gravel, tr.
					medium sand, some silt, damp
					2-3.5 drilling hard, cobbles
5		2	SS	8/8	4-5.5 Grayish brown SAND, fine,
				9	tr. fine gravel, tr. coarse sand,
					some medium sand, tr. silt, damp
					rec. 1.2
		3	SS	11/13	9-10.5 Grayish brown SAND, fine to
10				11	coarse, some fine gravel,
					damp to moist rec. 1.2
					hit water @ 13.5'
		4	SS	6/16	14-15.5 Olive brown SAND, fine
15				19	to coarse, tr. to some fine
					gravel, tr. silt, saturated
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

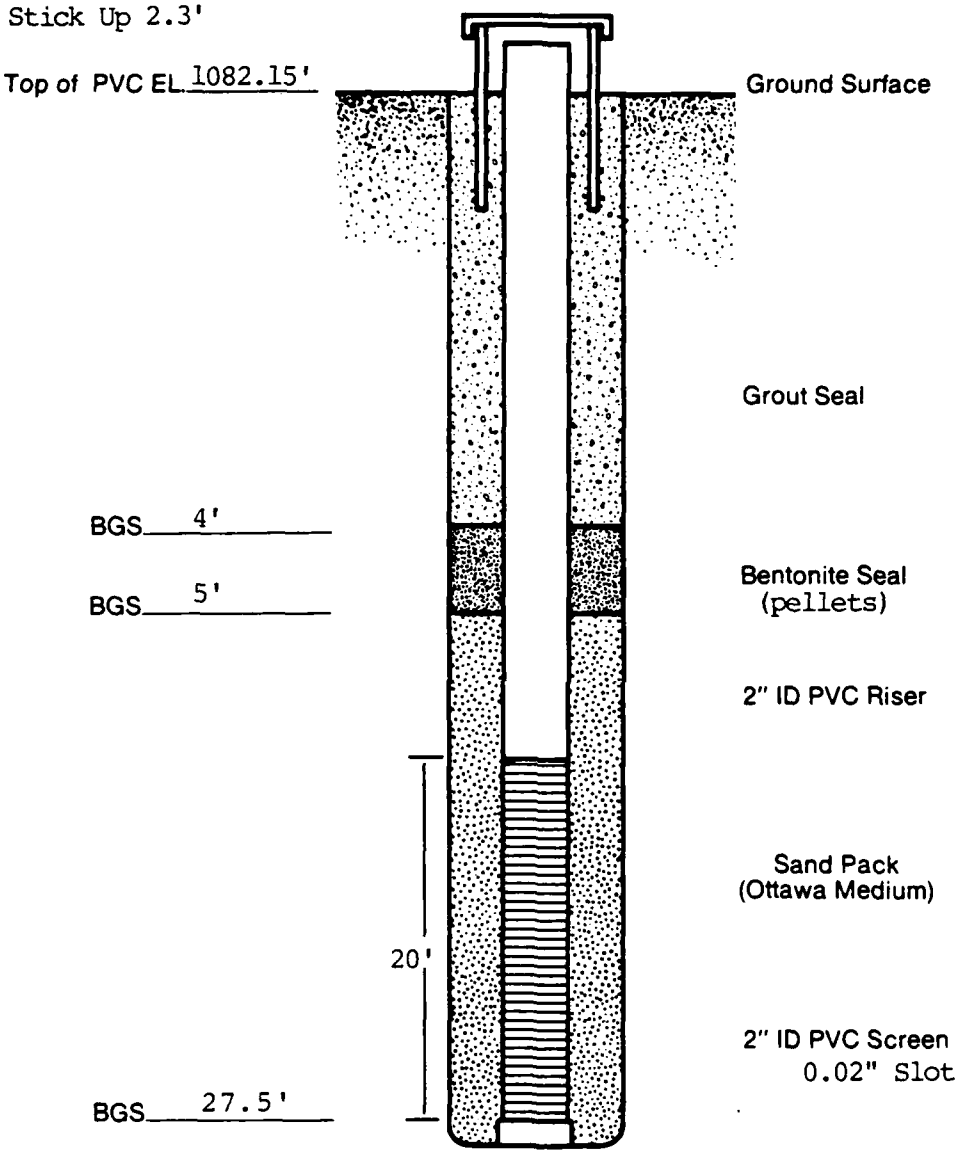
WELL NUMBER: MW-3
LOCATION: Golf Course Site 3
Waste Pit No. 2
SURFACE ELEVATION:
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH 32.5'
WATER LEVEL: 12.9'
DRILLING COMPANY: Stang Hydronics
DRILLING METHOD: Auger
DATE DRILLED: 12/1/83
DRILLER: DCL
HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	6/ 8 25	19-20.5 Dr. gray fine sandy SILT, low plasticity, damp to wet, micaceous
					grades to gray silty fine sand
					rec. 1.2
		6	SS	5/ 14 31	24-25.5 Olive brown SAND, coarse
25					tr. fine to medium sand, tr. silt,
					saturated rec. 1.0
30		7	SS	19/ 41 40	31-32.5 Olive gray SAND, fine
					to medium, tr. coarse sand,
					tr. silt, saturated; sample
					grades more coarse
35					No detectable HNU reading in any
					split spoon sample
40					

* ASTM D1586



NORTON AFB
MW-3

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-4 OWNER: USAF
LOCATION: Golf Course Site 4 Waste Pit 1 ADDRESS: Norton AFB
TOTAL DEPTH 65.5'
SURFACE ELEVATION: _____ WATER LEVEL: 20.2'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/14/83
DRILLER: DS HELPER: HM

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-1 Dr. olive brown SAND, fine to medium, some silt, moist
					1.0-4.0 same, some fine to coarse gravel.
		2	SS	23/32/27	4-5.5 Lt. gray SAND, medium to coarse, some fine gravel, tr. fine sand,
5					damp to moist rec 1.2
		3	SS	10/6/2	9-10.5 Lt. gray SAND, medium to coarse, some fine gravel, tr.
10					fine sand, moist; Dr. gray SILT, some fine sand, low plasticity, moist, micaceous, has
					organic smell rec 1.1
		4	SS	5/7/14/26	14-15.5 Lt. gray SAND, medium to coarse,
15					some fine gravel, tr. fine sand, moist; grayish brown SAND, fine, tr. medium to coarse sand, grades to coarse sand.

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-4
LOCATION: Golf Course Site 4 Waste Pit 1
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang Hydronics
DRILLER: DS
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 65.5'
WATER LEVEL: 20.2'
DRILLING METHOD: Auger
HELPER: HM
DATE DRILLED: 11/14/83

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	9/11/27	19-20.5 Dr. Olive gray silty SAND, fine, moist, micaceous, with Fe stains water @ 22' rec. 1.5'
		6	SS	11/30/38	24-25.5 Dr. Olive gray silty SAND, fine, saturated, micaceous; Olive gray SAND, medium to coarse, some fine sand, tr. silt, saturated.
25					
		7	SS	5/15/30	29-30.5 Olive gray SAND, fine tr. fine gravel, tr. medium sand, tr. silt, moist to wet, micaceous rec. 1.2'
30					
		8	SS	4/9/19	34-35.5 Olive gray SAND, fine to medium, tr. silt, saturated (possible cave in material); Dr. gray SILT, tr. fine sand, low plastic, damp to moist, micaceous
35					38' drilling hard
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-4 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 4
Waste Pit 1 TOTAL DEPTH 65.5'
SURFACE ELEVATION: _____ WATER LEVEL: 20.2'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/14/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	5/10	39-40.5 Dr. gray SILT, tr. fine sand,
				29	low plastic, damp to moist, micaceous;
					Olive brown silty SAND, damp, micaceous
					rec. 1.2'
45		10	SS	15/18	44-45.5 Olive gray silty SAND to
				35/4"	sandy SILT, fine sand, tr. clay, low plastic, moist,
					micaceous, with mottled reddish brown
					Fe stains
					rec. 1.5'
50		11	SS	7/16	49-50.5 Olive gray SAND, medium to coarse,
				31/2"	tr. fine sand, tr. silt,
					wet, with Dr. Olive gray silty
					fine sand lenses rec. 1.0
55		12	SS	25/43	54-55.5 Olive brown SAND, fine to medium,
				40/4"	tr. fine gravel, tr. silt, saturated

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

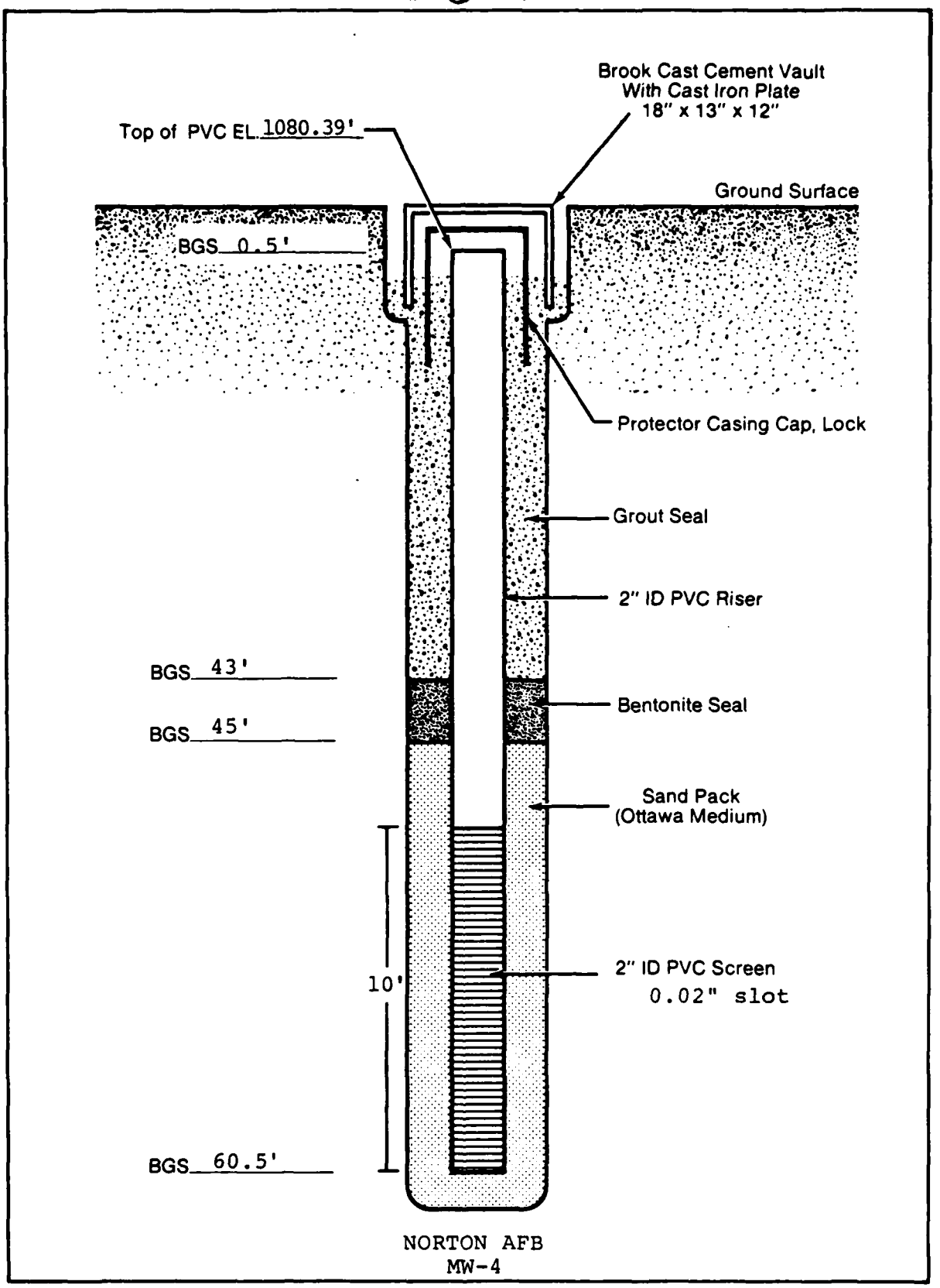
WELL NUMBER: MW-4 OWNER: USAF
LOCATION: Golf Course Site 4 Waste Pit 1 ADDRESS: Norton AFB
TOTAL DEPTH 65.5'
SURFACE ELEVATION: WATER LEVEL: 20.2'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/14/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
60		13	SS	35	59-60.5 Olive brown SAND, fine,
				60/5"	tr. medium sand, some silt, moist
					with some Dr. gray SILT lenses
		14	SS	16/28/41	64-65.5 Dr. Olive brown fine sandy
65					SILT, moist, with Olive brown
					silty fine sand lenses
					No detectable HNU reading in any split spoon sample.

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1080.39'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 43'
BGS 45'
Bentonite Seal
Sand Pack
(Ottawa Medium)
10'
2" ID PVC Screen
0.02" slot
BGS 60.5'
NORTON AFB
MW-4

WESTON

DRILLING LOG

WELL NUMBER: MW-5 OWNER: USAF

LOCATION: Golf Course, Site 10, Landfill 1 ADDRESS: Norton AFB

TOTAL DEPTH: 60.5'

SURFACE ELEVATION: _____ WATER LEVEL: 19.8'

DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/29/83

DRILLER: DS HELPER: DCL

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2 Olive gray SAND, fine to medium, tr. fine gravel, tr. coarse sand, tr. silt, moist, micaceous
5		2	SS	5/11 30/3"	4-5.5 Olive gray SAND, fine to medium, tr. fine gravel to coarse sand, tr. silt, moist, micaceous rec. 1.3
10		3	SS	22/30 27	9-10.5 Grayish brown SAND, fine to medium, tr. coarse sand, tr. silt, moist, micaceous rec. 1.4
15		4		11/21 25	14-15.5 Grayish brown SAND, fine to medium, tr. coarse sand, tr. silt, moist, micaceous, rec. 1.1
20					Note: a piece of glass came up augers between 5-20'

* A.S.T.M. D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-5
LOCATION: Golf Course
Site 10
Landfill 1
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang ~~Hydrolics~~
DRILLER: DS
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
WATER LEVEL: 19.8'
DRILLING METHOD: Auger
DATE DRILLED: 11/29/83
HELPER: DCL

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	14/24/28	19-20.5 Dr. gray fine sandy SILT, low plastic, moist; Olive gray silty SAND, wet, micaceous rec. 1.5
25		6	SS	10/20/20	24-25.5 Dr. gray SILT, tr. fine sand, plastic, grades wet to damp Possible perched water 23 to 25' rec. 1.3
30		7	SS	5/14/20	29-30.5 Dr. brownish gray silty SAND to sandy SILT, fine sand, moist, micaceous with some laminated Dr. gray, highly plastic clay water @ 27.4'
35		8	SS	5/11/21	34-35.5 Brownish gray SAND, fine to medium, tr. silt, saturated micaceous; Olive gray silty sand, fine, moist, with Fe stains
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-5
LOCATION: Golf Course, Site 10, Landfill 1
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang Hydronics
DRILLER: DS
LOG BY: BWB

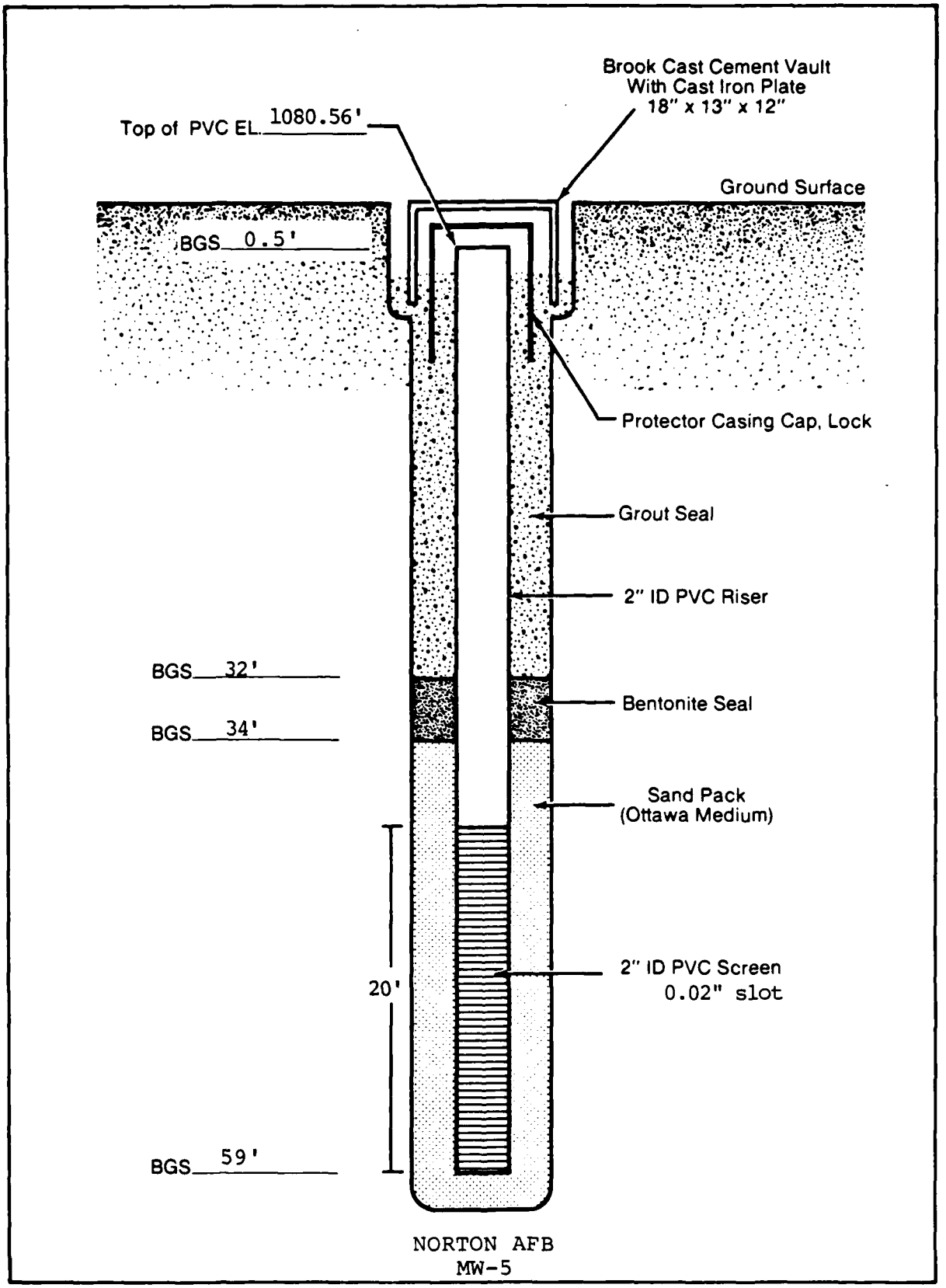
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 60.5'
WATER LEVEL: 19.8'
DRILLING METHOD: Auger
DATE DRILLED: 11/29/83
HELPER: DCL

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	14/24 50/5"	39-40.5 Olive gray SAND, medium to coarse, tr. fine gravel, tr. fine sand tr. silt, saturated
		10	SS	3/3 16	44-45.5 Dr. olive gray SAND, fine to medium sand, tr. to some silt moist to wet, micaceous
45					
		11	SS	3/4 14	49-50.5 Dr. Olive gray SAND, fine to medium, tr. silt, moist to wet, micaceous rec. 1.0
50					
		12	SS		54-55.5 Olive gray SAND, medium to coarse, tr. fine gravel, tr. fine sand, tr. silt, wet to saturated rec. 1.5
55					
		13	SS	29/35/47	59-60.5 Olive silty SAND, fine, tr. medium to coarse, saturated
60					No detectable HNU reading in any split spoon sample

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1080.56'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 32'
BGS 34'
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" slot
BGS 59'
NORTON AFB
MW-5

WESTON DESIGNSPHERE CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-6 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 10
Landfill 1 TOTAL DEPTH 55.5'
SURFACE ELEVATION: _____ WATER LEVEL: 29.5'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 12/6/83
DRILLER: DS HELPER: DCL

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2 Olive gray SAND, fine, tr. medium to coarse sand, some silt, damp
5		2	SS	9/11/10	4-5.5 Grayish brown SAND, fine to coarse, tr. silt, damp rec. 1.3
10		3	SS	9/10/3	9-10.5 Grayish brown SAND, fine to medium, tr. fine gravel, tr. coarse sand, damp to moist rec. 1.3
					13-14' drilling hard - gravel, cobbles
15		4	SS		14-15.5 Grayish brown SAND, fine to coarse, some fine to coarse gravel, moist rec. 1.1
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-6
LOCATION: Golf Course Site 10 Landfill 1
SURFACE ELEVATION: _____
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 55.5'
WATER LEVEL: 29.5'
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 12/6/83
DRILLER: DS
HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	13/32/28	19-20.5 Grayish brown SAND, fine to coarse, some fine to coarse gravel, moist; sand grades to mostly fine sand
25		6	SS	9/12/30	24-25.5 Lt. olive brown fine sandy SILT, tr. clay, moist, micaceous rec. 1.4
30		7	SS	17/18/25	29-30.5 Olive gray SILT, some fine sand, tr. clay, low plastic moist to wet, micaceous hit water @ 34'
35		8	SS	10/30/37	34-35.5 Olive gray SAND, fine to medium, tr. coarse sand, tr. silt, saturated rec. 1.5

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

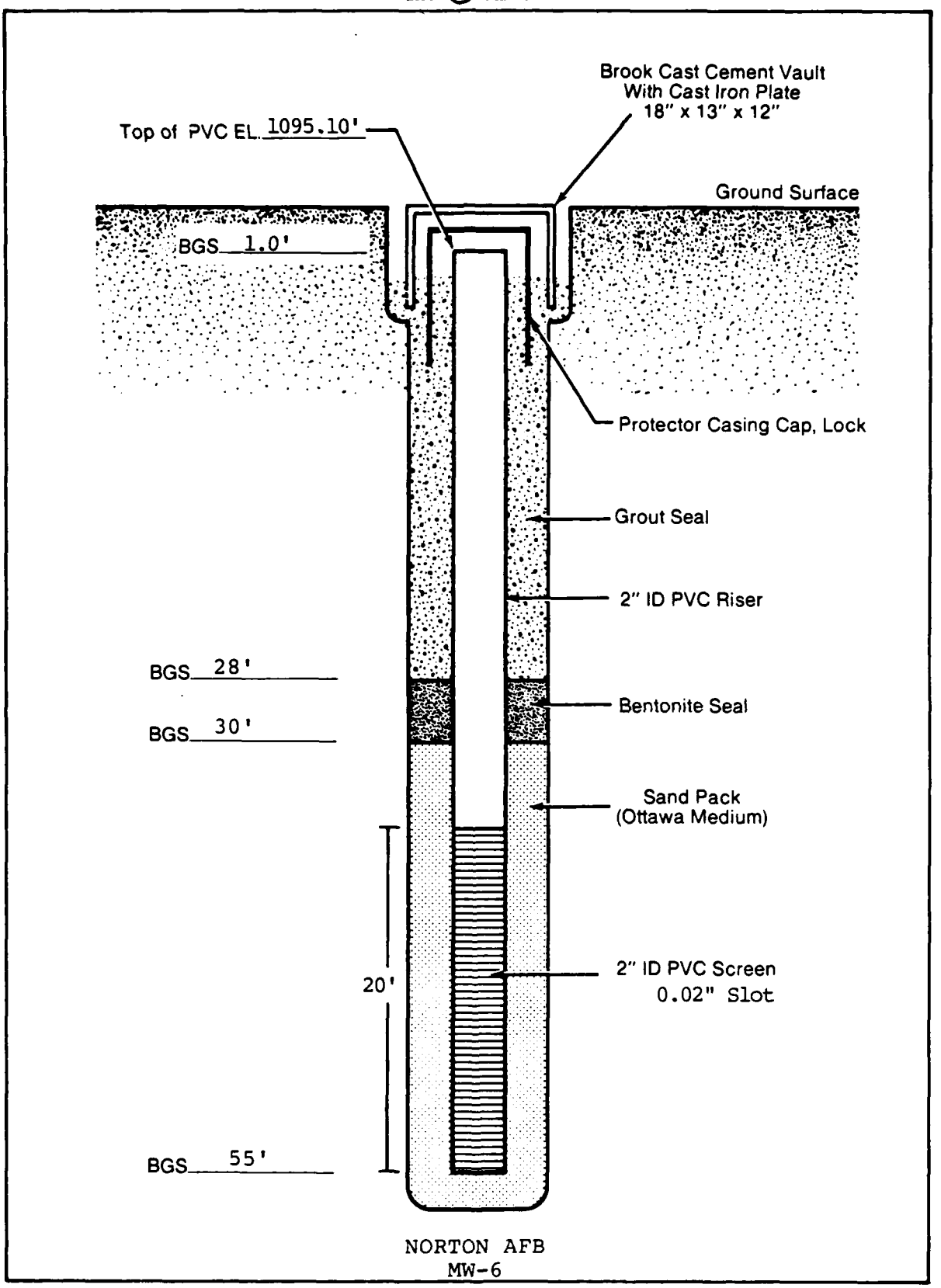
WELL NUMBER: MW-6 OWNER: USAF
LOCATION: Golf Course, Site 10, Landfill 1 ADDRESS: Norton AFB
TOTAL DEPTH 55.5'
SURFACE ELEVATION: WATER LEVEL: 29.5'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED:
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	4/12	39-40.5 Dr. gray silty SAND, fine,
				35	tr. medium sand, moist to wet,
					micaceous, grades to less silty on
					bottom
		10	SS	22/25	44-45.5 Dr. Olive gray SILT, low plastic,
45				60/4"	damp, micaceous, with medium
					plastic gray clay lamination
		11	SS	8/7	49-50.5 Olive gray SAND, fine to medium
50				10	tr. coarse sand, tr. silt,
					saturated
		12	SS	7/13	54-55.5 Olive gray SAND, fine to medium,
				22	tr. coarse sand, tr. silt,
55					wet
					No detectable HNU reading in any split spoon sample

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1095.10'
Ground Surface
BGS 1.0'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 28'
BGS 30'
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 55'
NORTON AFB
MW-6

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-7 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 10
Landfill 1 TOTAL DEPTH 60'
SURFACE ELEVATION: _____ WATER LEVEL: 27.6'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 12/5/83
DRILLER: DS HELPER: Steve
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		1	Aug		1-2' Olive gray SAND, fine to medium,
					tr. coarse sand, tr. silt, damp
					3-6' Hitting gravel
		2	SS	20	6-7.5 Grayish brown GRAVEL and COBBLES,
				37/-	some fine sand, damp, spoon
					pushing cobble
					rec. 0.3'
		3	SS	16/21	10-11.5 Grayish brown SAND, fine,
				25	tr. medium sand, damp
					13-14' hitting gravel, cobbles
		4	SS	7/17	14-15.5 Olive gray clayey SILT to silty
				25	CLAY, tr. fine sand, medium plastic,
					damp; Olive gray SAND, tr. silt,
					damp

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-7 OWNER: USAF
LOCATION: Golf Course Site 10 Landfill 1 ADDRESS: Norton AFB
TOTAL DEPTH 60'
SURFACE ELEVATION: WATER LEVEL: 27.6'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 12/5/83
DRILLER: DS HELPER: Steve
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		5	SS	24/31/30	19-20.5 Olive brown SAND, fine, tr. fine gravel, tr. medium to coarse sand, damp, micaceous; Dr. Olive gray SILT on bottom rec. 1.3'
		6	SS	16/30/34	24-25.5 Dr. olive gray SAND, fine to medium, tr. fine gravel, tr. coarse sand, tr. silt, moist to wet, grades to mostly medium sand rec. 1.3'
		7	SS	15/35/17/1"	29-30.5 Olive gray SAND, fine to coarse, tr. fine gravel, tr. silt, saturated
		8	SS	7/11/14	34-35.5 Dr. gray SILT, tr. fine sand, tr. clay, low plastic, damp, micaceous rec. 1.5'

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

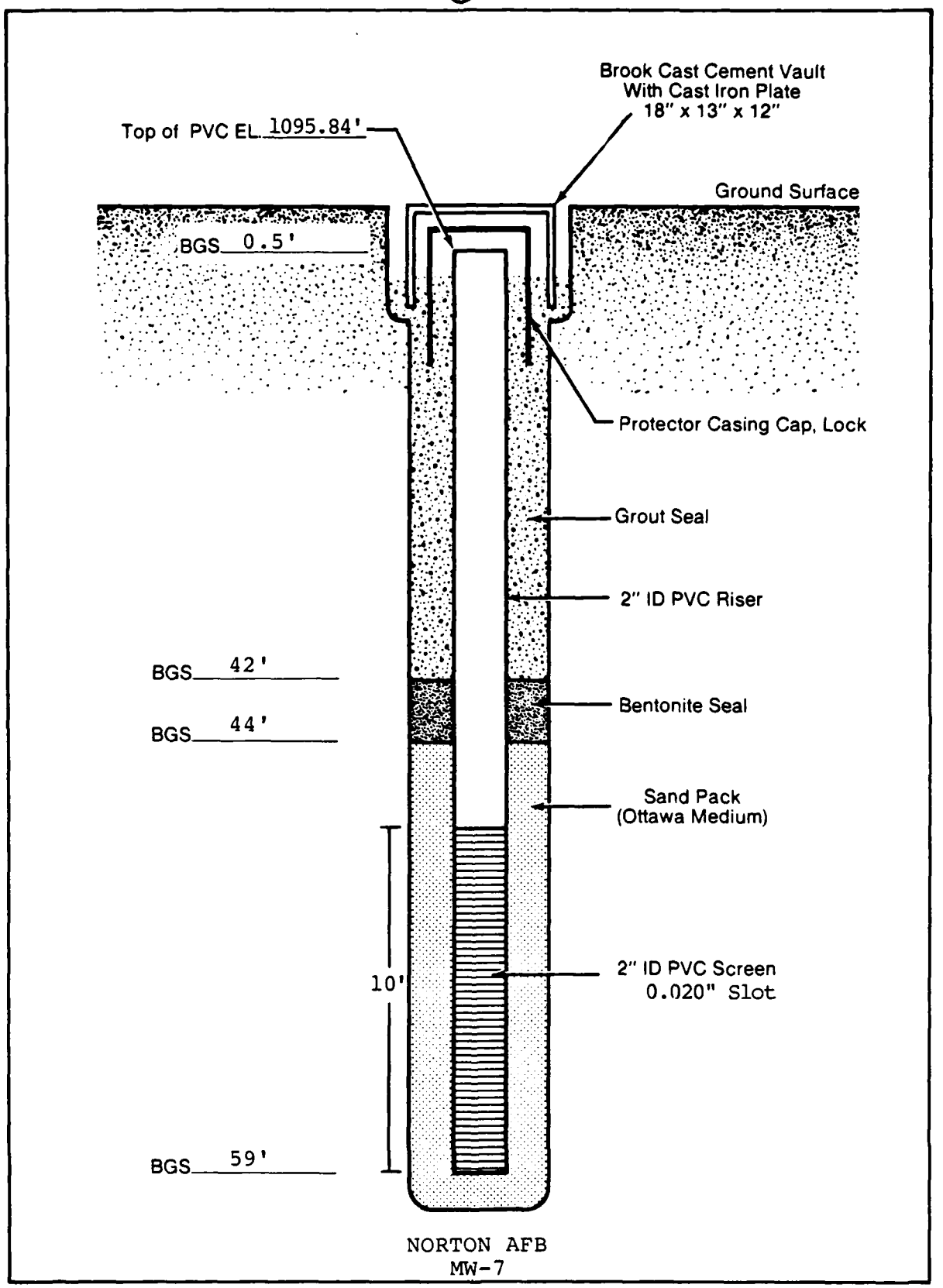
WELL NUMBER: MW-7 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 10
Landfill 1 TOTAL DEPTH 60'
SURFACE ELEVATION: WATER LEVEL: 27.6'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 12/5/83
DRILLER: DS HELPER: Steve
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	10/20 46	39-40.5 Dr. Gray SILT, low plastic damp; Olive gray SAND, fine, some silt, wet, micaceous rec. 1.5
45		10	SS	9/27 30	44-45.5 Dr. olive gray silty SAND, fine, damp to moist, grades more medium sand rec. 1.3
50		11	SS	6/30 51	49-50.5 Olive gray SAND, fine to coarse, tr. fine gravel, some silt, saturated rec. 1.2'
55		12	SS	10/45 37	54-55.5 Olive gray SAND, fine to coarse, tr. fine gravel, some silt, saturated
		13	SS	10/29 37	59-60.5 Olive gray SAND, fine to coarse, tr. fine gravel, some silt, saturated
60					No detectable HNU reading in any split spoon samples

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1095.84'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 42'
BGS 44'
Bentonite Seal
Sand Pack
(Ottawa Medium)
10'
2" ID PVC Screen
0.020" Slot
BGS 59'
NORTON AFB
MW-7

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-8 OWNER: USAF
LOCATION: Golf Course, Site 12, Waste Pit 3 ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
SURFACE ELEVATION: _____ WATER LEVEL: 24.7'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/28/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2	Olive brown SAND, fine, tr. medium to course, tr silt, damp
					2.5-4'	Hitting coarse gravel
5		2	SS	25/26	4-5.5	Grayish brown SAND, fine to coarse, damp; Grayish brown GRAVEL, fine to coarse, damp
				40/4"		rec. 1.2'
10		3	SS	5/11/26	10.5-12	Dr. gray SILT, tr. fine sand, low plastic, moist, micaceous; Grayish brown SAND, grades fine to medium, damp, top of spoon above silt was saturated
15		4	SS	15/15/11	14-15.5	Grayish brown SAND, fine to medium, tr. coarse sand, moist; Dr. gray SILT, low plastic, moist
20						

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-8 OWNER: USAF
LOCATION: Golf Course ADDRESS: Norton AFB
Site 12
Waste Pit 3 TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 24.7'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/28/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	10/21/26	19-20.5 Grayish brown silty SAND, fine, damp to moist, micaceous rec. 1.5'
		6	SS	18/15/13	24-25.5 Olive brown SAND, fine to coarse, moist; Reddish brown CLAY, tr. fine sand, medium plastic, moist rec. 1.5'
25					
		7	SS	16/29/28	29-30.5 Olive gray SAND, fine to medium, tr. coarse sand, saturated, grades siltier on bottom of spoon rec. 1.3'
30					
		8	SS	5/14/37	34-35.5 Olive gray SAND, fine to medium, saturated; Dr. Olive gray fine sandy SILT, damp, micaceous
35					
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

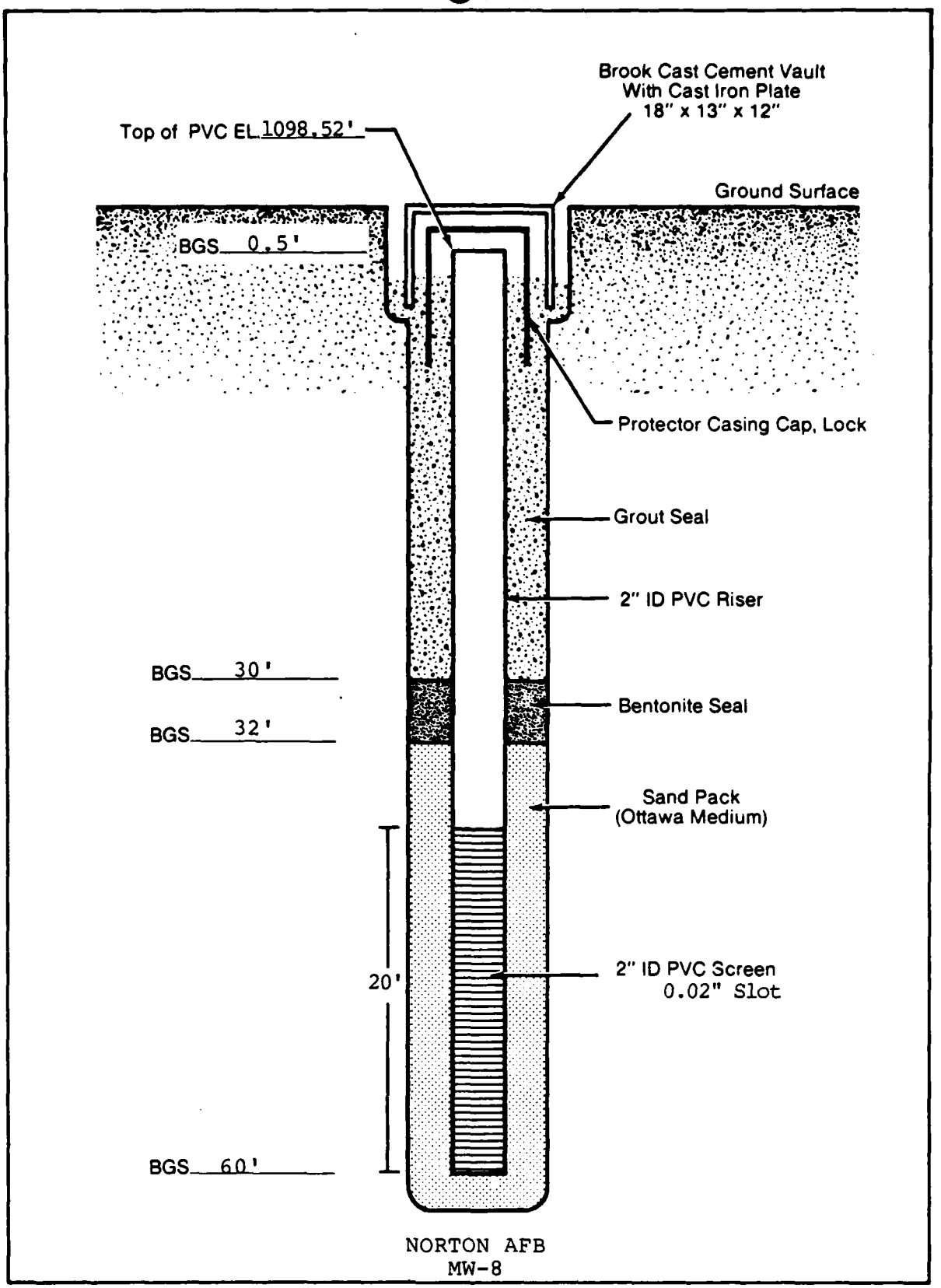
WELL NUMBER: MW-8 OWNER: USAF
LOCATION: Golf Course Site 12 Waste Pit 3 ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 24.7'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/28/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	11/45 50/4"	39-40.5 Dr. Olive gray silty SAND, moist, micaceous, some medium sand on bottom of spoon saturated
45		10	SS	4/3 27	44-45.5 Grayish brown SAND, fine to coarse, tr. fine gravel, saturated
			SS	6/28 25	49-50.5 Lost sample
50		11	SS	4/16 20	50.5-52 Grayish brown SAND, fine to coarse, tr. fine gravel, saturated
		12		1/1 5	54-55 Olive gray SAND medium to coarse, tr. fine sand, saturated micaceous, sampled material in auger, sample representative, blow count not valid
55		13		5/15 50	59-60.5 Dr. gray SAND, fine to coarse, saturated; Dr. gray Gravel, fine to coarse, saturated
60					No detectable HNU reading in any SS sample

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1098.52'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 30'
BGS 32'
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 60'
NORTON AFB
MW-8

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-9 OWNER: USAF
LOCATION: Fire Training Site 5 ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 29.8'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auter DATE DRILLED 11/19/83
DRILLER: DS HELPER: WH
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2 Dr. grayish brown silty SAND, fine, tr. medium, dry
					3.5' drilling hard cobbles
5		2	SS	11/18 17	5-6.5 lost sample - pushed gravel
		3	SS	16/34 50/4"	9-10.5 Grayish brown fine gravelly SAND, fine to coarse, damp rec. 1.2'
10					12-14' drilling hard
		4	SS	19/44 52	14-15.5 Grayish brown SAND, fine to medium, tr. coarse, tr. silt, damp rec. 1.1
15					
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-9 OWNER: USAF
LOCATION: Fire Training Site 5 ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 29.8'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/19/83
DRILLER: DS HELPER: WH
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	13/35 34	19-20.5 Grayish brown SAND, coarse, tr. fine gravel, tr. fine to medium sand, moist rec. 1.2'
		6	SS	10/21 22	24-25.5 Dr. gray SILT, tr. fine sand, low plastic, moist, micaceous; Lt. gray SAND, fine, some silt, moist rec. 1.5'
25					
		7	SS	15/19 32	29-30.5 Olive gray silty SAND, fine, moist, with a 1" dr. gray silt lense; Lt gray SAND, fine some silt, moist rec. 1.3'
30					
		8		15/32 40	34-35.5 Olive gray silty SAND, fine, moist, micaceous
35					
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

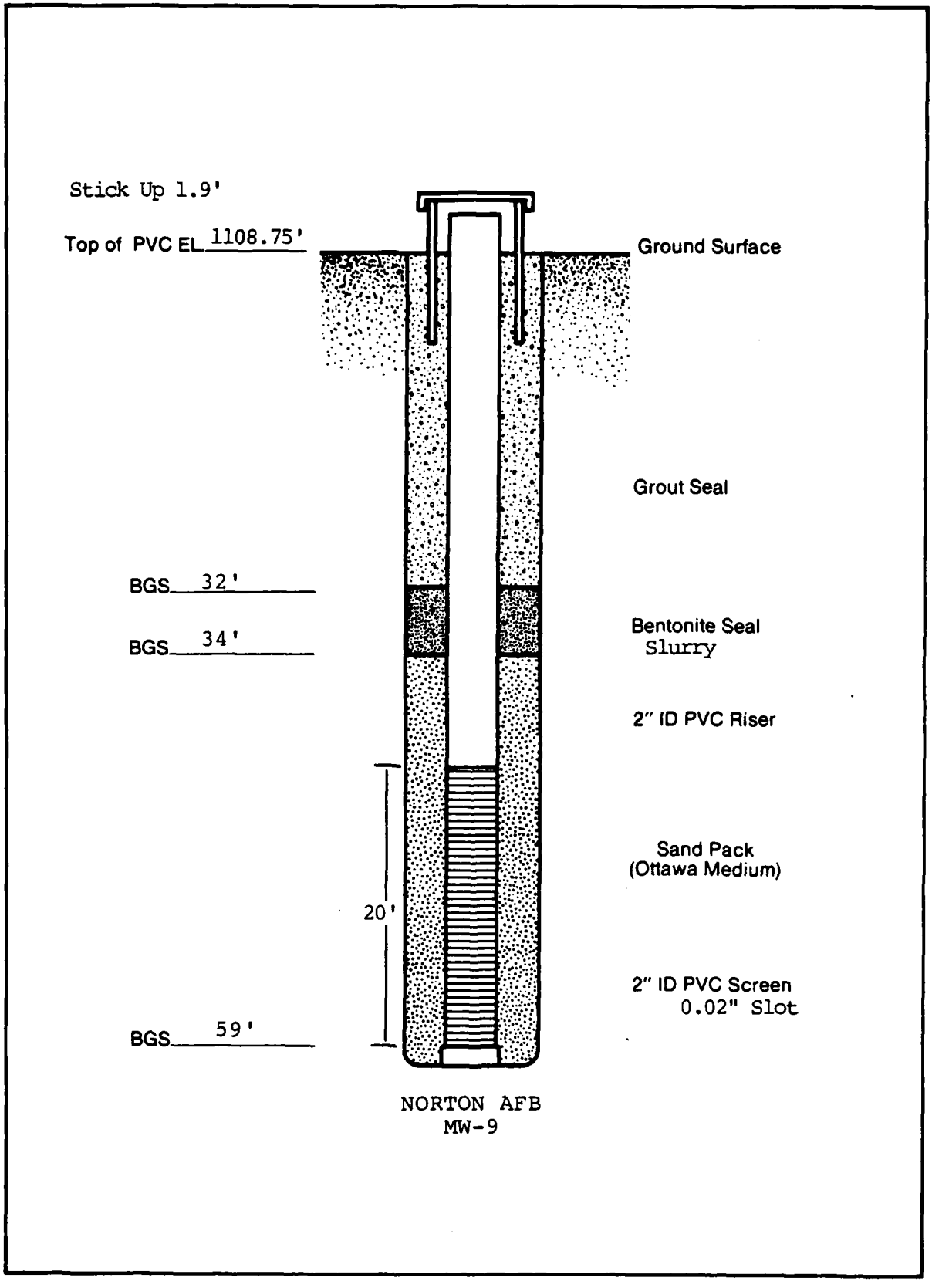
WELL NUMBER: MW-9 OWNER: USAF
LOCATION: Fire Training Site 5 ADDRESS: Norton AFB
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 29.8'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11-19-83
DRILLER: DS HELPER: WH
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	14/20 25	39-40.5 Dr. gray SILT, tr. to some fine sand, low plastic, moist to wet, micaceous, grades sandier, wet rec. 1.3
45		10	SS	4/17 65 4"	45-50.5 Olive gray SAND, fine to coarse, tr. silt, saturated micaceous rec. 0.8'
50		11	SS	14/8 5	49-50.5 Grayish brown SAND, fine to medium, tr. fine gravel, tr. coarse sand saturated rec. 0.6'
55		12	SS	1/1 14	54-55.5 Yellowish brown SAND coarse, tr. fine gravel, tr. fine to medium sand, saturated
		13	SS	2/8 7	59-60.5 - As above rec. 0.4'
60					Note: Samples 10 through 13 had small recoveries because of excessive running sand in augers. No detectable HNU reading in any SS sample

* A.S.T.M. D1586


Stick Up 1.9'
Top of PVC EL 1108.75'
Ground Surface
Grout Seal
BGS 32'
BGS 34'
Bentonite Seal
Slurry
2" ID PVC Riser
20'
Sand Pack
(Ottawa Medium)
2" ID PVC Screen
0.02" Slot
BGS 59'
NORTON AFB
MW-9

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-10
LOCATION: IWTP Site 7 Sludge Beds
SURFACE ELEVATION: Stang
DRILLING COMPANY: Hydronics
DRILLER: DS
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 30.5'
WATER LEVEL: 15.1'
DRILLING METHOD: Auger
DATE DRILLED: 11/5/83
HELPER: HM

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-1' Dr. grayish brown SAND, fine, tr. fine gravel, tr. medium sand, tr. silt, damp
5		2	SS	9/20/26	4-5.5 Grayish brown SAND, fine to coarse, tr. fine gravel, damp
10		3	SS	18/25/12	9-10.5 Grayish brown SAND, fine to coarse, tr. fine gravel, damp
15		4	SS	15/20/15	14-15.5 Grayish brown SAND, fine to medium, tr. fine gravel, tr. coarse sand, tr. silt, moist to wet hit water 15.5'
20					

* A.S.T.M. D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-10 OWNER: USAF
LOCATION: IWTP ADDRESS: Norton AFB
Site 7
Sludge Beds TOTAL DEPTH 30.5'
SURFACE ELEVATION: _____ WATER LEVEL: 15.1'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/5/83
DRILLER: DS HELPER: HM

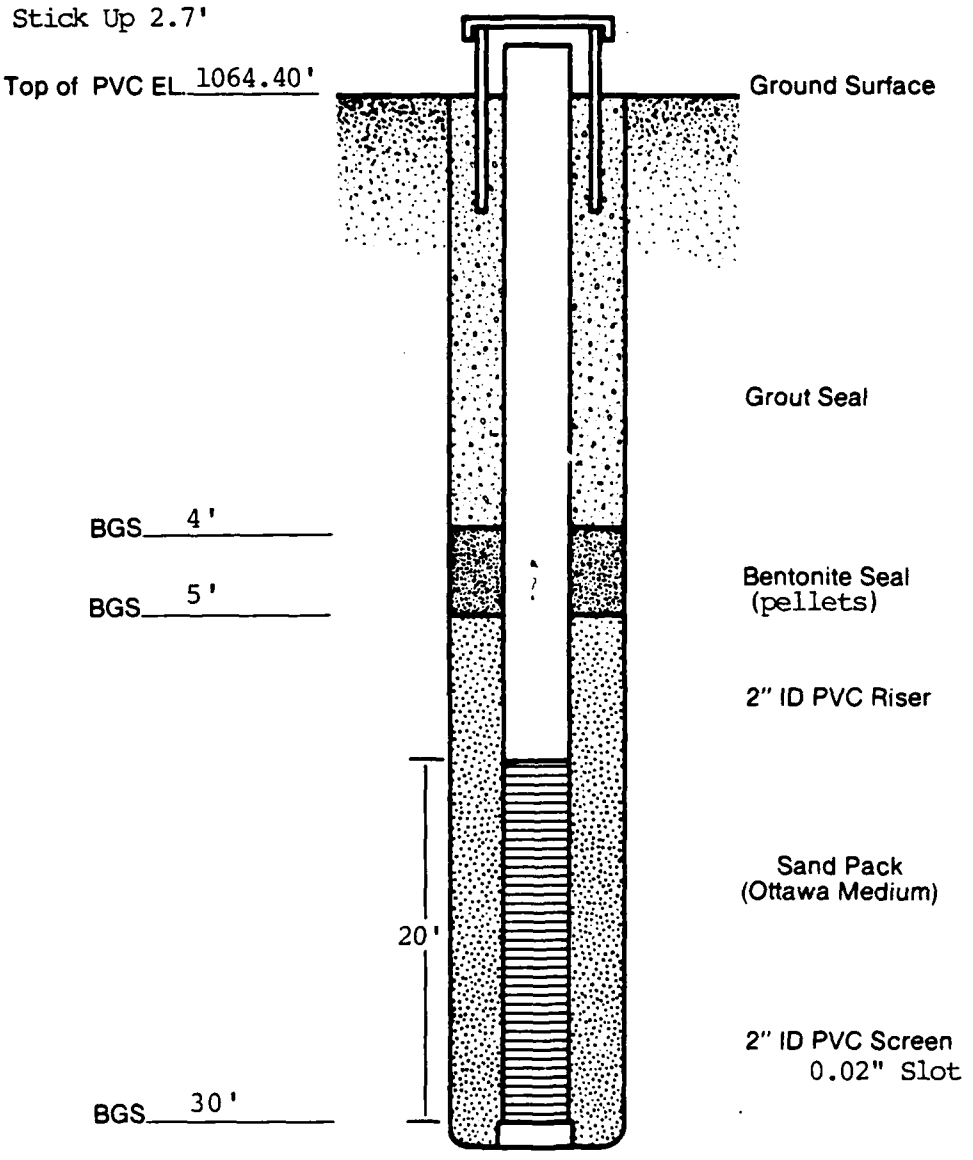
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	1/1	19-20.5 Grayish brown SAND, fine to medium
				1	tr. fine gravel, tr. coarse
					sand, tr. silt, saturated
					rec. 0.5'
		6	SS	7/13	24-25.5 Grayish brown SAND, fine
25				16	to coarse, tr. fine gravel,
					saturated rec. 1.4'
		7	SS	13/12	29-30.5 Olive gray silty SAND, fine, tr. medium
				16	sand, saturated, micaceous
30					rec. 1.5'
					No detectable HNU reading in any
					split spoon sample

* A.S.T.M. D1586



NORTON AFB
MW-10

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-11
LOCATION: Landfill
Waste Management
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH 62.5'
SURFACE ELEVATION: _____
WATER LEVEL: 43'
DRILLING COMPANY: Stang Hydronics
DRILLING METHOD: Auger
DATE DRILLED: 11/17/83
DRILLER: DS
HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-1' Grayish brown SAND, fine to
					medium, some coarse sand, tr. silt
					damp
		2	SS	10/12	4-5.5 Grayish brown SAND, fine to
				21	coarse, tr. fine gravel, moist
5					
		3	SS	20/20	9-10.5 Grayish brown SAND, fine to
				21	coarse, gr. fine gravel, moist,
					grades to more fine sand on bottom
10					
					13-14' Drilling hard; coarse gravel,
					cobbles
					@ 14' bit bouncing on boulder
15					On 11-17-83 attempted 3 borings between 15' N and 15'S
					of proposed well site. Auger refusal at approximately 15'.
					On 12-4-83 attempted 6 more locations between 20-50'N
					of proposed well site.

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-11 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 62.5'
SURFACE ELEVATION: WATER LEVEL: 43'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/8/83
DRILLER: DS HELPER: DCL

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
10					On 12-7-83 Attempted 2 more locations approx. 30 yards and 50 yards west of proposed well site- hitting boulders at 14-20'. On 12-8-83 - Offset 75 yards west of proposed well site - drilled to 60'
15		4	SS	13/23	14-15.5 Grayish brown fine to medium SAND
				20	tr. coarse sand, damp
		5	SS	50/6"	19-20.5 Grayish brown Gravel, fine to coarse
20					and cobbles, some fine to coarse Grayish brown sand, damp
		6	SS	-	23-24.5 Grayish brown SAND, fine, tr. silt,
				-	damp, grades siltier
25					on bottom
		7		21	29-30.5 Grayish brown SAND, fine,
				50/5"	tr. coarse gravel, tr. medium to coarse sand, damp
30					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-11 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 62.5'
SURFACE ELEVATION: WATER LEVEL: 43.0'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/8/83
DRILLER: HM HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
30					
					33' drilling moderately hard
		8	SS	9/17	34-35.5 Dr. olive gray silty SAND to sandy
35				20	SILT, fine sand, low plastic, damp
					micaceous
		9	SS	6/31	39-40.5 Olive gray sandy SILT, fine
40				40	sand, low plastic, damp
					micaceous, Fe stains
					Hit water 45'
		10	SS	4/7	44-45.5 Dr. olive gray SAND, fine
45				5	to medium, tr. fine gravel, tr. coarse
					sand, saturated
50					

* A.S.T.M. D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

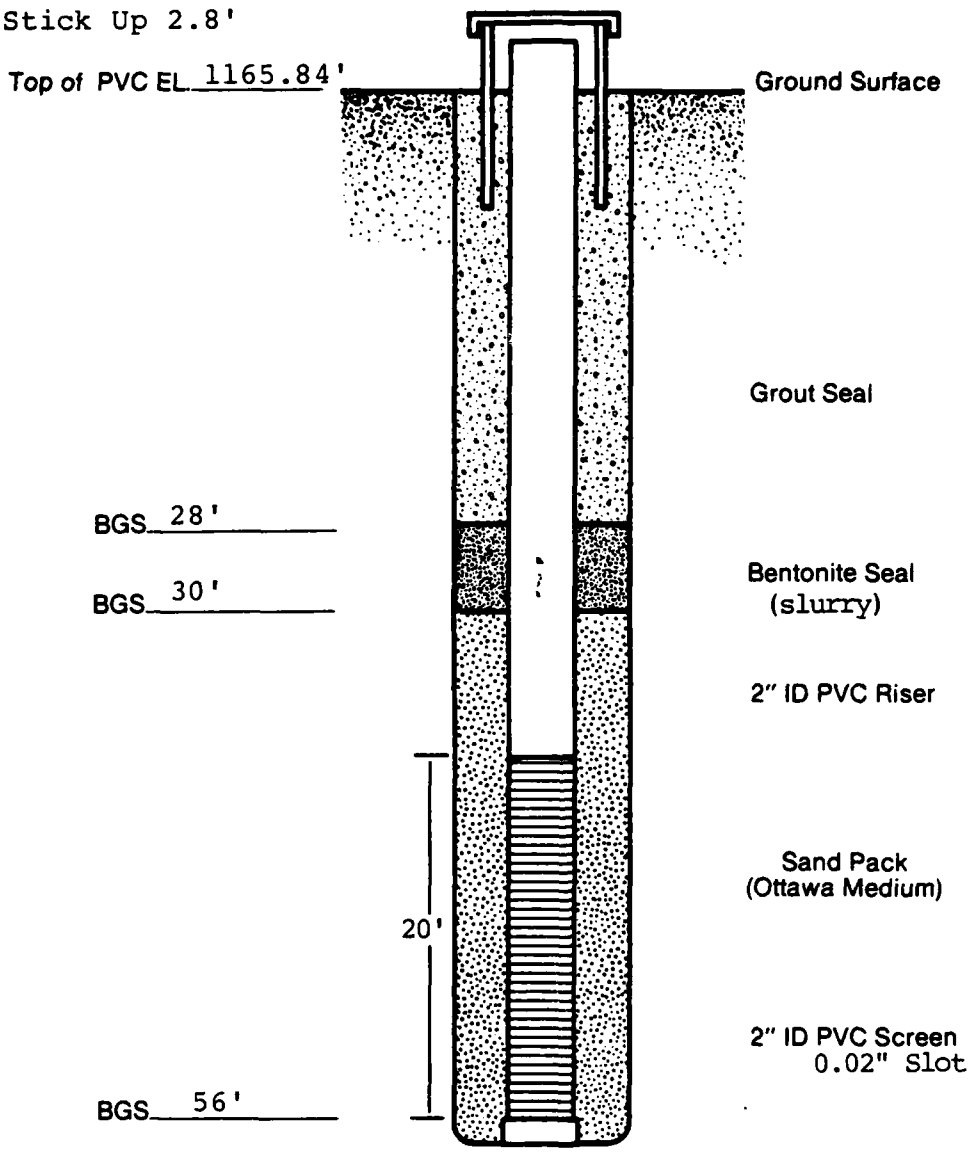
WELL NUMBER: MW-11 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 62.5'
SURFACE ELEVATION: WATER LEVEL: 43.0'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 12/8/83
DRILLER: HM HELPER: DCI
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
50		11	SS	20/26 30 4"	49.5-51 Olive gray SAND, fine to coarse, tr. fine gravel, tr. silt, saturated
55		12	SS	3/9 19	54-55.5 Olive gray SAND, fine to coarse, tr. fine gravel, tr. silt, saturated
		13	SS	5/22 13	61-62.5 Olive gray SAND, fine to medium, tr. fine gravel, tr. coarse sand, tr. silt, saturated
60					No detectable HNU reading in any split spoon sample

* A.S.T.M. D1586



NORTON AFB
MW-11

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-12
LOCATION: Landfill
Waste Management
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 60.5'
SURFACE ELEVATION: _____
WATER LEVEL: 41.0'
DRILLING COMPANY: Stang Hydronics
DRILLING METHOD: Auger
DATE DRILLED: 11/16/83
DRILLER: DS
HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2	Grayish brown SAND, fine to medium,
						tr. coarse sand, tr. silt, damp
						3' drilling hard hitting coarse
						gravel
		2	SS	7/9	4-5.5	Grayish brown SAND, fine to
5				8		coarse, tr. fine gravel, damp; Grayish
						brown SAND, fine to medium, damp
					7-8.5'	hitting cobbles - Offset
						3' N and drilled to 9'
		3	SS	12/14	9-10.5	Grayish brown SAND, medium, tr.
10				19		fine sand, damp
		4	SS	16	14-15.5	Grayish brown SAND, medium to
15				27/3"		coarse, some fine sand, moist
					15.5-18'	drilling hard
20						

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-12 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 41.0'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/16/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	16/38/43	19-20.5 Grayish brown SAND, fine to coarse, tr. fine gravel, damp. micaceous rec. 1.2
		6	SS	25/50/3"	24-25.5 Grayish brown SAND, fine to coarse, some fine gravel, moist rec. 1.3
25					
		7	SS	25/27/19	29-30.5 Grayish brown SAND, fine to coarse, some fine gravel, moist; Olive brown SILT, tr. fine sand, low plastic, damp micaceous rec. 1.0
30					
		8	SS	20/44/50/4"	34-35.5 Olive brown, grayish brown SAND, fine to coarse, tr. silt, damp
35					
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-12 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 41.0'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/16/83
DRILLER: DS HELPER: DCL

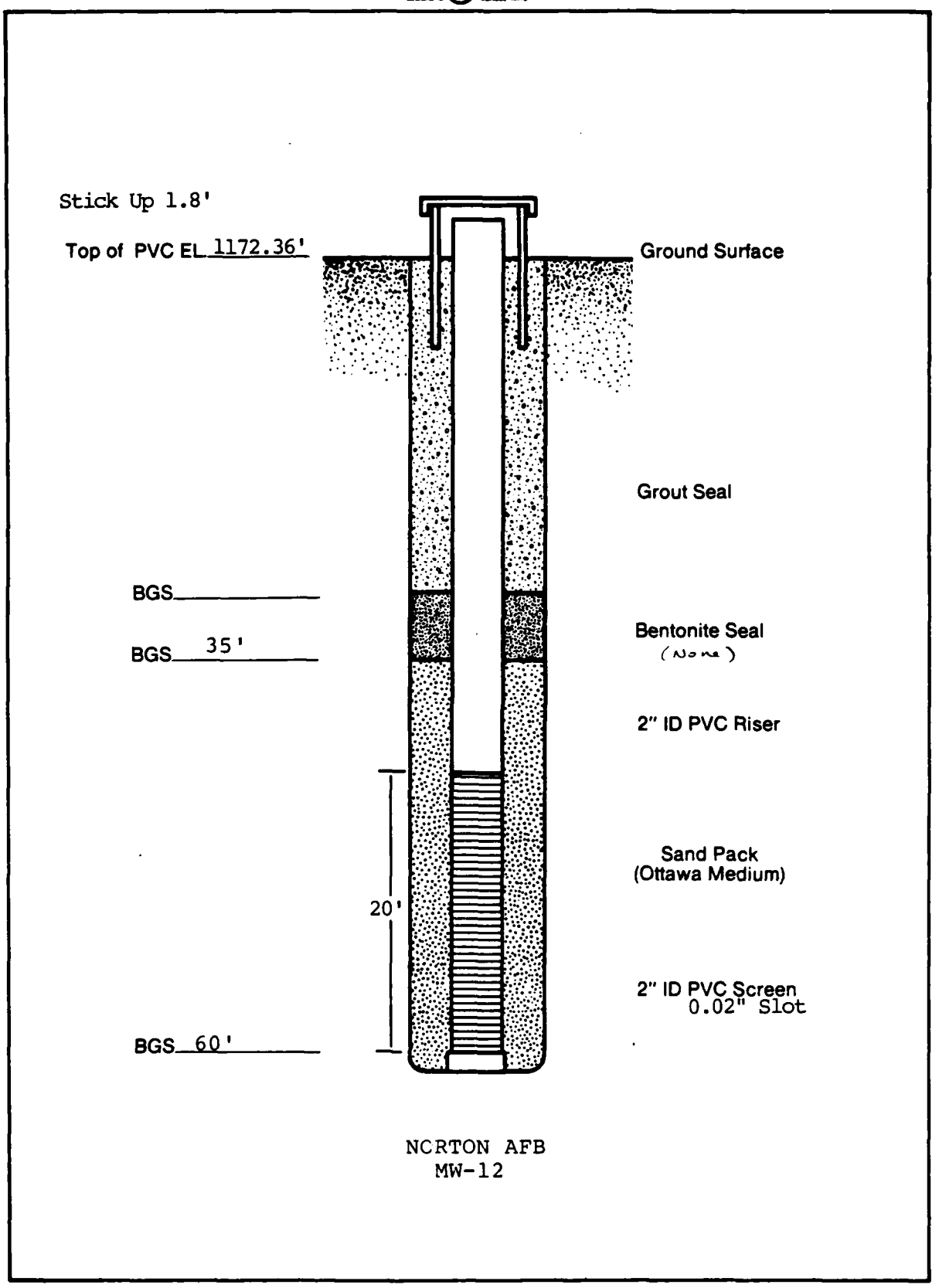
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	27/36 50	39-40.5 Olive gray SAND, fine, some medium sand, tr. silt, moist
					hit water @ 42'
		10	SS	25/36 50/4"	44-45.5 Olive gray SAND, fine to coarse tr. fine gravel, saturated
45					
					48' hit cobbles, drilled to 51' prior to sampling.
		11	SS	6/12	51-52.5 Olive gray SAND, fine to coarse, tr. fine gravel, saturated
50					sand running up auger - adding water to induce head
		12	SS	5/7	56-57.5 Olive gray SAND, fine to coarse, saturated; Blow counts not valid; Soil sample is valid
55		13			59.5-61 Olive brown SAND, fine to coarse, tr. fine gravel, saturated
					No detectable HNU reading in any split spoon samples.
60					

* A.S.T.M. D1586



NCRTON AFB
MW-12

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-13 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 43.1'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/19/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		1-2	Grayish brown SAND, medium to coarse, tr. fine gravel, tr. fine sand damp
5		2	SS	14/19 43	4-5.5	Grayish brown SAND, fine to coarse, tr. fine gravel, damp
10		3	SS	9/15 16	9-10.5	Grayish brown SAND, fine to medium some coarse sand, damp
15		4	SS	13/25 26	14-15.5	Grayish brown SAND, fine to coarse, tr. silt, damp
20						

* A.S.T.M. D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-13 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 43.1'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/18/83
DRILLER: DS HELPER: DCL
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	22/34/35	19-20.5 Grayish brown SAND, fine to coarse, tr. silt, dry rec. 1.1
25		6	SS	16/44/48	24-25.5 Grayish brown SAND, medium to coarse, some fine gravel, some fine sand, dry rec. 1.2
					28-29' drilling hard
30		7	SS	19	29-30.5 Lt. grayish brown SAND, medium to coarse, some fine gravel, some fine sand, dry
		8	SS	25	34-35.5 Lt. grayish brown SAND,
35				40/111	fine, tr. fine gravel, tr. medium to coarse sand, damp
					37-38' drilling moderately hard
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-13 OWNER: USAF
LOCATION: Landfill ADDRESS: Norton AFB
Waste Management
TOTAL DEPTH 60.5'
SURFACE ELEVATION: WATER LEVEL: 43.1'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/18/83
DRILLER: DS HELPER: DCL

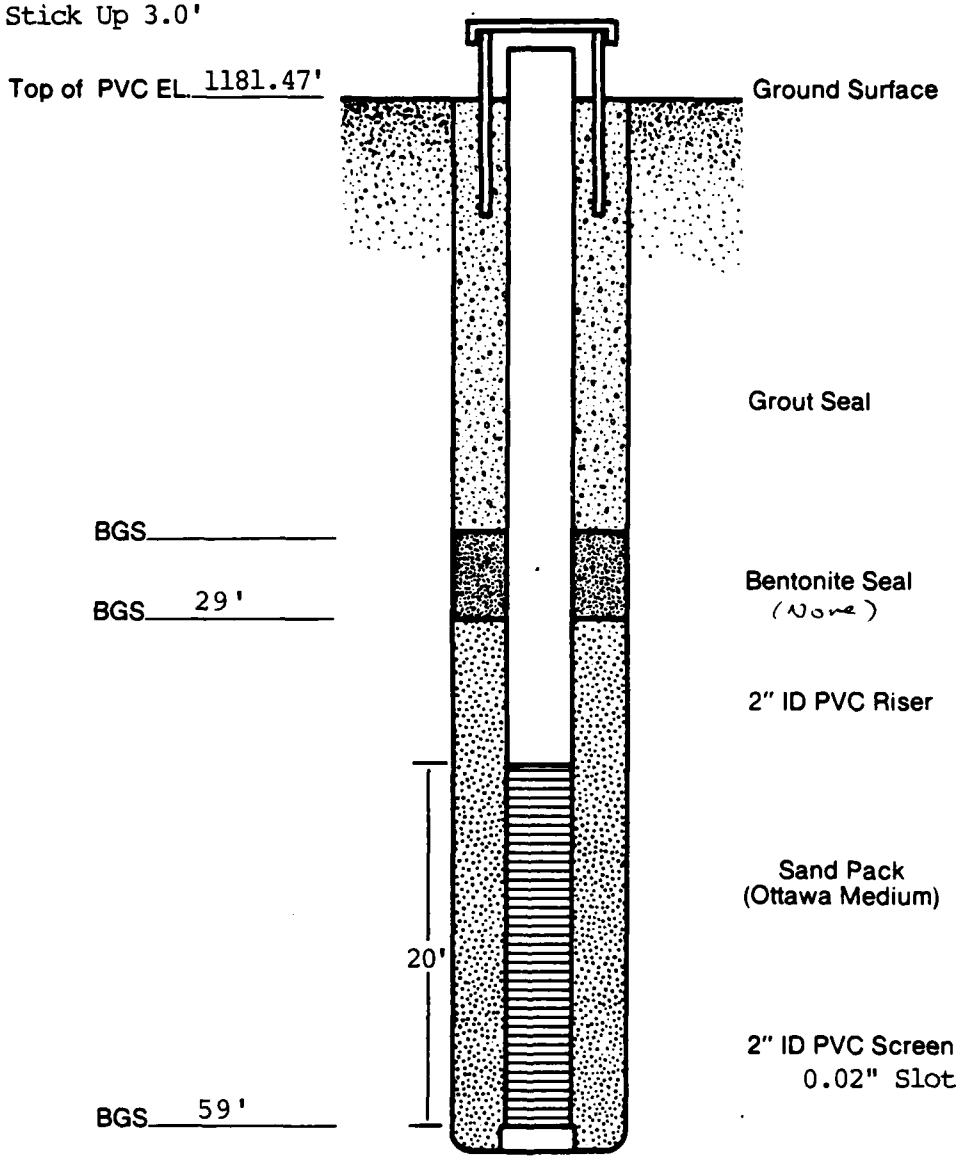
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		9	SS	26/32/36	39-40.5 Olive gray SAND, fine to coarse, tr. silt, damp to moist
		10	SS	25/27/37	44-45.5 Olive gray SAND, medium to coarse, tr. fine sand, saturated, with black fine sand laminations rec. 1.5
		11	SS	10/38/55	40-50.5 Olive gray SAND, fine to coarse, saturated
					51-52' drilling moderately hard
					53-54' drilling hard
		12	SS	2/-/-	55-56.5 Dr. greenish brown SAND medium to coarse, tr. fine gravel, tr. fine sand, saturated
					sand in auger, blow count not valid
		13	SS		59-60.5 Olive gray SAND, fine to coarse, some fine gravel, saturated.
					No detected HNU readings in any SS samples

* A.S.T.M. D1586



NORTON AFB
MW-13

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-14
LOCATION: Waste Pit 4 Site 14
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang
DRILLER: DS
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFb
TOTAL DEPTH 69'
WATER LEVEL: 20.2'
DRILLING METHOD: Auger
DATE DRILLED: 11/8/83
HELPER: HM

SKETCH MAP

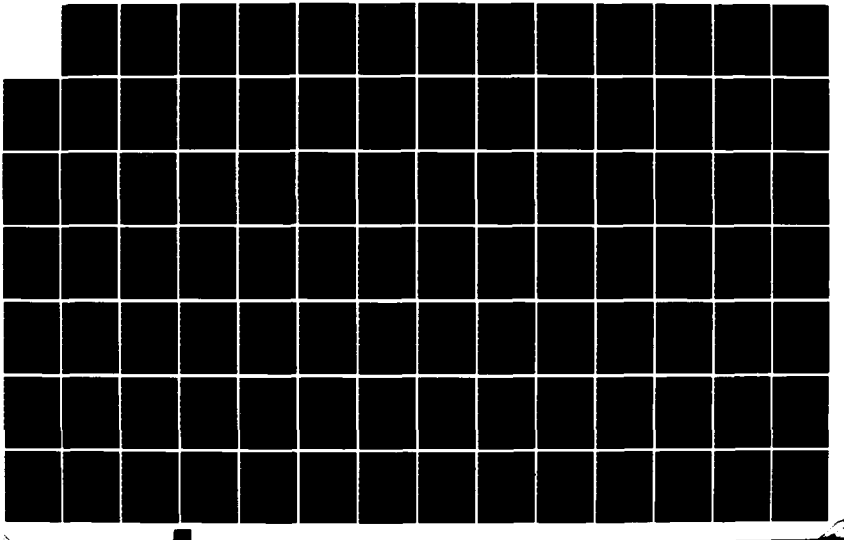
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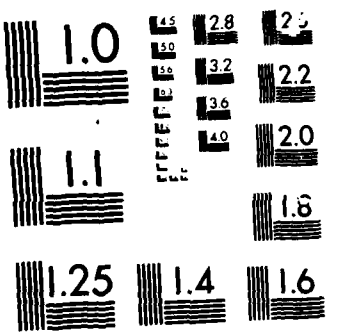
DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0					0-0.5 Black ASPHALT
		1	Aug		0.5-1 Dr. grayish brown SAND, fine, tr. medium to coarse sand, some silt, damp
		2	SS	4/5	4-5.5 Yellowish brown SAND, fine,
5				5	tr. silt, damp, micaceous rec. 1.4
		3	SS	10/13	9-10.5 Dr. Yellow Brown SAND,
				12	medium to coarse, tr. fine gravel
10					tr. fine sand, moist rec. 1.2
		4	SS	15/20	14-15.5 Gray SAND, fine to medium
				23	tr. fine gravel, tr. coarse sandy,
15					damp to moist rec.
20					

* A.S.T.M. D1586

AD-A165 514 INSTALLATION RESTORATION PROGRAM FINAL REPORT PHASE II 2/5
STAGE 1 - PROBLEM..(U) WESTON (ROY F) INC WEST CHESTER
PA JUL 85 F33615-80-D-4006

UNCLASSIFIED F/G 13/2 NL





MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

WESTON

DRILLING LOG

WELL NUMBER: MW-14 OWNER: USAF
LOCATION: Waste Pit 4 Site 14 ADDRESS: Norton AFB
TOTAL DEPTH 69'
SURFACE ELEVATION: WATER LEVEL: 20.2'
DRILLING COMPANY: Stang Hydronics DRILLING METHOD: Auger DATE DRILLED: 11/8/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	9	19-20.5 Grayish brown SAND, fine to coarse, some fine gravel, moist rec. 0.8'
25		6	SS	23/48	24-25.5 Grayish brown SAND, fine, tr. medium sand, moist
				25/1"	
					25-28' hard drilling, cobbles
		7	SS	1/19	29-30.5 Olive gray SAND, fine to medium
				35	tr. fine gravel, tr. coarse sand, tr. silt saturated rec. 1.2'
30					Using bentonite & water to keep hole open
		8	SS	17/23	Very dark gray fine sandy SILT, low plastic,
				26	damp, micaceous with green chlorite mineral
35					
40					

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-14 OWNER: USAF
LOCATION: Waste Pit 4 Site 14 ADDRESS: Norton AFB
TOTAL DEPTH 69'
SURFACE ELEVATION: WATER LEVEL: 20.2'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/9/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	39/-	41-41.5 Grayish brown, olive brown silty
				-	SAND, fine, damp, micaceous
					(might be cave in) rec. 0.5
		10	SS	18/19	44-45.5 Dr. grayish brown silty SAND, fine
45				36	damp, micaceous
					45-47' drilling hard
			SS	23	49-49.8 No recovery
50				33/3"	49.8-52' drilling very hard
			SS	7	53-53.8 no recovery
				36/3"	53.8-53.9 no recovery
			SS	34/1"	
55		11	SS	19/65	56-57.5 Very dark grayish brown
				65	SAND, medium to coarse, tr.
					coarse gravel, tr. fine sand
					tr. silt, wet rec. 1.4

* A.S.T.M. D1586

WESTON

DRILLING LOG

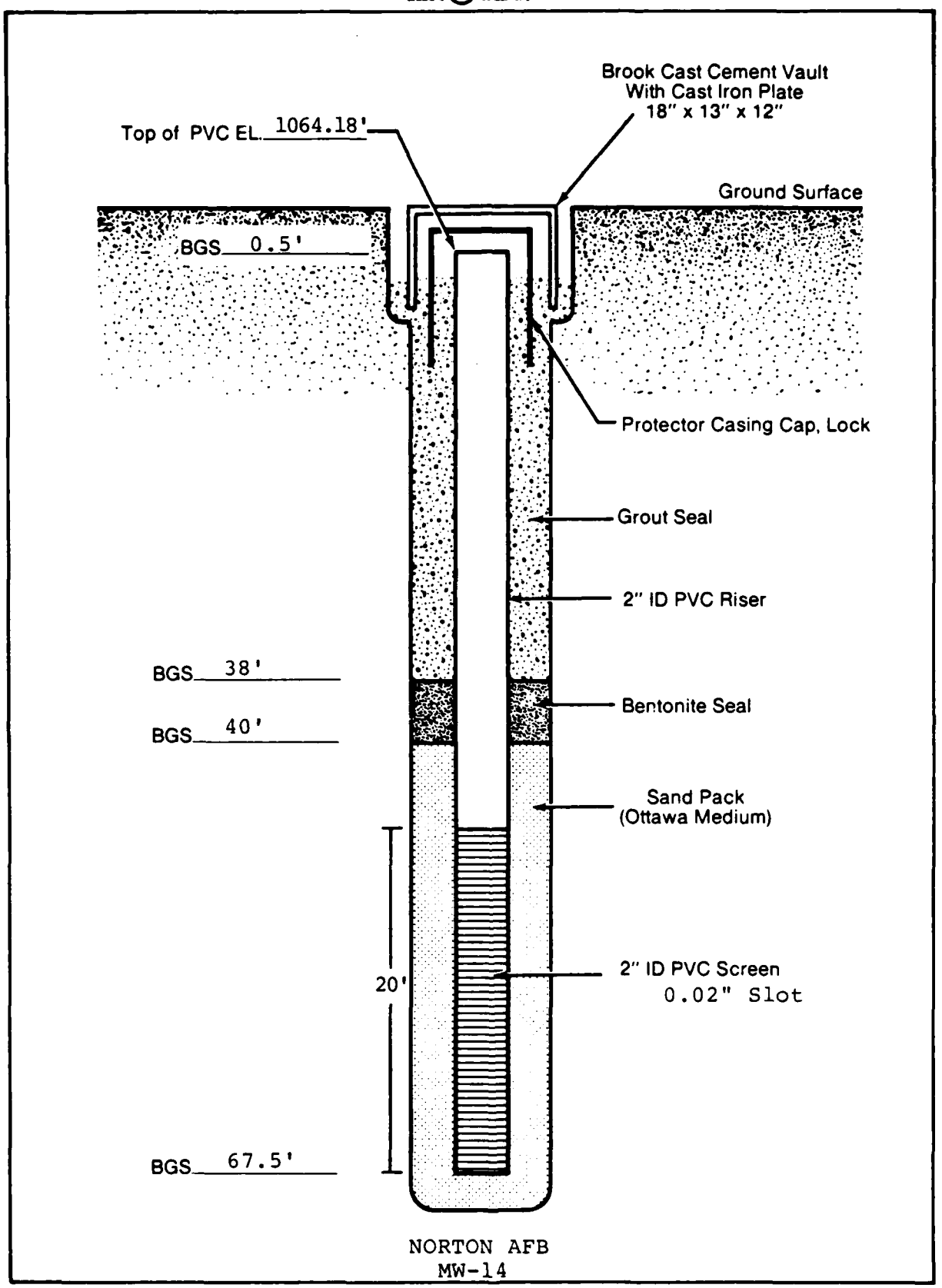
WELL NUMBER: MW-14 OWNER: USAF
LOCATION: Waste Pit 4 Site 14 ADDRESS: Norton AFB
TOTAL DEPTH 69'
SURFACE ELEVATION: WATER LEVEL: 20.2'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/9/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
60		12	SS	23/61	60-61.5 Grayish brown SAND, tr. medium
				50/4"	sand, some silt, moist to wet, micaceous,
					with reddish brown laminations
		13	SS	39	64-65.5 Grayish brown SAND, medium
65					to coarse, tr. fine sand, saturated
					micaceous, with a ¼" black
					fine sand lamination, fine gravel
					on bottom
					65-69' hard drilling
70					69' Auger drilling refusal
					No detectable HNU readings in any split spoon samples

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1064.18'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 38'
Bentonite Seal
BGS 40'
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 67.5'
NORTON AFB
MW-14

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-15 OWNER: USAF
LOCATION: Underground Waste Oil Storage Site 6 ADDRESS: Norton AFB
TOTAL DEPTH 50.5'
SURFACE ELEVATION: WATER LEVEL: 28'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 11/10/83
DRILLER: DS HELPER: HM
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	Split Spoon HNU (ppm)
0					0-0.2' Black ASPHALT	
		1	Aug		0.5-1 Olive brown SAND, fine, tr. medium to coarse, some silt, damp	
5		2	SS	9/11/16	4-5.5 Olive brown, reddish brown SAND fine, tr. medium to coarse sand, some silt damp rec. 1.5	0
					8.5-9 drilling hard coarse gravel	
10		3	SS	7/12/14	9-10.5 Reddish brown SAND, medium tr. fine gravel, tr. coarse sand, tr. fine sand, damp rec. 1.2	0
15			SS	23/1"	14-15.5 Gravel and Cobbles refusal @ 14.1' no recovery	0

* A.S.T.M D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-15
LOCATION: Underground Waste Oil Storage Site 6
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang
DRILLER: DS
LOG BY: BWB

OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 50.5'
WATER LEVEL: 28'
DRILLING METHOD: Auger
DATE DRILLED: 11/10/83
HELPER: HM

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	Split Spoon HNU (ppm)
20		4	SS	27/42	19-20.5 Grayish brown fine gravelly	0
				50	SAND, fine to coarse, damp rec. 1.2	
					22-24 drilling hard	
			SS	87/0"	24' no recovery, spoon bouncing	
25						
30		5	SS	8	29-30.5 Reddish brown SAND, medium	0
					to coarse, moist; Dr. gray SILT,	
					some fine sand, low plastic, damp	
					micaceous rec. 1.0	
					hit water 34.0'	
		6	SS	31	34-35.5 Olive gray SAND, medium	0
35				25/2"	to coarse, tr. fine sand, saturated	
					Using bentonite to keep hole open;	
					losing mud at 35'	
40						

* A.S.T.M D1586

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-15
LOCATION: Underground Waste Oil Storage Site 6
SURFACE ELEVATION: _____
DRILLING COMPANY: Stang
DRILLER: DS
LOG BY: BWB

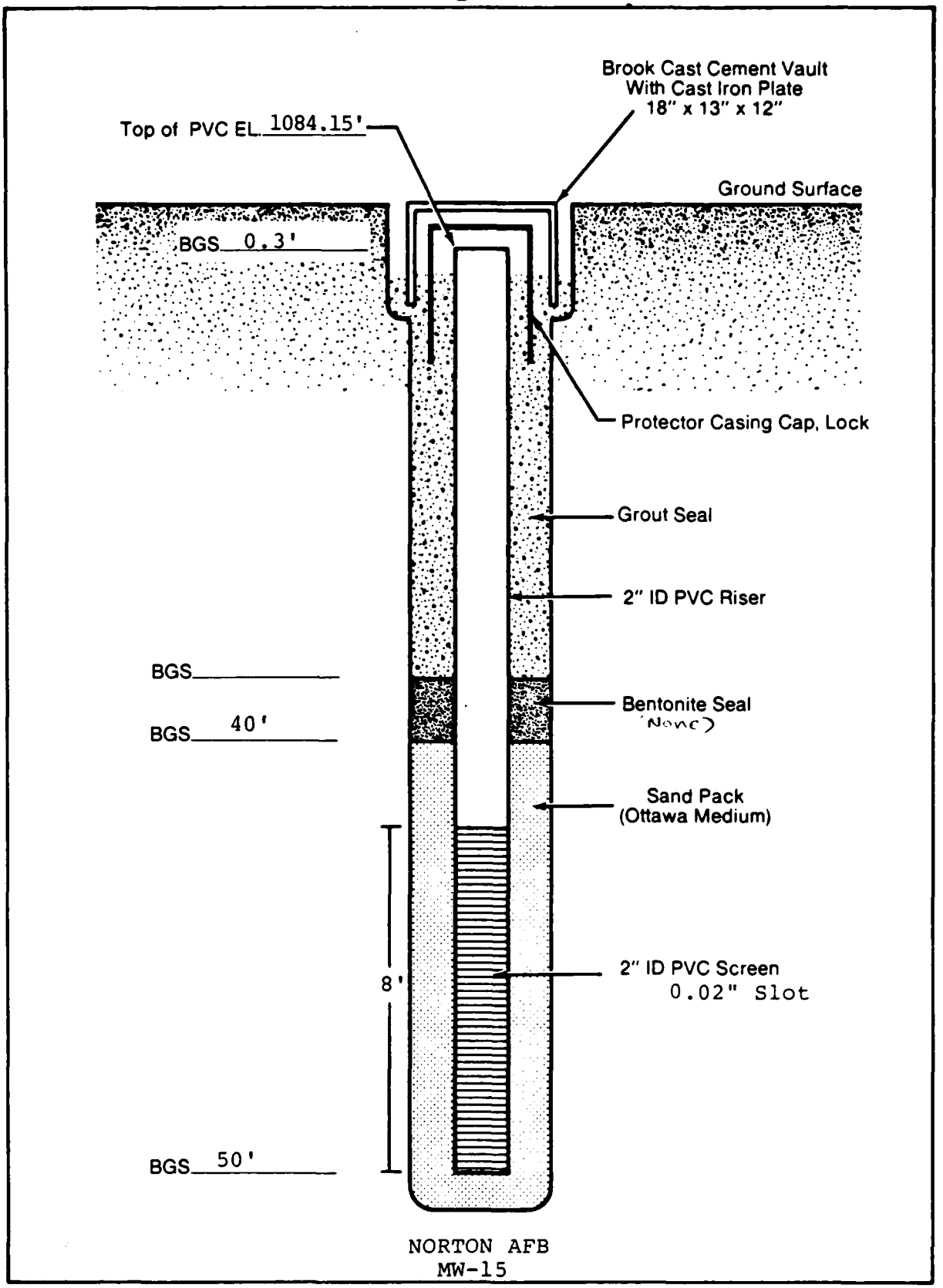
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH: 50.5'
WATER LEVEL: 28'
DRILLING METHOD: Auger
DATE DRILLED: 11/10/83
HELPER: HM

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)	Split Spoon HNU (ppm)
40		7	SS	12/28 21	39-40.5 Dr. Olive gray silty SAND, fine, saturated, micaceous, has fuel/oil odor	20
45		8	SS	13/47 50	44-45.5 Olive gray SAND, medium tr-fine to coarse gravel, some coarse sand, some fine sand, tr. silt, saturated, fuel/oil odor	30
50		9		26	49-50.5 Olive gray SAND, fine to coarse, tr. fine gravel, tr. silt, saturated, with 1" Dr. gray fine sandy silt lense.	4
					Auger refusal at 50.5'	
					Note: Samples 6, 7, 8, & 9 were monitored with the explosimeter: no measurements detected	

* A.S.T.M D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1084.15'
Ground Surface
BGS 0.3'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS
BGS 40'
Bentonite Seal
(None)
Sand Pack
(Ottawa Medium)
8'
2" ID PVC Screen
0.02" Slot
BGS 50'
NORTON AFB
MW-15

WESTON

DRILLING LOG

WELL NUMBER: MW-16
LOCATION: DAVA Evap. Ponds
SURFACE ELEVATION:
OWNER: USAF
ADDRESS: Norton AFb
TOTAL DEPTH: 55.5'
WATER LEVEL: 38.7'
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 5/24/84
DRILLER: DS
HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		1	Aug		1-2' Grayish brown SAND, fine, tr. silt
					dry
					2-3.5' hit cobbles, coarse gravel
		2	SS	9/11	4-5.5 Lt. gray SAND, fine, dry
				50/2"	
		3	SS	35	9-10.5 Gray COBBLES and GRAVEL,
					some coarse sand, dry
					10-13' hitting cobbles, gravel
		4	SS	8/15	14-15.5 Lt. tannish brown SAND, fine,
				31	some silt, damp
					18-19' hitting cobbles

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-16 OWNER: USAF
LOCATION: DAVA EVAP Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55.5'
SURFACE ELEVATION: WATER LEVEL: 38.7'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/24/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		5	SS	13	19-20.5 Dr. olive gray silty SAND, fine,
				50/5"	damp, gravel wedged in sampler
					rec. 0.5'
		6	SS	20	24-25.5 Orangish brown SAND, fine to
				50/2"	medium, tr. coarse, tr. fine gravel
					damp rec. 0.7'
		7	SS	7/19/20	29-30.5 Olive gray to gray SAND, some
					silt, moist
					33.5' Hitting gravel
		8	SS	11	34-35.5 Olive gray SAND, fine to
					coarse, tr. fine/coarse gravel
					moist, with olive gray
					silty clay seam ½", low
					plastic, moist
					hit water at 38.7'

* A.S.T.M. D1586

WESTON

DRILLING LOG

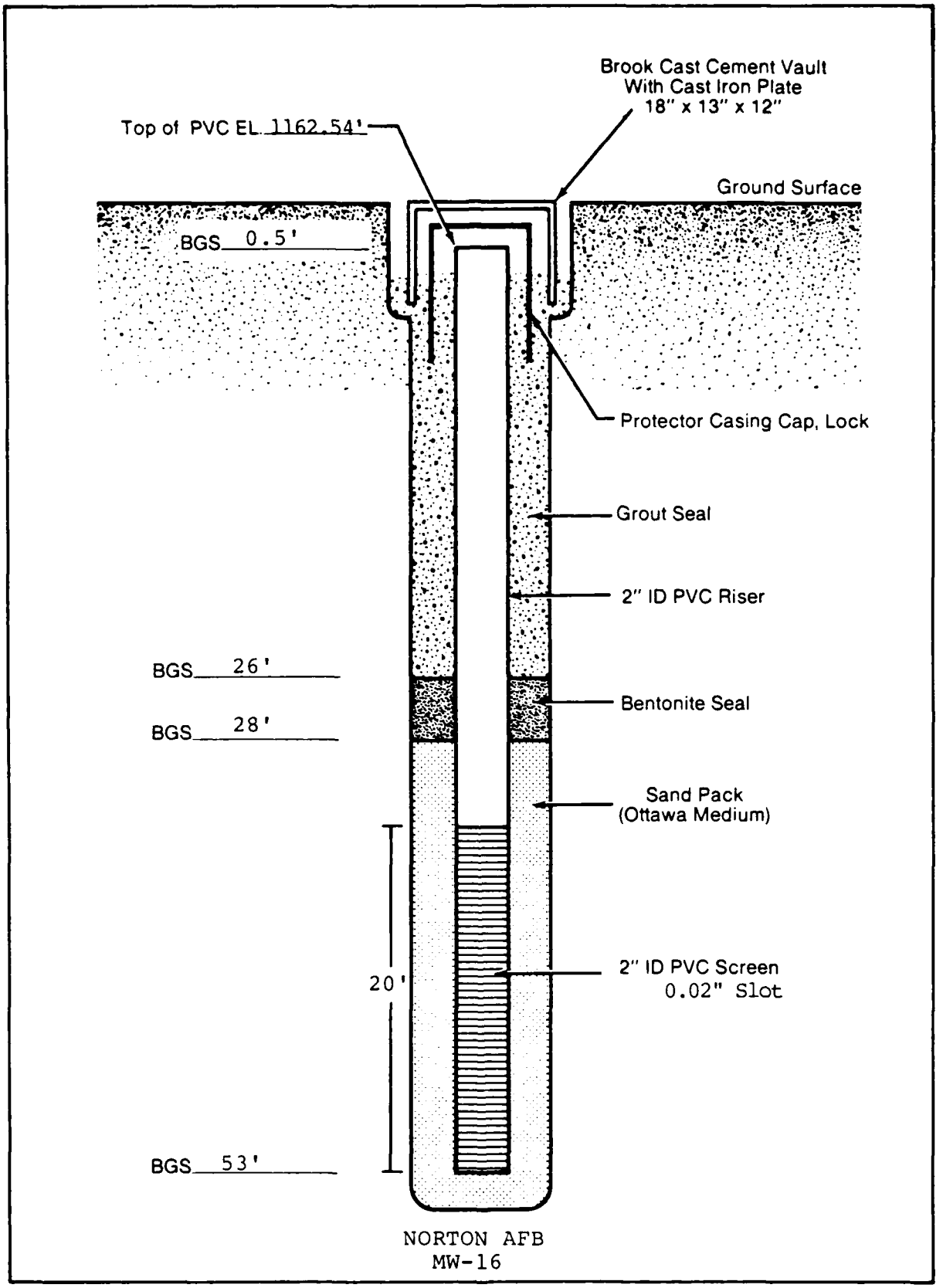
WELL NUMBER: MW-16 OWNER: USAF
LOCATION: DAVA ADDRESS: Norton AFB
EVAP Ponds
TOTAL DEPTH 55.5'
SURFACE ELEVATION: WATER LEVEL: 38.7'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/25/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
		9	SS	3/25	39-40.5' Olive brown SAND, fine
				50	to coarse, tr. fine gravel, tr.
					silt, saturated
					rec. 1.5'
		10	SS	12/26	44-45.5' Olive brown SAND, fine
				30	to coarse, tr. fine gravel, tr.
					silt, saturated
					rec. 1.5'
					46-48' hitting coarse gravel/cobbles
		11	SS	15/41	49-50.5 Olive brown SAND, fine
				50/3"	some fine/coarse gravel, tr.
					silt, saturated
		12	SS	5/15	54-55.5 Olive brown SAND, fine
				50/4"	some fine/coarse gravel, tr.
					silt, saturated
					Note: No detectable HNU readings in any split
					spoon samples

* A.S.T.M D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1162.54'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 26'
BGS 28'
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 53'
NORTON AFB
MW-16

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-17
LOCATION: DAVA
EVAP Ponds
SURFACE ELEVATION: _____
OWNER: USAF
ADDRESS: Norton AFB
TOTAL DEPTH 55.5'
WATER LEVEL: 40.0'
DRILLING COMPANY: Stang
DRILLING METHOD: Auger
DATE DRILLED: 5/24/84
DRILLER: DS
HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-2' Grayish brown SAND, fine, tr. fine gravel, tr. silt, dry
5		2	SS	13/24 31	4-5.5 Orangish brown SAND, fine to coarse, tr. fine gravel, dry
					5-8' Coarse gravel
10		3	SS	26	9-10.5 Orangish brown SAND, fine to coarse, some fine gravel, dry
		4	SS	10/26 30	16-17.5 Lt. gray SAND, fine to coarse tr. silt, damp
15					
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-17 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55.5'
SURFACE ELEVATION: WATER LEVEL: 40.0'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/24/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20					19-23' GRAVEL, coarse
		5	SS	14/30	23-24.5 Lt. gray SAND, fine to medium
25				35	tr. fine/coarse gravel, dry
					25-26 hitting coarse gravel
		6	SS	20	29-30.5 Orangish brown SAND, fine to
30				50/4"	coarse, damp to moist
		7	SS	10/23	34-35.5 Orangish brown SAND, fine to
35				31	medium, moist, with olive brown
					silt seam (½")

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

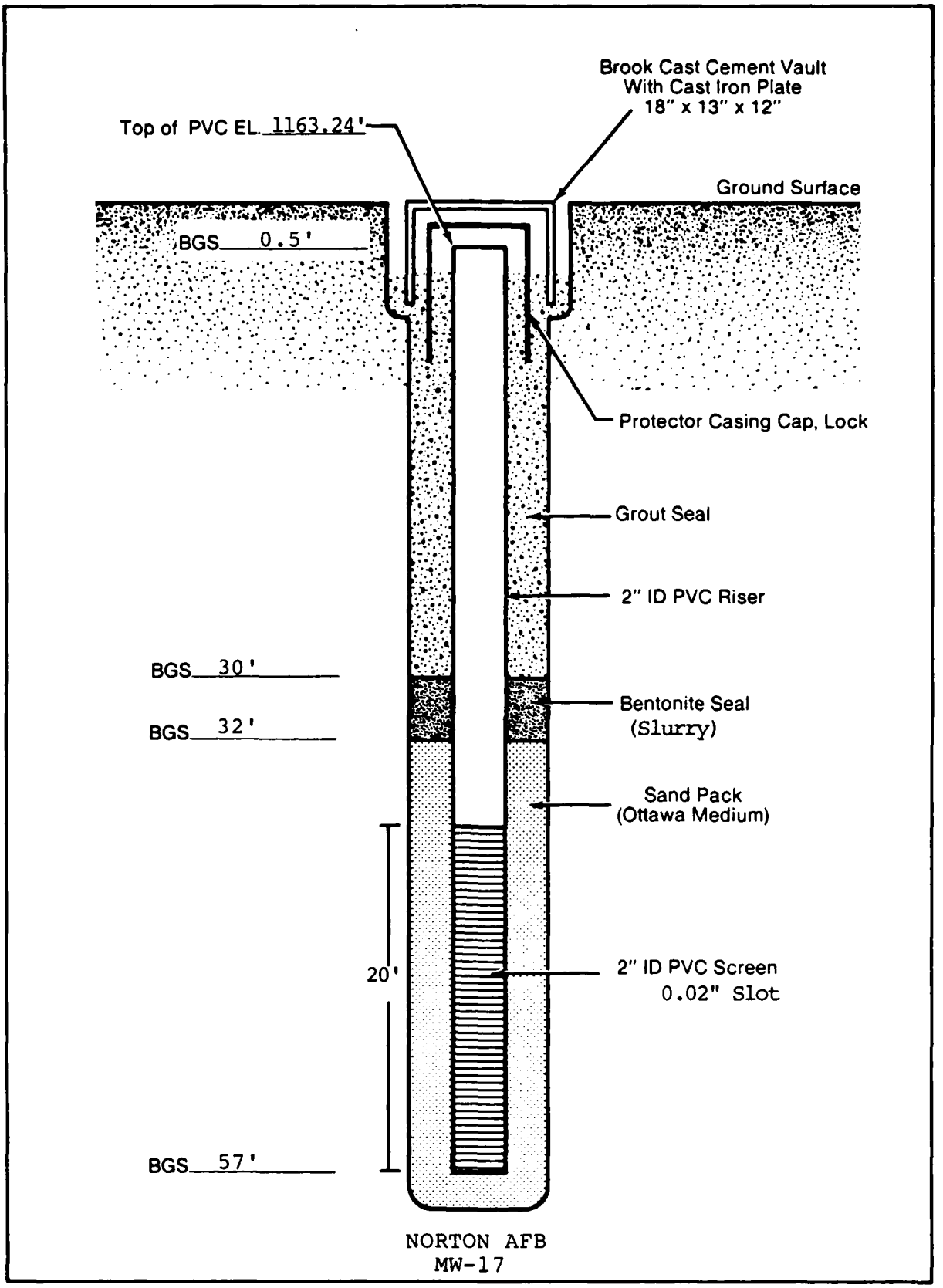
WELL NUMBER: MW-17 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55.5'
SURFACE ELEVATION: WATER LEVEL: 40.0'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED:
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		8	SS	14/25/36	39-40.5 Olive brown SAND, fine to coarse, tr. fine gravel, tr. silt, saturated, hit water at 40.0'
45		9	SS	3/7/20	44-45.5' Olive brown SAND, fine to coarse, tr. fine gravel, tr. silt, saturated, increase in gravel
50		10	SS	3 / 50/2"	49-50.5 Olive brown SAND, fine to coarse, tr. fine gravel, tr. silt saturated
55		11	SS	27/20/50	54-55.5 Olive brown SAND, fine to coarse, tr. fine gravel, tr. silt saturated
					Note: No detectable HNU readings in any split spoon samples

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1163.24'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 30'
BGS 32'
Bentonite Seal
(Slurry)
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 57'
NORTON AFB
MW-17

WESTON

DRILLING LOG

WELL NUMBER: MW-18 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 56.5'
SURFACE ELEVATION: WATER LEVEL: 41.7'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/18/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		2-3 Tan SAND, fine, tr. silt, dry
					@ 4.5' hitting cobbles
		2	Aug		6-7' COBBLES, granite
5					out of cobbles at 9'
		3	SS	8/9/14	10-11.5 Lt grayish brown SAND, fine to coarse, some fine/coarse gravel, tr. silt, dry
10					12-14' hitting cobbles
		4	SS	6/16/12	14-15.5 Lt. gray SAND, fine to medium, some gravel, tr. silt, dry rec. 1.0
15					
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-18 OWNER: USAF
LOCATION: DAVA ADDRESS: Norton AFB
Evap Ponds
TOTAL DEPTH 56.5'
SURFACE ELEVATION: WATER LEVEL: 41.7'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/23/84
DRILLER: DS HELPER: DT

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	13/38	19-20.5 Lt. brown SAND, fine to coarse,
				35	damp rec 1.5'
					23-23.5 hitting gravel
25		6	SS	20	24-25.5 Lt. brown SAND, fine to coarse,
				50/5"	damp, cobble zone
					25-26' hitting cobbles
		7	SS	19/30	29-30.5 Lt. orangish brown SAND, fine to
30				34	coarse, tr. gravel, damp.
					Lt. brown SAND, tr. silt, damp
		8	SS	22/35	34-35.5 Lt. orangish brown SAND, fine
35				30/3"	to coarse, tr. fine gravel, damp
					rec. 1.1'
40					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

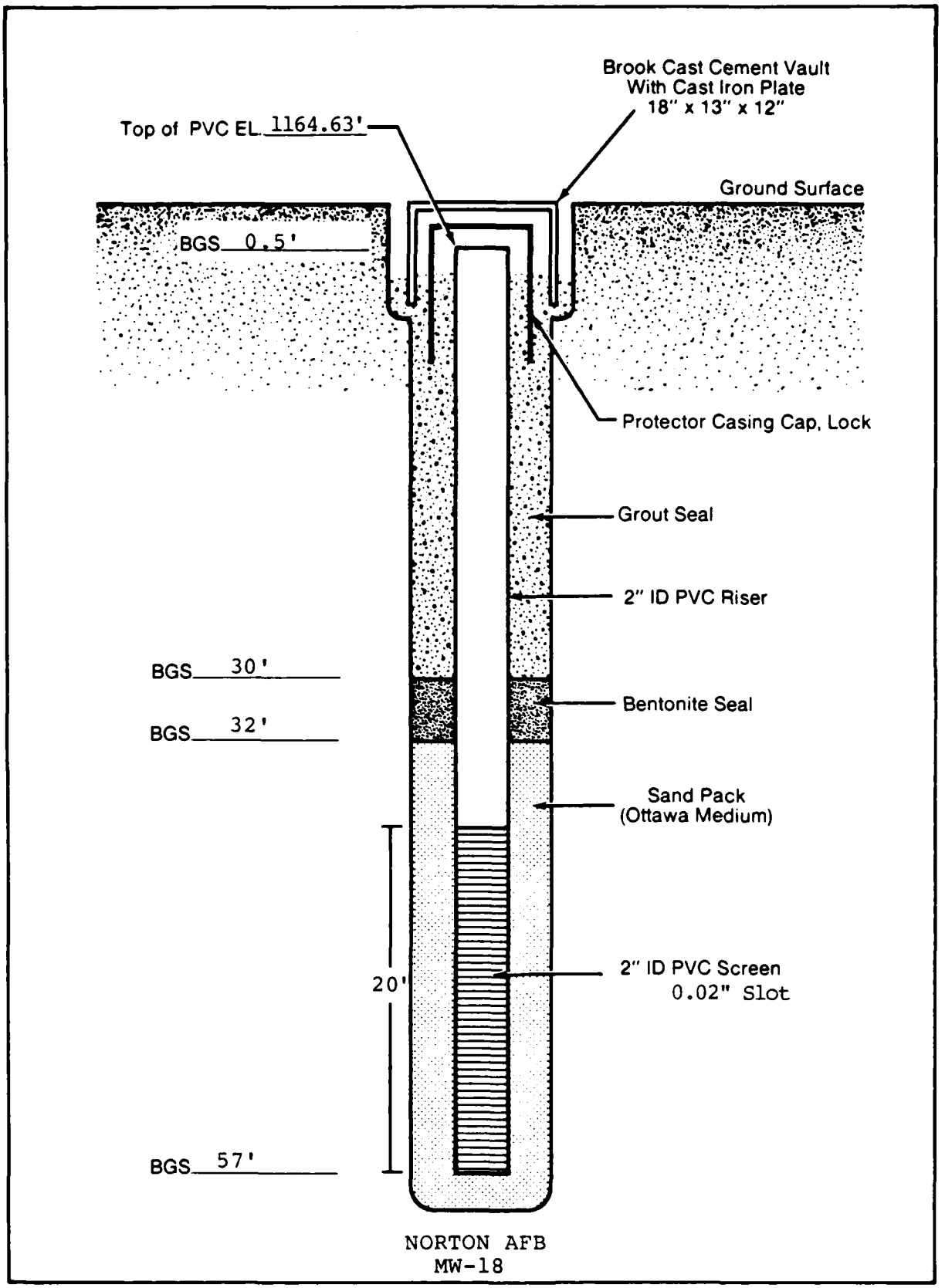
WELL NUMBER: MW-18 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 57'
SURFACE ELEVATION: WATER LEVEL: 41.7'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/23/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	17	39-40.5 Orangish brown SAND, fine
				36/35	to coarse, tr. fine gravel, wet
					hit water at 41.7'
45		10	SS	3/24	44-45.5 Orangish brown SAND, fine to
				43	coarse, tr. fine gravel, wet, with
					one fine sand lense 3-4"
50		11	SS	3/10	50-51.5 Orangish brown SAND, medium
				8	to coarse, tr. fine gravel, saturated
55		12	SS	5/15	55-56.5 Olive gray SAND, fine to
				17	medium, some fine gravel, tr. silt, saturated
					Note: No detectable HNU readings in any
					split spoon samples

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1164.63'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 30'
Bentonite Seal
BGS 32'
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 57'
NORTON AFB
MW-18

WESTON

DRILLING LOG

WELL NUMBER: MW-19 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55'
SURFACE ELEVATION: WATER LEVEL: 39.3'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/17/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-2' Grayish brown SAND, fine, to coarse sand, dry
					at 2' hitting cobbles
5		2	Aug		5-6' COBBLE zone - using stinger bit to advance auger
				29/27	
10		3			9.5-11' Lt. brown SAND, fine to coarse, some fine/coarse gravel damp
					rec. 1.2
15					at 14' hitting cobbles
		4	Aug		14' hitting cobbles - drilling hard

* A.S.T.M. D1586

WESTON

DRILLING LOG

WELL NUMBER: MW-19 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55'
SURFACE ELEVATION: WATER LEVEL: 39.3'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/17
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	50/4"	19-21.5 refusal @ 19.4'
					Brown SAND and coarse GRAVEL,
					damp
					19.4-23 cobbles - drilling hard
25		6	SS	31/40	24-25.5 Grayish brown SAND, fine to
				32	coarse, some fine, coarse gravel,
					damp rec. 1.5'
		7	SS	20/22	29-30.5 Grayish brown SAND, fine to
30				27	coarse, some fine/coarse gravel,
					damp, with one olive gray silt
					seam (½")
35		8	SS	25	34-35.5 Reddish brown SAND, fine to coarse,
					some fine/coarse gravel,
					moist
					34.9-36' and 37-38' drilling hard
40					hitting gravel

* A.S.T.M D1586

WESTON

DRILLING LOG

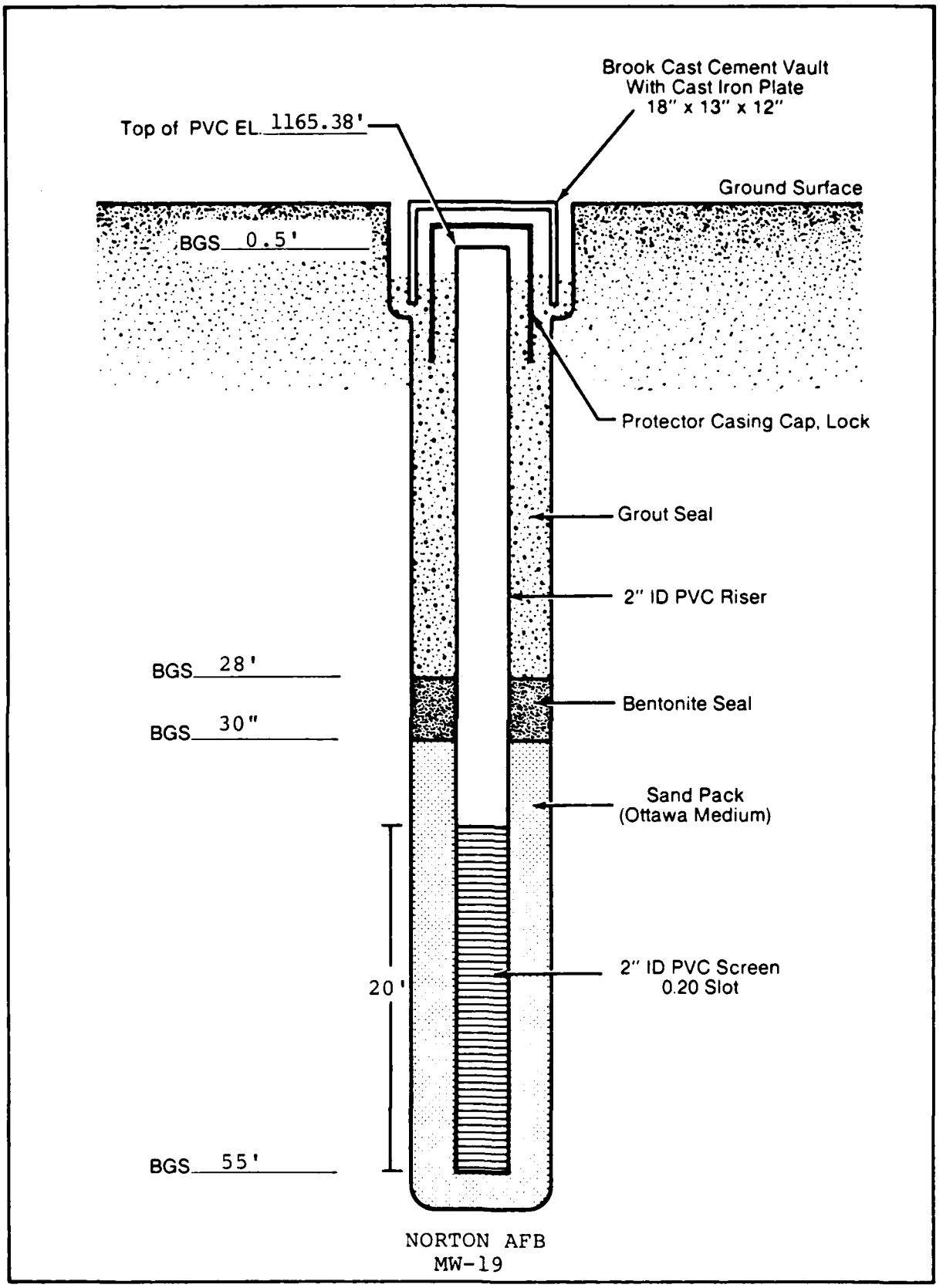
WELL NUMBER: MW-19 OWNER: USAF
LOCATION: DAVA Evap Ponds ADDRESS: Norton AFB
TOTAL DEPTH 55'
SURFACE ELEVATION: WATER LEVEL: 39.3'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/18/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		9	SS	3/19	39-40.5 Grayish brown SAND, fine to coarse, tr. fine gravel, tr. silt, saturated rec. 1.5
					43.5-44.5 hitting coarse gravel
		10	SS	20/30	45-46.5
45				50	Reddish brown SAND, fine, tr. silt saturated rec. 1.5'
					47' hitting coarse gravel
		11	Aug		52' GRAVEL, coarse, subround; with some sand matrix
50					
		12	Aug		GRAVEL, coarse, subround; with some sand matrix
55					No HNU reading exceeding 0.5 ppm.

* A.S.T.M D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1165.38'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 28'
BGS 30"
Bentonite Seal
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.20 Slot
BGS 55'
NORTON AFB
MW-19

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-20 OWNER: USAF
LOCATION: IWTP SFSS ADDRESS: Norton AFB
TOTAL DEPTH 30.5'
SURFACE ELEVATION: WATER LEVEL: 13.8'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/21/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-2 Brown SAND, fine, tr. silt, damp
5		2	SS	7/32 29	4-5.5 Olive gray SAND, fine to coarse, some coarse gravel, damp rec. 0.8'
10		3	SS	4/8 12	9-10.5 Dr. olive gray fine sandy SILT, tr. clay, moist
15		4	SS	8/12 15	14-15.5 Dr. olive gray silty SAND, fine to coarse, saturated
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

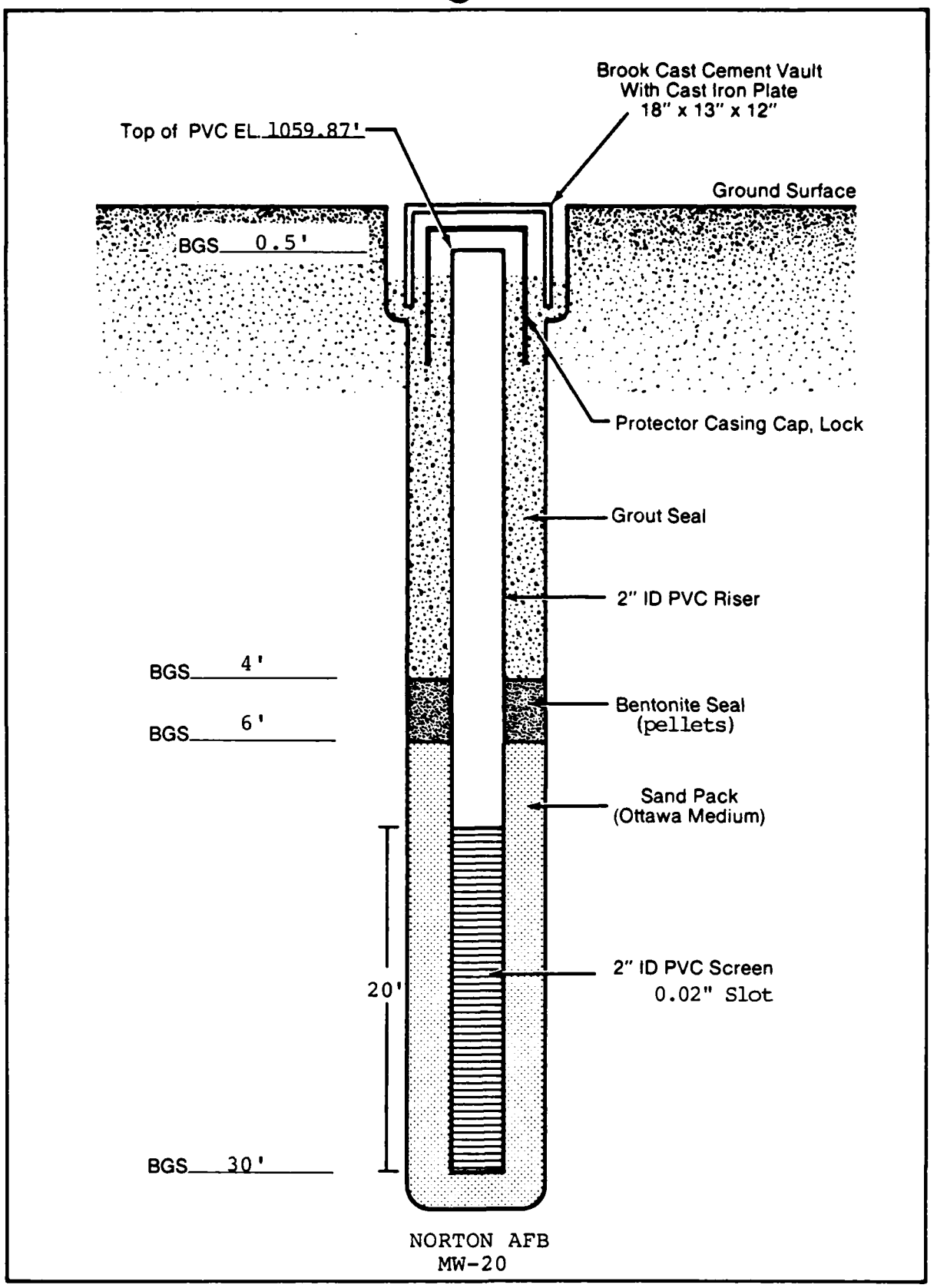
WELL NUMBER: MW-20 OWNER: USAF
LOCATION: IWTP SFSS ADDRESS: Norton AFB
TOTAL DEPTH 30.5'
SURFACE ELEVATION: WATER LEVEL: 13.8'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/21/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	7/28	19-20.5 Olive gray SAND, coarse, tr. fine gravel, saturated, grades to Dr. olive gray SAND, fine, tr. fine gravel, saturated
25		6	SS	15/12 50	24-25.5 Dr. Olive gray SAND, fine to medium, tr. silt, saturated
30		7	SS	5/12 29	29-30.5 Dr. gray SAND, fine to medium, some silt, saturated
35					No detectable HNU reading in any split spoon sample
40					

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL. 1059.87'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 4'
BGS 6'
Bentonite Seal
(pellets)
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 30'
NORTON AFB
MW-20

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-21 OWNER: USAF
LOCATION: IWTP ADDRESS: Norton AFB
Treatment Discharge
TOTAL DEPTH 32'
SURFACE ELEVATION: WATER LEVEL: 16.0'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/21/84
DRILLER: DS HELPER: DT

LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*		DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0		1	Aug		0-1	Brown SAND, fine, tr. silt, damp
		2	SS	5/12	4-5.5	Lt. gray SAND, fine to medium,
5				21		tr. fine gravel, damp
						rec. 1.0'
					6.5-8.0'	Hitting coarse gravel
		3	SS	6/7	9-10.5	Lt. gray SAND, fine to medium,
				9		moist, Olive gray sandy SILT, moist
10						rec. 1.3'
		4	SS	4/10	14-15.5	Dr. olive gray silty SAND, fine
15				15		to medium, moist to wet;
						Dr. olive gray fine sandy SILT, wet
						rec. 1.5

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

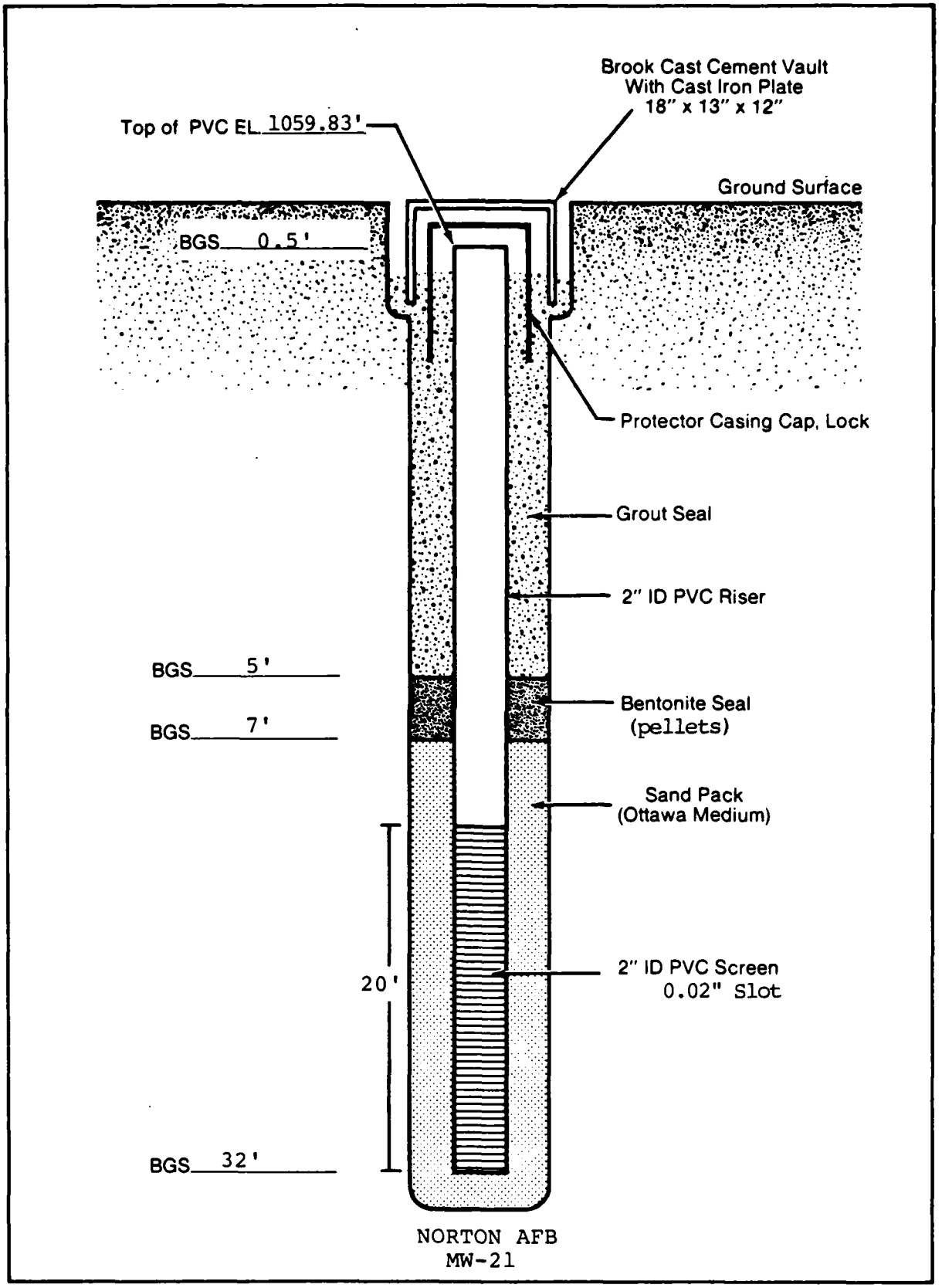
WELL NUMBER: MW-21 OWNER: USAF
LOCATION: IWTP Treatment Discharge ADDRESS: Norton AFB
TOTAL DEPTH 32'
SURFACE ELEVATION: WATER LEVEL: 16.0'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/21/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		5	SS	5/18 50+	19-20.5 Olive brown SAND, fine to medium, tr. coarse, saturated; Olive gray SAND, fine to medium, tr. fine gravel, tr. silt, saturated
25		6	SS	8/25 21	24-25.5 Olive brown SAND, fine to medium, tr. coarse, saturated sand grades siltier on bottom of spoon
30		7	SS	5/30 36	30-31.5 Dr. gray SAND, fine to medium, tr. coarse, tr. silt, saturated
					Note: No detectable HNU reading in split spoon samples
35					

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1059.83'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 5'
BGS 7'
Bentonite Seal
(pellets)
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 32'
NORTON AFB
MW-21

WESTON
DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-22 OWNER: USAF
LOCATION: IWTP ADDRESS: Norton AFB
TOTAL DEPTH 40.5'
SURFACE ELEVATION: WATER LEVEL: 22.8'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/22/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
0			Aug		0-2' Lt. gray SAND, fine to medium, tr. silt, dry
5		1	SS	1/2	4-5.5 Gray/reddish brown SAND, fine
				17	to medium, tr. gravel, tr. silt, dry
10		2	SS	3/3	9-10.5 Lt. gray SAND, fine to
				4	medium, tr. fine gravel, tr. silt,
					damp
15		3	SS	12/22	14-15.5 Lt. gray SAND, fine to medium,
				35	tr. fine gravel, tr. silt,
					damp to moist
20					

* A.S.T.M. D1586

WESTON DESIGNERS CONSULTANTS

DRILLING LOG

WELL NUMBER: MW-22 OWNER: USAF
LOCATION: IWTP ADDRESS: Norton AFB
TOTAL DEPTH 40.5'
SURFACE ELEVATION: WATER LEVEL: 22.8'
DRILLING COMPANY: Stang DRILLING METHOD: Aguer DATE DRILLED: 5/22/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
20		4	SS	12/24	19-20.5 Dr. olive gray SAND, fine, tr.
				25	fine gravel, some silt, moist
					water at 22.8'
		5	SS	10/30	24-25.5 Olive brown SAND, fine to
25				35	medium, tr. coarse, saturated
					rec. 1.5
		6	SS	4/5	29-30.5 Dr. gray SAND, fine, tr.
30				19	medium/coarse, tr. silt, saturated
					rec. 1.0
		7	SS	5/16	34-35.5 Dr. gray SAND, fine to coarse,
35				40	saturated, Dr. gray silty SAND, fine
					saturated rec. 1.5
40					

* A.S.T.M. D1586

WESTON

DRILLING LOG

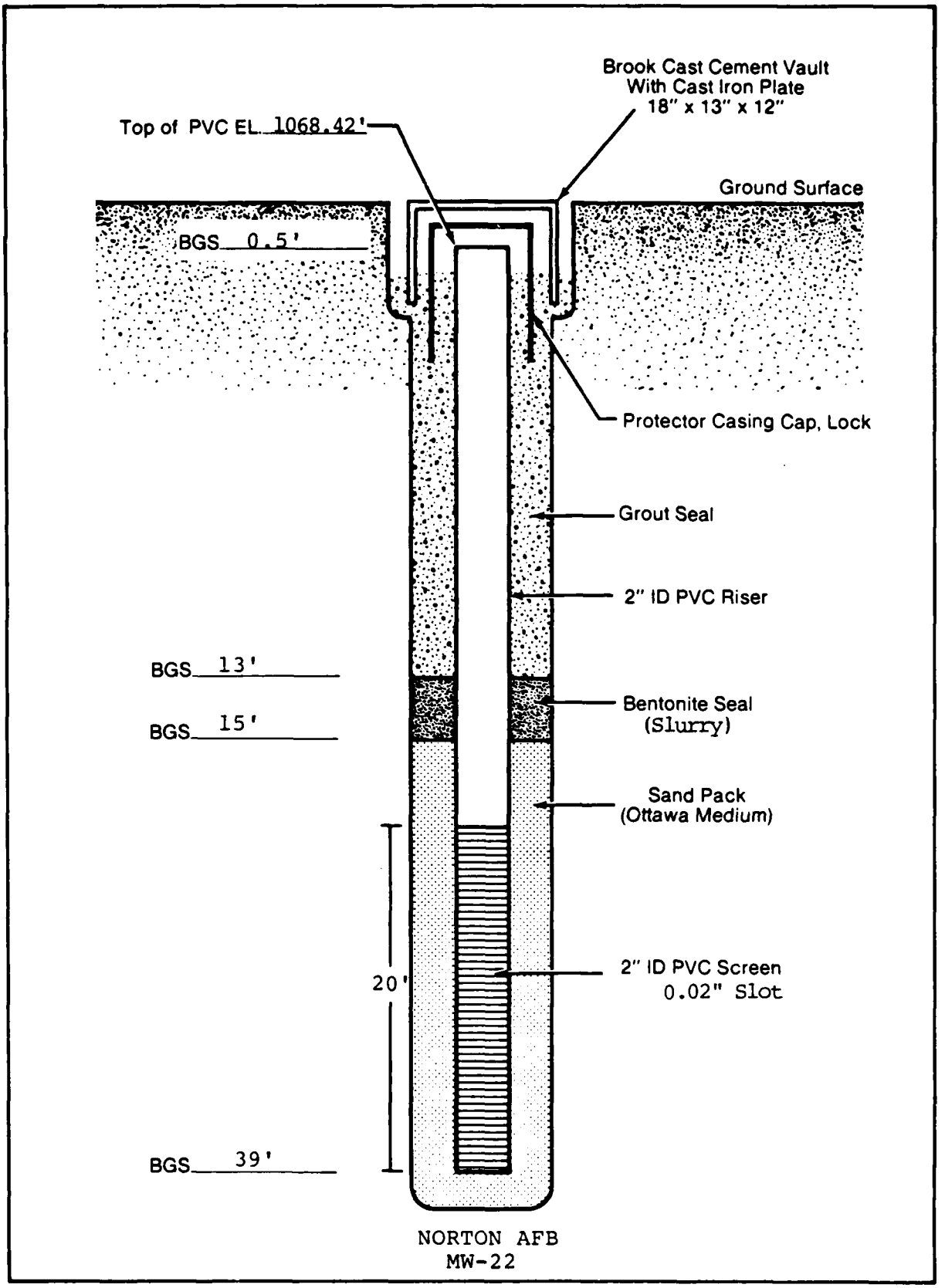
WELL NUMBER: MW-22 OWNER: USAF
LOCATION: IWTP ADDRESS: Norton AFB
TOTAL DEPTH 40.5'
SURFACE ELEVATION: WATER LEVEL: 22.8'
DRILLING COMPANY: Stang DRILLING METHOD: Auger DATE DRILLED: 5/22/84
DRILLER: DS HELPER: DT
LOG BY: BWB

SKETCH MAP

NOTES:

DEPTH (FEET)	GRAPHIC LOG	SAMPLE NUMBER	SAMPLE TYPE	SAMPLE BLOWS*	DESCRIPTION / SOIL CLASSIFICATION (COLOR, TEXTURE, STRUCTURES)
40		8	SS	8/20/32	39-40.5 Dr. gray SILT, tr. fine sand, tr. clay, moist to saturated, with brown organic fibers
					No detectable HNU readings in any split-spoon samples.

* A.S.T.M. D1586


Brook Cast Cement Vault
With Cast Iron Plate
18" x 13" x 12"
Top of PVC EL 1068.42'
Ground Surface
BGS 0.5'
Protector Casing Cap, Lock
Grout Seal
2" ID PVC Riser
BGS 13'
BGS 15'
Bentonite Seal
(Slurry)
Sand Pack
(Ottawa Medium)
20'
2" ID PVC Screen
0.02" Slot
BGS 39'
NORTON AFB
MW-22

APPENDIX F

FIELD SAMPLING AND QA/QC PLAN

APPENDIX F

FIELD SAMPLING AND QA/QC PLAN

F.1 SOIL SAMPLING

All soil sampling accomplished using a drill rig will follow the Standard Penetration Test (ASTM Method 1586) using a steel split-spoon sampler. Prior to taking each sample, the following procedures will be followed:

1. The split-spoon sampler will be washed thoroughly with an Alconox and water solution, and rinsed in tap water from the Base-approved source for drilling.

2. After assembly of the sampler, the sampler will be lowered into the boring and the sample taken by the Standard Penetration Test Method.

3. Upon recovery of the sampler, the spoon will be split and the sample examined for soil characteristics.

4. The sample will then be cleaned of any smeared sample around the outside of the sampler, and the cleaned, representative sample will be put in a marked and labelled 1 pint clear glass sampling jar with a screw cap.

F.2 GROUNDWATER SAMPLING

In general, groundwater samples will be collected from pump discharge after pumping 3 well volumes from each well. Prior to initiation of well pumping, however, a small volume of sample will be collected from the top of the water column for analysis of oils and greases. The parameters to be sampled for and appropriate containers are described in separate attachments.

All monitor wells will be pumped using a Johnson-Keck stainless-steel submersible pump connected to a Teflon discharge line. The Johnson-Keck Model SP-81 Sampling Pump is a 1.5-inch diameter, all stainless-steel, Archimedes-screw

impeller submersible pump capable of a steady discharge of about 1 gpm. The pump and discharge line will have been completely decontaminated (including purging with a detergent, nitric acid and distilled water) prior to first use at the site.

Between monitor wells, the equipment will be decontaminated by flushing the inside and hosing the outside with approximately 20 gallons of potable water from a Base source (use the same source throughout the sampling). At the end, approximately 3-5 gallons of distilled or deionized water will be flushed through the pump.

The following procedure will be followed in sampling each well:

1. Measure the SWL with reference to the measuring point marked on the top of the casing.

2. Lower a bottom-loading Teflon bailer slowly down to the air-water interface, and draw off the top 6 inches, approximately, of the water column. Transfer this sample into the container for oils and greases analysis until it 3/4 full.

3. Using Table 1, calculate the volume of standing water in the well.

4. Lower the pump and begin pumping. Record the pumping rate and total time to pump at least 3 well volumes.

5. At the end of this time, decrease the pumping rate, if necessary, and begin sampling.

6. Collect grab samples for immediate measurement of temperature, pH and specific conductance.

7. Gently fill each sample container from the pump line, taking care to avoid aeration or turbulence of the sample water. All containers should be filled completely (taking care not to spill preservatives if they are predosed) except for the bottles for oil and

grease analysis, which are to be only 3/4 full.

8. Filter 750-1000 ml of sample using a field filtering apparatus with as little exposure to air as possible. Transfer the filtered sample to an appropriate container and add sufficient nitric acid to lower pH of sample below 2 (approximately 2 ml).

9. Wrap the sample containers in protective packaging and pack with ice in a thermal chest to insure cooling to 4°C.

F.3 BOTTOM SEDIMENT SAMPLING

The parameters to be analyzed are listed in a separate attachment and consist of VOA's (including MEK) and phenols.

1. Fully decontaminate the core sampler and PVC insert prior to use (including rinsing in a detergent solution and distilled water).

2. Row to the middle of the pond and sound pond with a weight and tape measure.

3. Drop the core sampler to the bottom, allowing it to sink into the bottom sediment by force of gravity.

4. Send messenger down the line to close top of sampler and create suction.

5. Gently pull up on the sampler to remove the core from the pond bottom.

6. Remove the PVC insert and quickly transfer a small portion of the sample to 3 glass vials with as little disturbance as possible, using a stainless-steel blade or scoop if necessary. Close the Teflon-lined crimp-top immediately.

7. Transfer the remainder of the sample to a wide mouth amber glass jar and cap immediately.

F.4 SURFACE WATER SAMPLING

The parameters to be analyzed and appropriate containers are described in separate attachments.

1. Fully decontaminate the closing sampler before each use including rinsing in a detergent solution, nitric acid solution, and distilled water.

2. Row to the middle of the pond and sound pond depth with a weight and tape measure. Place a marker on the sampler line so that it will be lowered to 1 foot from the pond bottom.

3. Lower the sampler to 1 foot from the pond bottom. Send messenger down the line to close the sampler.

4. Gently fill each sampler container by pouring from the sampler, taking care to avoid any excessive aeration of the sample. All containers should be completely filled except for the bottles for oils and greases, which are to be only 3/4 full.

5. Refill the sampler at the same depth, and row to shore keeping the sampler closed.

6. Filter 750-1000 ml of the remaining sample and transfer to an appropriate container. Preserve with nitric acid.

7. Use the rest of the sample to make field determination of temperature, pH, and specific conductance.

F.5 FISH TISSUE SAMPLING

Fish will be sampled exclusively for the determination of metals uptake/body burden. Actual tissue samples to be digested and analyzed will be fillets of muscle tissue excluding bone. Keep this in mind when selecting actual samples for analysis.

APPENDIX G

FIELD SAMPLE LOG SHEETS

1. Collect a minimum of four fish from each of the three ponds, using a Smith-Root Backpack Electroshocker and/or 25 foot seine.

2. Collect both bottom (e.g. carp, suckers) and predatory (e.g. bass, sunfish) feeders if available (two each). Preferentially select fish in 6-10 inch size range if available.

3. Wrap each whole fish in separate plastic bags. Include label containing information on date and location of capture and sampling method.

4. Cool samples on ice, and freeze as soon as possible. Maintain frozen until analysis.

5. Identify and enumerate all fish captured along with samples held for each species. Return to water unharmed.

F.6. QC SAMPLES

Approximately 20% additional samples will be collected for the purpose of validating field and analytical techniques. These will include 2 field blanks (1 associated with groundwater, 1 with surface water), 2 duplicate groundwater samples and 1 duplicate bottom sediment sample.

The field blanks will consist of distilled water collected using methods and equipment the same as, or as close as possible to, those used in actual sample collection: e.g. distilled water will be pumped from a clean glass jar through the pump and line to obtain the field blank associated with groundwater sampling, and distilled water will be poured into the closing sampler and from there into sample containers to obtain the field blank associated with surface water sampling. Duplicates will be collected as separate samples, not splits of a single sample.

In addition, a trip blank of distilled water in two 30 ml glass vials with Teflon-lined septa will accompany each ice chest when it leaves the laboratory. This blank will not be manipulated during sampling, and will be returned un-opened with the ice chest as it is shipped back to the laboratory.

Field Sample Log

Project Norton

Site	Date	Type	Sampler
MW #7	7,19,84	Pumped (O+G) Grab	NLR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	0612	0635	0635	

	Before Purge	Other Total	Notes
Water Levels	34.39	56.98	

	Calculated	Other
Purge Volume	3.8 x 3 = 11.4 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	0800	7.3	20.7	659	

	Duplicate Sample Number	Cleaning Water Sample Taken Yes	No
Special Items			

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0098
Bailer seen in well about 3-4 ft. down.
did not attempt to lower pump 7-12-84 straightened well
so pump would lower properly Sampled 7-13-84

Site	Date	Type	Sampler
MW 7	7 19 84	Pumped (O+G) Grab	NLR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	0642		0653	

	Before Purge	Other Total	Notes
Water Levels	38.62	59.87	

	Calculated	Other
Purge Volume	3.5 x 3 = 10.5 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	1500	7.16	20.5	684	

	Duplicate Sample Number	Cleaning Water Sample Taken Yes	No
Special Items			

Notes TOC TOX UOA, O+G, Pb + other metals
#0096

WESTON

Field Sample Log

Project Norton A+B

Site: MW17		Date: 7.11.84	Type: ~~Pumped~~	O+G Grab	Sampler: N/B
Times	Start Purge: 0859	Stop Purge:	Sample Taken: 0908	Notes:	
Water Levels	Before Purge: 40.86	~~Other~~ total: 56.16		Notes:	
Purge Volume	Calculated: 2.7×3 = 8.1			Other:	
Water Conditions	Time:	PH: 6.65	Temp: 20.5	Conduct: 782	Notes:
Special Items	Duplicate Sample Number:		Cleaning Water Sample Taken: Yes	No	

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0092

Site: MW18		Date: 7.11.84	Type: ~~Pumped~~	O+G Grab	Sampler: N/B
Times	Start Purge: 0924	Stop Purge: 0932	Sample Taken:	Notes:	
Water Levels	Before Purge: 41.59	~~Other~~ total: 57.11		Notes:	
Purge Volume	Calculated: 2.7×3 = 8.1			Other:	
Water Conditions	Time:	PH: 6.59	Temp: 20.1	Conduct: 627	Notes:
Special Items	Duplicate Sample Number:		Cleaning Water Sample Taken: Yes	No	

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0093

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type	O+G	Sampler
MW4	7.9.84	Pumped	Grab	NB

Times	Start Purge	Stop Purge	Sample Taken	Notes
	1930	1944	19	

Water Levels	Before Purge	Other ~~total~~	Notes
	32.66	58.93	

Purge Volume	Calculated	Other
	4.4 x 3 = 13.2 gal	

Water Conditions	Time	PH	Temp	Conduct	Notes
		7.21	22.2	514	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
	X			(No)

Notes TOC, TOX, UOA-MEK, O+G, Phenol
Pb + other metals

#0082

Site	Date	Type	O+G	Sampler
MW10	7.10.84	Pumped	Grab	MD

Times	Start Purge	Stop Purge	Sample Taken	Notes
	2026		2034	

Water Levels	Before Purge	~~Other~~ total	Notes
	18.47	31.26	

Purge Volume	Calculated	Other
	2.2 x 3 = 6.6 gal	

Water Conditions	Time	PH	Temp	Conduct	Notes
		~~6.74~~ 7.01	18.3	525	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No

Notes TOC, TOX, UOA ~~Phenol~~, O+G, Pb + other metals

#0097

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type	Sampler
MW 11	7,10,84	Pumped (O+G) Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1920	~~1~~	1927	

	Before Purge	Other	Notes
Water Levels	45.83	Other Total 57.82	

	Calculated	Other
Purge Volume	2.1 x 3 = 6.3 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		6.88	20.2	342	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items	#0063 MW23			

Notes TOC, TOX, UOA, O+G, Li Pb + other metals

#0062

Site	Date	Type	Sampler
MW12	7.10 84	Pumped (O+G) Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1741		1749	

	Before Purge	~~Other~~ Total	Notes
Water Levels	44.78	58.43	

	Calculated	Other
Purge Volume	2 x 3 = 7.2 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		6.97	20.0	251	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA, O+G, (Li, Pb + other metals)

0086

WESTON

Field Sample Log

Project Loring AFB

Site	Date	Type	Sampler
MW 2	7.9.84	Pumped / ~~Grab~~ O+G	NLR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1050		1113	

	Before Purge	Other ~~Total~~	Notes
Water Levels	41.50	87.46	

	Calculated	Other
Purge Volume	7.6 x 3 = 22.8 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.17	20.8	510	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA-MEK, O+G, Phenol, Pb + other metals
#0077

Site	Date	Type	Sampler
MW 5	7.9.84	(Pumped) / ~~Grab~~ O+G	NLR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1355	1408		

	Before Purge	Other	Notes
Water Levels	32.27	57.26	

	Calculated	Other
Purge Volume	4.1 x 3 = 12.3	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.11	20.4	584	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items	[redacted]			

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0078

WESTON

Field Sample Log

Project Norton AFB

Site MW #3 Norton AFB		**Date** 7,8,84	**Type** ~~Pumped~~ Grab O+G		**Sampler** N/B
Times	**Start Purge** 17:55	**Stop Purge** 18:00 →	**Sample Taken**	**Notes**	
Water Levels	**Before Purge** 18.40	**Other** Total Depth 27.29		**Notes**	
Purge Volume	**Calculated** 1.5 x 3 = 4.5 gal.			**Other**	
Water Conditions	**Time** 18:00	**PH** 7.81	**Temp** 31°C	**Conduct** 1480	**Notes**
Special Items	**Duplicate Sample Number** no		**Cleaning Water Sample Taken** Yes	(No)	

Notes TOC, TOX, UOA - mek, O+G, Phenol
Pb + other metals
#0056

Site Norton MW #1		**Date** 7.8.84	**Type** (Pumped)	(O+G Grab)	**Sampler** N/B
Times	**Start Purge** 1845	**Stop Purge** 18.54 →	**Sample Taken**		
Water Levels	**Before Purge** 25.53	**Other**		**Notes** Total Depth 42.55	
Purge Volume	**Calculated** ~~7.82~~ 3 x 2.9 = 8.7			**Other**	
Water Conditions	**Time**	**PH** 6.81	**Temp** 20.8	**Conduct** 674	**Notes**
Special Items	**Duplicate Sample Number**		**Cleaning Water Sample Taken** Yes	No	

Notes TOC, TOX, UOA - mek, O+G, Phenol
Pb + other metals
#0061

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type	Sampler
MW 6	7.19.84	Pumped / (O+G) Grab	N/R

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1802		1810	

	Before Purge	~~Other~~	Notes
Water Levels	39.75	Total 52.26	

	Calculated	Other
Purge Volume	2.2 X 3 = 6.6 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.41	22.8	714	

	Duplicate Sample Number	Cleaning Water Sample Taken
Special Items	#0079 MW24 ~~sent to UOA~~	Yes / (No)

Notes TOC, TOX, UOA, O+G, PB + other metals
#0080

Site	Date	Type	Sampler
MW 8	7.19.84	Pumped / (O+G) Grab	N/R

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1855	1908 →		

	Before Purge	~~Other~~	Notes
Water Levels	32.87	Total 52.80	

	Calculated	Other
Purge Volume	4.1 X 3 = 12.3 gal ~~3 8 X 3 = 11.4 gal~~	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.27	22.5	5000	

	Duplicate Sample Number	Cleaning Water Sample Taken
Special Items		Yes ☐ No ☐

Notes TOC, TOX, UOA O+G, Phenols, Cn
PB + other metals

Field Sample Log

Project North Hill

Site Morton MW #20		Date 7/9/84	Type Pumped / ~~Grab~~		Sampler NR
Times	Start Purge 9:39	Stop Purge 9:51	Sample Taken 9:48	Notes	
Water Levels	Before Purge 12.30	~~Other~~ Total 28.14		Notes	
Purge Volume	Calculated 2.7 x 3 = 8.1			Other	
Water Conditions	Time	PH 7.24	Temp 21.2	Conduct 464	Notes
Special Items	Duplicate Sample Number ✓		Cleaning Water Sample Taken Yes / No		

Notes TOC, TOX, UOA, O+G, Pb + other metals #0075
Diazanon being sprayed — meter box filled with water

Site MW #21		Date 7.9.84	Type Pumped / ~~Grab~~		Sampler NR
Times	Start Purge 1012	Stop Purge ~~1020~~	Sample Taken 1020	Notes	
Water Levels	Before Purge 14.57	~~Other~~ Total 29.25		Notes	
Purge Volume	Calculated 2.5 x 3 = 7.5 gal			Other	
Water Conditions	Time	PH 7.40	Temp 21.3	Conduct 366	Notes
Special Items	Duplicate Sample Number		Cleaning Water Sample Taken Yes / No		

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0076

WESTON

Field Sample Log

Project Norton AFB

Site MW19		Date 7,11,84	Type Pumped / (O+G Grab)		Sampler N/B
Times	Start Purge 0801	Stop Purge	Sample Taken 0809	Notes	
Water Levels	Before Purge 40.87	~~Other~~ total 53.94		Notes	
Purge Volume	Calculated 2.4 x 3 = 7.2 gal			Other	
Water Conditions	Time 1500	PH 6.47	Temp 20.3	Conduct 423	Notes
Special Items	Duplicate Sample Number		Cleaning Water Sample Taken Yes	No	

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#00094

Site MW22		Date 7 11 84	Type Pumped / (O+G Grab)		Sampler N/B
Times	Start Purge 606	Stop Purge	Sample Taken 615	Notes	
Water Levels	Before Purge 22.62	~~Other~~ total 37.71		Notes	
Purge Volume	Calculated 2.7 x 3 = 8.1			Other	
Water Conditions	Time 1500	PH 6.53	Temp 20.0	Conduct 802	Notes
Special Items	Duplicate Sample Number		Cleaning Water Sample Taken Yes	No	

Notes TOC, TOX, UOA, O+G, Pb + other metals

#00095

WESTON DESIGNERS CONSULTANTS

Field Sample Log Project _____

Site	Date	Type		Sampler
MW15	/ /	~~Type~~ Pumped	O+G Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	11??		1120	

	Before Purge	~~Other~~ total	Notes
Water Levels	33.50	49.26	

	Calculated	Other
Purge Volume	2.7 x 3 = 8.1 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	1500	7.02	21.6	1177	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA, O+G, Pb

#0089

Site	Date	Type		Sampler
MW16	7/11/94	~~Type~~ Pumped	O+G Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	0831	0837 →		

	Before Purge	~~Other~~ total	Notes
Water Levels	40.15	50.65	

	Calculated	Other
Purge Volume	1.9 x 3 = 5.7 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	1500	6.53	21.4	1637	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items	#0091 MW25			

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0090

WESTON

Field Sample Log

Project Norton

Site	Date	Type (Pumped)	(O+G / Grab)	Sampler
MW13	7/10/84		O+G	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1828		1833	

	Before Purge	Other Tot'l	Notes
Water Levels	48.96	52.00	

	Calculated	Other
Purge Volume	1.4 x 3 = 4.2 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		6.94	18.2	365	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0087

Site	Date	Type (Pumped)	(O+G / Grab)	Sampler
MW 14	7/11/84		O+G	N/R

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1015		1035	

	Before Purge	Other Tot'l	Notes
Water Levels	27.12	66.65	

	Calculated	Other
Purge Volume	6.6 x 3 = 19.8 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	1500	6.75	21.8	1053	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items			☐	☐

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0088

APPENDIX H

SAMPLE CHAIN-OF-CUSTODY RECORDS

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone: _____

SHIP TO: Weston (Westech) Stockton

ATTENTION: _____

Phone No. _____

SHIPPING INFORMATION

Site: Norton AFB

Shipper: samples

Address: _____

Date Shipped: 2-11-84

Shipment Service: Fed Exp.

Airbill No. _____

Cooler No. _____

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0062-UOA	MW11	2-10-84	UOA	
0063-UOA	MW23			
0086	MW12			
0087	MW13			
0097	MW10	↓		
0088	MW14	2-11-84		
0089	MW15			
0090	MW16			
0091	MW25			
0092	MW17			
0093	MW18			
0094	MW19			
0095	MW22			
0096	MW9			
0099	S-0637			
0100	S-0635			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F. Weston (Stkn)
7220 Lorraine Ave #102
Stockton Ca 95336

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sample

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	Oil + Grease	
0097	MW 10	7-10-84		
0062	MW 11	7-10-84		
0086	MW 12	7-10-84		
0087	MW 13	7-10-84		
0088	MW 14	7-11-84		
0089	MW 15	7-11-84		
0090	MW 16	7-11-84		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Nackels

Phone: (209) 957-3405

SHIP TO:

Roy F. Weston
7220 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bogner		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	TOC	
0097	MW 10	7-10-84		
0062	MW 11			
0086	MW 12			
0087	MW 13			
0088	MW 14	7-11-84		
0089	MW 15			
0096	MW 9		TOX	
0097	MW 10	7-10-84		
0062	MW 11			
0086	MW 12			
0087	MW 13			
0088	MW 14	7-11-84		
0089	MW 15			
0090	MW 16		TOC	
0092	MW 17			
0090	MW 16		TOX	
0092	MW 17			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) Maggie Neckels
Phone: (209) 957-3405

SHIP TO:
Roy F. Weston
7720 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:
Phone No.

SHIPPING INFORMATION

Site Norton AFB
Shipper Samples
Address
Date Shipped 7-11-84
Shipment Service Fed Exp.
Airbill No.
Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bagnar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0093	MW 18	7-11-84	TOC	
0094	MW 19	7-11-84		
0095	MW 22	7-11-84		
0063	MW 23	7-10-84		
2091	MW 25	7-11-84		
0093	MW 18	7-11-84	TOX	
0094	MW 19	7-11-84		
0095	MW 22	7-11-84		
0063	MW 23	7-10-84		
0091	MW 25	7-11-84		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F. Weston
2220 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0081	MW 8	7-9-84	Cyanides	
0090	MW 16	7-11-84		
0092	MW 17			
0093	MW 18			
0094	MW 19			
0091	MW 25			

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) Maggie Neckels
Phone: (209) 957-3405

SHIP TO:
Roy F Weston
Weston Way
Weschester PA

ATTENTION:
Phone No.

SHIPPING INFORMATION
Site Norton AFB
Shipper Samples
Address
Date Shipped 7-11-84
Shipment Service Fed. Exp.
Airbill No.
Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0092	MW17	7-11-84	Disolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0093	MW18	↓	↓	
0094	MW19			
0095	MW22	↓		
0063	MW23	7-10-84	Disolved Metals Li, Pb, Cr, Ni, Cd, As Zn, Cu, Hg	
0091	MW25	↓	Disolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels

Phone: (201) 857-3405

SHIP TO:

Roy F Weston

Weston Way

Weschester PA.

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bagnar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	Dissolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0097	MW 10	7-10-84	Dissolved Metals Li, Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0062	MW 11	↓	↓	
0086	MW 12			
0087	MW 13	↓	Dissolved Metals Pb, Cr, Ni Cd, As, Zn, Cu, Hg	
0088	MW 14	7-11-84	↓	
0089	MW 15	↓		
0090	MW 16			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels
Phone: (209) 957-3405

SHIP TO:
Roy F Weston (Stkn)
1720 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:
Phone No.

SHIPPING INFORMATION
Site Norton AFB
Shipper Samples
Address
Date Shipped 7-11-84
Shipment Service Fed Exp.
Airbill No.
Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0092	MW17	7-11-84	Oil + Grease	
0093	MW 18	7-11-84		
0094	MW 19	7-11-84		
0095	MW22	7-11-84		
0063	MW 23	7-10-84		
0091	MW 25	7-11-84		

White · Sampler Canary · Return Copy To Shipper Pink · Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) John Bognar

Phone:

SHIP TO:
Ray F Weston
Weston Way
Westchester PA

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-13-84

Shipment Service Fed Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John De Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0098	MW7	7-13-84	TOC TOX Dissolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) John D Bognar

Phone:

SHIP TO:
Roy F Weston
2720 Lorraine Ave #102
Stockton Ca

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-13-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condit Upon Receipt
0098	MW7	7-13-84	VOA	
↓	↓	↓	Oil + Grease	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

WESTON ENGINEERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nichols

Phone: (209) 957-3405

SHIP TO:

Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sample

Address

Date Shipped 2-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075 (x3)	MW20	2-9-84	UOA	
0076	MW21			
0078	MW5			
0080	MW6			
0077	MW2		UOA + MEK	
0081	MW8			
0082	MW4			
0084	S-0630			
0085	S-0625			

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WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nebb

Phone:

SHIP TO: Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site: Norton AFB

Shipper: sampler

Address:

Date Shipped: 7-9-84

Shipment Service: Fed. Exp

Airbill No.

Cooler No. 212

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nebb		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0053W (x3)	Pond 1-W	7-6-84	VOA + MEK	
0054	Pond 1-S	↓	↓	
0055	Pond 1-S1	↓	↓	
0056	MW-3	7-8-84	↓	
0057	Pond 2-S	7-7-84	↓	
0058	Pond 3-S	↓	↓	
0059	Pond 2-W	↓	↓	
0060	Pond 3-W	↓	↓	
0061	MW-1	7-8-84	↓	
0071	ER-1	↓	↓	
0072	TB#S-0633	X	↓	
0073	TB#S-0646			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Neclaels

Phone: (209) 957-3405

SHIP TO:

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper samplers

Address

Date Shipped 7-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neclaels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075 Ofb	MW20	7-9-84	Oil and Grease	
0076	21			
0077	2			
0078	~~15~~			
0079	24			
0080	6			
0081	8			
0082 ✓	4	✓		

White · Sampler Canary · Return Copy To Shipper Pink · Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Nechels

Phone: (209) 957-3405

SHIP TO:
Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper samplers

Address

Date Shipped 7-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nechels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0077-Phenols	MW2	7-9-84	Phenols (preserved H_3PO_4-$CuSO_4$)	
0081-Phenols	MW8		↓	
0082-Phenols	MW4		↓	
0075-TOC	MW20		as labled	
0075-TOX	↓			
0076-TOC	MW21			
0076-TOX	↓			
0077-TOC	MW2			
0077-TOX	↓			
0078-TOC	MW5			
0078-TOX	↓			
0079-TOC	MW24			
0079-TOX	↓			
0080-TOC	MW6			
0080-TOX	↓			
0081-TOC	MW8			
0081-TOX	↓			
0082-TOC	MW4			
0082-TOX	↓	↓		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone: (209) 9573405

SHIP TO:
Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper samplers

Address

Date Shipped 7-10-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075-Metals	MW20	7-9-84	Dissolved Metals	
0076 Metals	MW21		Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0077 Metals	MW2			
078 Metals	MW5			
0079 Metals	MW24			
080 Metals	MW6			
0081 Metals	MW8			
082 Metals	MW4			

samples filtered and acidified (HNO_3) in field

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Neebels

Phone:

SHIP TO: Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sample

Address

Date Shipped 7-9-84

Shipment Service Fed Exp

Airbill No.

Cooler No. 1 of 2

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neebels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
053W	Pond 1-W	7-6-84	Oil & Grease	
059	Pond 2-W	7-7-84	↓	
060	Pond 3-W	↓		
2056	MW3	7-8-84		
0061	MW1	↓		
2071	FB1			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Neebels

Phone: (209) 9573405

SHIP TO:

Weston - WestChester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sampler

Address

Date Shipped 7-9-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neebels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0058W-Metals	Pond1-W	7-6-84	Dissolved Metals Pb, Ni, Cd, As, Zn, Cr, Cu, Hg	
0058W-Phenols	↓	↓	↓	
0058W-TOC	↓	↓	↓	
0058W-TOX	↓	↓	↓	
0059-Metals	Pond2-W	7-7-84	↓	
0059-Phenols	↓	↓	↓	
0059-TOC	↓	↓	↓	
0059-TOX	↓	↓	↓	
0060-Metals	Pond3-W	↓	↓	
0060-Phenols	↓	↓	↓	
0060-TOC	↓	↓	↓	
0060-TOX	↓	↓	↓	
			metals filtered and acidified (HNO_3) in field	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nichols

Phone: (209) 957-3405

SHIP TO: Weston - Westchester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper MSM

Address Weston - Sth.

Date Shipped 7-9-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No. X

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0054	Pond 1-S	7-6-84	Phenols	
0055	Pond 1-SI	↓	↓	
0057	Pond 2-S	7-7-84		
0058	Pond 3-S	↓		

please return rec sheet + blue air to Stockton office

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CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Neckels

Phone: (209) 9573405

SHIP TO:
Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton

Shipper Sampler

Address

Date Shipped 7-9-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neckels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0056-Metals	MW#3	7-8-84	Dsolved Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0056-TOC	↓	↓		
0056-TOX	↓	↓		
0061-Metals	MW#1	↓		
0061-TOC	↓	↓		
0061-TOX	↓	↓		
0071-Metals	FB-1	↓		
0071-TOC	↓	↓		
0071-TOX	↓	↓		
0056-Phenols	MW#3	↓	(metals filtered and acidified (HNO_3) in field	
0061-Phenols	MW#1	↓		
0071-Phenols	FB-1	↓		

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APPENDIX I

LABORATORY ANALYTICAL METHODS

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Purgeable Halocarbons—Method 601

1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Bromodichloromethane	32101	75-27-4
Bromoform	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	100-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Dichlorodifluoromethane	34668	75-71-8
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethene	34501	75-35-4
trans-1,2-Dichloroethene	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropene	34704	10061-01-5
trans-1,3-Dichloropropene	34699	10061-02-6
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethane	34511	79-00-5
Trichloroethene	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39175	75-01-4

1.2 This is a purge and trap gas chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identification should be supported by at least one additional qualitative

technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse cap with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15-mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated backing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel, 7.7 gm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should

not be heated higher than 180 °C and the remaining sections should not exceed 220 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long × 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long × 0.1 in ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120) mesh or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope. The electrolytic conductivity detector was used to develop the method performance statements and MDL listed in Tables 1 and 2. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25 μL, 0.006 in ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon (Filtrasorb-300 or equivalent (Calgon Corp.)).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials

6.3.1 Coconut charcoal (6/10 mesh sieved to 26 mesh), (Barnaby Chaney, CA-580-26 lot # M-2649 or equivalent).

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on 60/80 mesh Chromosorb-W or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methyl Alcohol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methyl alcohol using assayed liquids or gas cylinders as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methyl alcohol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methyl alcohol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methyl alcohol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methyl alcohol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, in Cincinnati, Ohio.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the method detection limit (See Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 hours, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after one hour.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard, according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1 } RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard.
C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in methyl alcohol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 μL of the check sample concentrate to each of a minimum of four 5-mL aliquots of reagent water. A representative waste-

water may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3.1 Calculate upper and lower control limts for method performance:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R − 3s

where R and s are calculated as in Section 8.2.3. The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly.[7]

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 11.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should maintain constant surveillance of both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as above, add a volume to give 7500 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume (15 ng/μL). If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2). Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. USEPA methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.

10. Sample Extraction and Gas Chromatography

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this Table are estimated retention times and method detection limits that can be achieved by this method. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the

syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0 ± .1 minutes at ambient temperature.

10.7 After the 11-minute purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If rapid heating of the trap cannot be achieved, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C.

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for four minutes recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately seven minutes turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool the trap is ready for the next sample.

10.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for the peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of material from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration $\mu g/L = (A_s C_{is})/(A_{is})(RF)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_s = Concentration of the internal standard.

11.2 Report results in micrograms per liter. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

11.3 For samples processed as part of a set where the spiked sample recovery falls outside of the control limits which were established according to Section 8.3, data for the affected parameters must be labeled as suspect.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL up to 1000 × MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000 × MDL.

12.3 In a single laboratory (Monsanto Research), using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 2 were obtained.[9] The standard deviation of the measurement in percent recovery is also included in Table 2[9].

12.4 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1. See Appendix A.
2. Bellar, T.A., and Lichtenberg, J.J. *Journal American Water Works Association, 66,* 739, (1974).
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8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, U.S. Environmental Protection Agency,

Environmental Monitoring and Support Laboratory—Cincinnati, Ohio 45268, March 1979.
9. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (In preparation).

Table 1. *Chromatographic Conditions and Method Detection Limits*

Parameter	Retention Time (min.) Column 1	Retention Time (min.) Column 2	Method Detection Limit µg/L
Chloromethane	1.50	5.28	0.08
Bromomethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.67	5.28	0.18
Chloroethane	3.33	8.68	0.52
Methylene chloride	5.25	10.1	0.25
Trichlorofluoromethane	7.18	nd	nd
1,1-Dichloroethene	7.93	7.72	0.13
1,1-Dichloroethane	9.30	12.6	0.07
trans-1,2-Dichloroethene	10.1	9.38	0.10
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
trans-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethene	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
cis-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13
Bromoform	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethene	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3-Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15
1,4-Dichlorobenzene	35.4	22.3	0.24

nd = not determined

Column 1 conditions: Carbopack B 60/80 mesh coated with 1% SP-1000 packed in an 8 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45°C for 3 min. then programmed at 8°C/min. to 220° and held for 15 min.

Column 2 conditions: Porasil-C 100/120 mesh coated with n-octane packed in a 6 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50°C for 3 min then programmed at 6°C/min to 170° and held for 4 min.

Table 2. *Single Operator Accuracy and Precision*

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (µg/L)	Number of Analyses	Matrix Types
Bromodichloromethane	100.9	5.0	0.43-46.7	21	3
Bromoform	89.5	9.0	1.45-50	20	3
Bromomethane	105.0	17.3	3.39-49.2	21	3
Carbon tetrachloride	82.5	25.6	0.55-50	19	3
Chlorobenzene	93.9	8.9	2.21-50	20	3
Chloroethane	91.5	22.4	3.95-50	21	3
2-Chloroethylvinyl ether	96.3	9.9	4.39-133	20	3
Chloroform	101.7	20.6	0.44-50	20	3
Chloromethane	91.4	13.4	0.55-23.9	21	3
Dibromochloromethane	98.3	6.5	0.75-93.0	21	3
1,2-Dichlorobenzene	10.20	2.0	4.89-154	21	3
1,3-Dichlorobenzene	91.6	4.3	2.94-46.7	21	3
1,4-Dichlorobenzene	97.5	9.3	2.99-51.6	21	3
Dichlorodifluoromethane	87.8	18.0	2.18-43.4	21	3
1,1-Dichloroethane	102.3	5.5	0.44-46.7	21	3
1,2-Dichloroethane	97.8	4.8	0.44-46.7	21	3
1,1-Dichloroethene	101.1	21.7	0.37-50	19	3
trans-1,2-Dichloroethene	91.0	19.3	0.44-98.0	20	3
1,2-Dichloropropane	97.7	8.8	0.29-39.0	21	3
cis-1,3-Dichloropropene	86.7	6.0	0.44-46.7	21	3
trans-1,3-Dichloropropene	73.5	17.2	0.43-50	20	3
Methylene chloride	97.9	2.6	0.73-46.7	21	3
1,1,2,2-Tetrachloroethane	91.9	15.0	0.46-46.7	21	3
Tetrachloroethene	94.1	18.1	0.50-35.0	21	3
1,1,1-Trichloroethane	75.1	12.5	0.37-29.0	21	3
1,1,2-Trichloroethane	91.0	25.1	0.45-50	21	3
Trichloroethene	106.1	7.4	0.38-46.7	21	3
Trichlorofluoromethane	89.3	13.9	149	14	2
Vinyl chloride	101.9	11.4	0.82-32.3	21	3

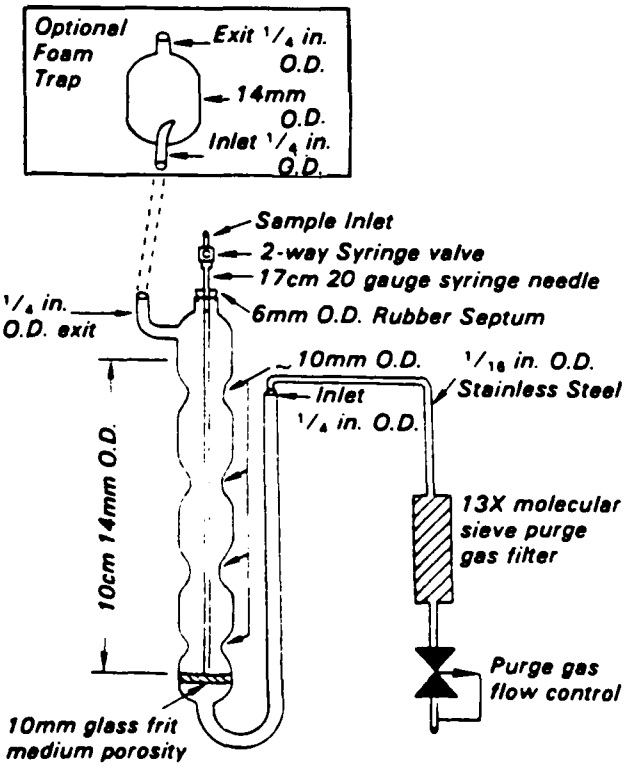


Figure 1. *Purging device*

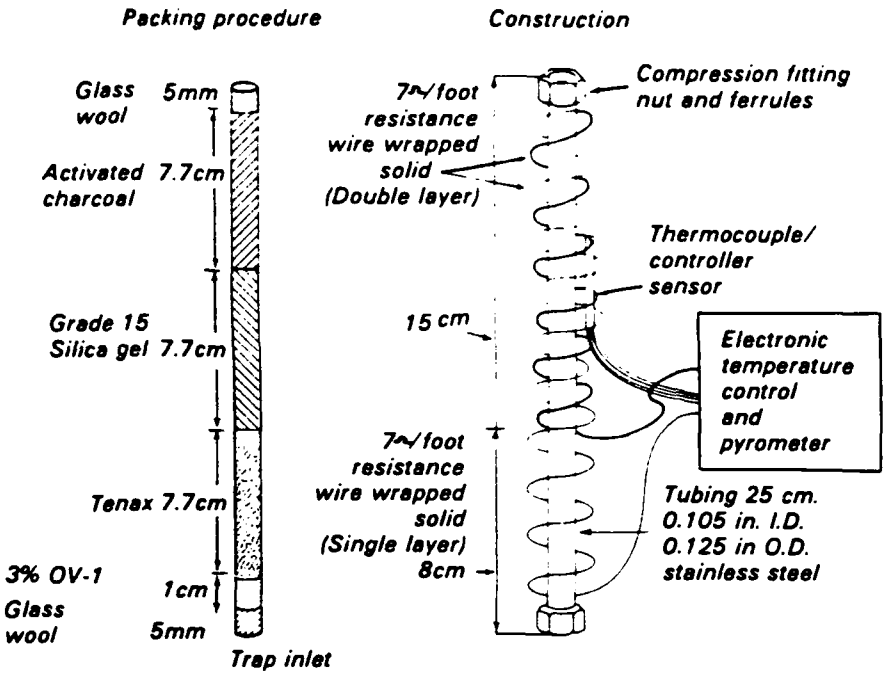


Figure 2. *Trap packings and construction to include desorb capability*

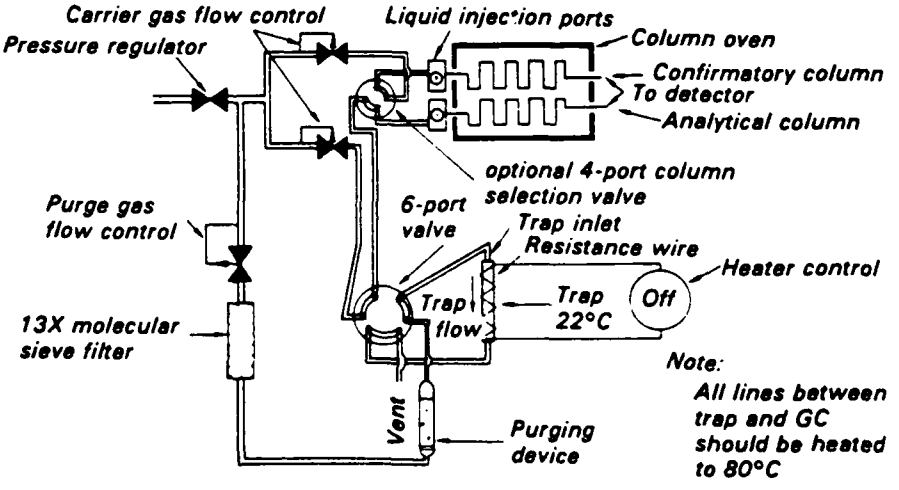


Figure 3. *Schematic of purge and trap device — purge mode*

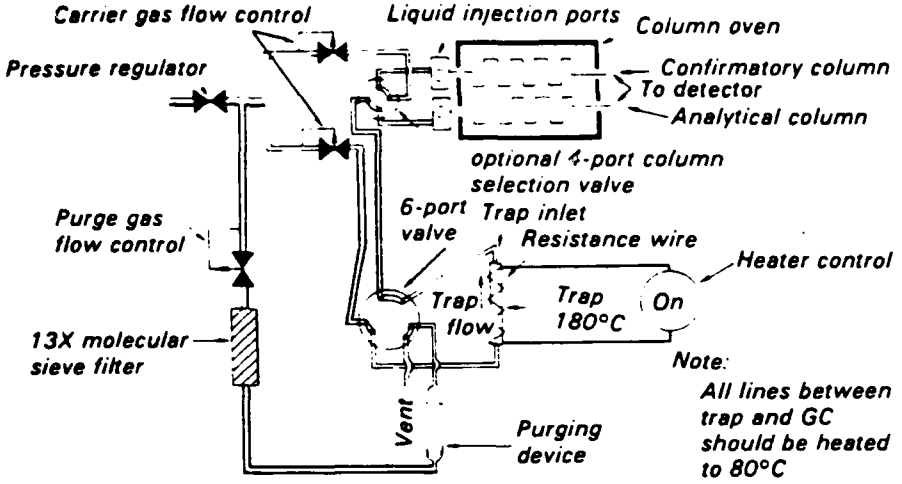


Figure 4. *Schematic of purge and trap device — desorb mode*

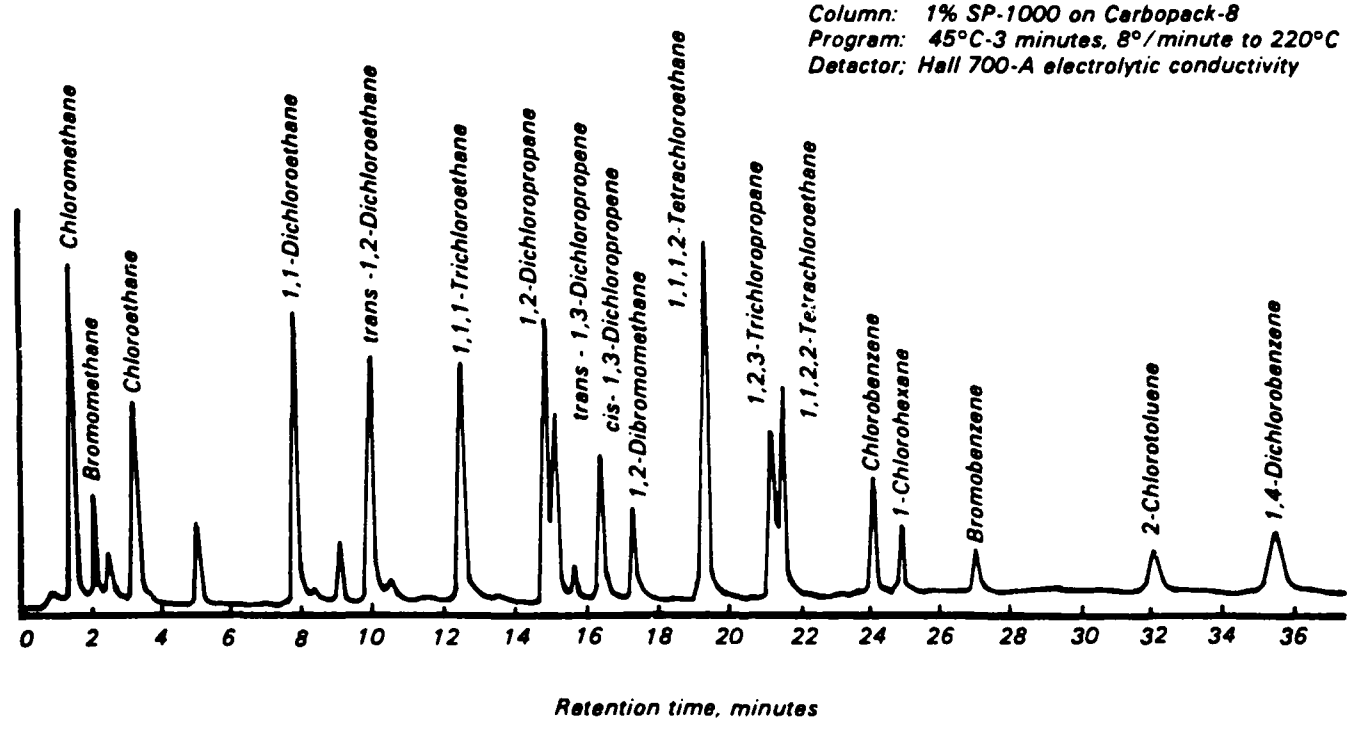


Figure 5. *Gas chromatogram of purgeable halocarbons*

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Purgeable Aromatics—Method 602

1. Scope and Application

1.1 This method covers the determination of various purgeable aromatics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Benzene	34030	71-43-2
Chlorobenzene	34301	108-90-7
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Ethylbenzene	34371	100-41-4
Toluene	34010	108-88-3

1.2 This is a purge and trap gas chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1[1]) for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from these listed depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval for alternate test procedures under 40 CFR 136.4 and 136.5

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The aromatics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After

purging is completed, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected with a photoionization detector[2,3].

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high aromatic levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene and 1,4-dichlorobenzene. Primary standards of these toxic compounds should be prepared in a hood. An NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch.

5.2.2.1 The trap is packed with 1 cm of methyl silicone and 23 cm 2,6-diphenylene oxide polymer as shown in Figure 2. This trap was used to develop the method performance statements in Section 12.

5.2.2.2 Alternatively, either of the two traps described in Method 601 may be used, although water vapor will preclude the measurement of low concentrations of benzene.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3, 4, and 5.

5.3 Gas chromatograph—Analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and stripchart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—6 ft long × 0.082 in ID stainless steel or glass, packed with 5% SP-1200 and 1.75% Bentone-34 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements and the MDLs listed in Tables 1 and 2. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—8 ft long × 0.1 in ID stainless steel or glass, packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on Chromosorb W-AW (60/80 mesh) or equivalent.

5.3.3 Detector—Photoionization detector (h-nu Systems, Inc. Model PI-51-02 or equivalent). The type of detector has been proven effective in the analysis of wastewaters for the parameters listed in the scope, and was used to develop the performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purge device.

5.5 Micro syringes—25 μL, 0.006 in ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Bottle—15-mL screw-cap with Teflon cap liner.

5.8 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon. (Filtrasorb-300 or equivalent (Calgon Corp.)).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Hydrochloric acid (1 + 1)—Add 50 mL of concentrated HCl to 50 mL of reagent water.

6.4 Trap Materials

6.4.1 2,6-Diphenylene oxide polymer-Tenax, (60/80 mesh) chromatographic grade or equivalent.

6.4.2 Methyl silicone—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.5 Methyl alcohol—Pesticide quality or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methyl alcohol using assayed liquids. Because benzene and 1,4-dichlorobenzene are suspected carcinogens, primary dilutions of these materials should be prepared in a hood.

6.6.1 Place about 9.8 mL of methyl alcohol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.6.2 Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter 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. Commercially prepared stock standards can be used, at any concentration, if they are certified by the manufacturer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 All standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methyl alcohol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary solution standards must be stored with zero headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, in Cincinnati, Ohio.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4.).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (see Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (≤10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that

is applicable to all samples. The compound, α,α,α-trifluorotoluene, recommended as a surrogate spiking compound in Section 8.7 has been used successfully as an internal standard.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard, according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe as indicated in Section 10.4. Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1} \quad RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard.
C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in methyl alcohol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 μL of the check sample concentrate to each of a minimum of four 5-mL aliquots of reagent water. A representative wastewater may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.3.1 Calculate upper and lower control limits for method performance:

$$\text{Upper Control Limit (UCL)} = R + 3s$$
$$\text{Lower Control Limit (LCL)} = R - 3s$$

where R and s are calculated as in Section 8.2.3

The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly[7].

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results

reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 11.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should maintain constant surveillance of both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and blank with surrogate compounds (e.g. α,α,α-trifluorotoluene). From stock standard solutions prepared as above, add a volume to give 7500 μg of each surrogate to 45 mL of organic-free water contained in a 50-mL volumetric flask, mix and dilute to volume (15 ng/μL). If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2). Dose 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis.

9. Sample Collection, Preservation, and Handling

9.1 The samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottles just prior to shipping to the sampling site. USEPA Methods 330.4 or 330.5 may be used to measure residual chlorine[8]. Field Test Kits are available for this purpose.

9.2 Collect about 500 mL sample in a clean container. Adjust the pH of the sample to about 2 by adding 1 + 1 HCl while stirring gently. Fill the sample bottle in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Sample Extraction and Gas Chromatography

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and method detection limits that can be achieved by this method. An example of the separations achieved by Column 1 is shown in Figure 6. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow sample to come to ambient temperature prior to introducing it into the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (Section 8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 12.0 ± 0.1 minutes at ambient temperature.

10.7 After the 12-minute purge time, disconnect the purge chamber from the trap. Dry the trap by maintaining a flow of 40 mL/min of dry purge gas through it for six minutes. See Figure 4. A dry purger should be inserted into the device to minimize moisture in the gas. Attach the trap to the chromatograph, adjust the device to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If rapid heating cannot be achieved, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry and random retention time problems persist) instead of the initial program temperature of 50 °C.

10.8 While the trap is being desorbed onto the GC column, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

10.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for the peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of material from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration $\mu g/L = (A_s C_{is})/(A_{is})(RF)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard.

11.2 Report results in micrograms per liter. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

11.3 For samples processed as part of a set where the spiked sample recovery falls outside of the control limits which were described in Section 8.3, data for the affected parameters must be labeled as suspect.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero[1]. The MDL concentrations listed in Table 1 were obtained using reagent water[9]. Similar results were achieved using representative wastewaters.

12.2 This method has been demonstrated to be applicable for the concentration range from the MDL up to 1000 × MDL[9]. Direct aqueous injection techniques should be used to measure concentration levels above 1000 × MDL.

12.3 In a single laboratory (Monsanto Research), using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 2 were obtained[9]. The standard deviation of the measurement in percent recovery is also included in Table 2.

12.4 The Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1. See Appendix A.
2. Bellar, T.A., and Lichtenberg, J.J. *Journal American Water Works Association, 66,* 739, (1974).
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5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised January 1976).
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8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.
9. "EPA Method Validation Study 24, Method 602 (Purgeable Aromatics)," Report for EPA Contract 68-03-2856 (In preparation).

Table 1. *Chromatographic Conditions and Method Detection Limits*

Parameter	Retention Time (min.) Column 1	Retention Time (min.) Column 2	Method Detection Limit µg/L
Benzene	3.33	2.75	0.2
Toluene	5.75	4.25	0.2
Ethylbenzene	8.25	6.25	0.2
Chlorobenzene	9.17	8.02	0.2
1,4-Dichlorobenzene	16.8	16.2	0.3
1,3-Dichlorobenzene	18.2	15.0	0.4
1,2-Dichlorobenzene	25.9	19.4	0.4

Column 1 conditions: Supelcoport 100/120 mesh coated with 5% SP-1200 and 1.75% Bentone-34 packed in a 6 ft. × 0.085 in ID stainless steel column with helium carrier gas at 36 cc/min flow rate. Column temperature held at 50°C for 2 min. then programmed at 6°C/min to 90°C for a final hold.

Column 2 conditions: Chromosorb W-AW 60/80 mesh coated with 5% 1,2,3-Tris(2-cyanoethyoxy)propane packed in a 6 ft. × 0.085 in ID stainless steel column with helium carrier gas at 30 cc/min flow rate. Column temperature held at 40°C for 2 min then programmed at 2°C/min to 100°C for a final hold.

Table 2. *Single Operator Accuracy and Precision*

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (µg/L)	Number of Analyses	Matrix Types
Benzene	91	10.0	0.5-9.7	21	3
Chlorobenzene	97	9.4	0.5-100	21	3
1,2-Dichlorobenzene	104	27.7	0.5-10.0	21	3
1,3-Dichlorobenzene	97	20.0	0.5-4.8	21	3
1,4-Dichlorobenzene	120	20.4	0.5-10.0	21	3
Ethylbenzene	98	12.4	0.5-9.9	21	3
Toluene	77	12.1	0.5-100	21	3

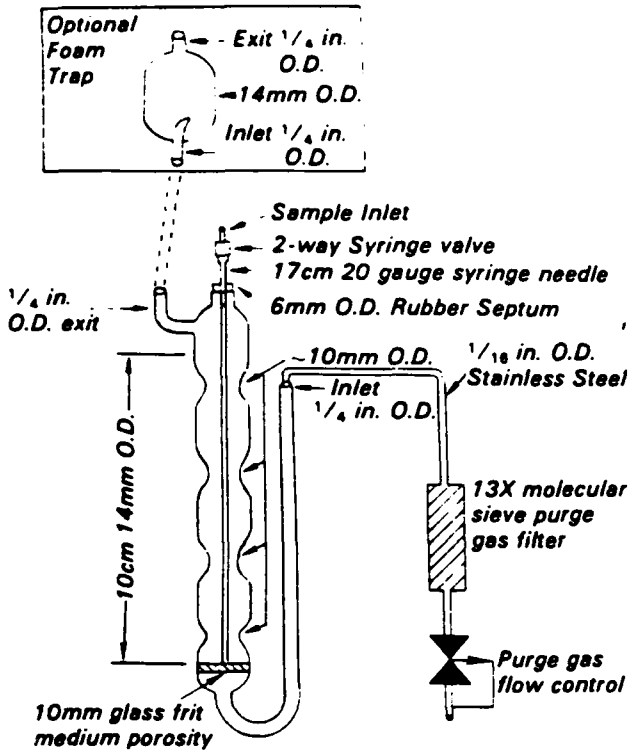


Figure 1. *Purging device*

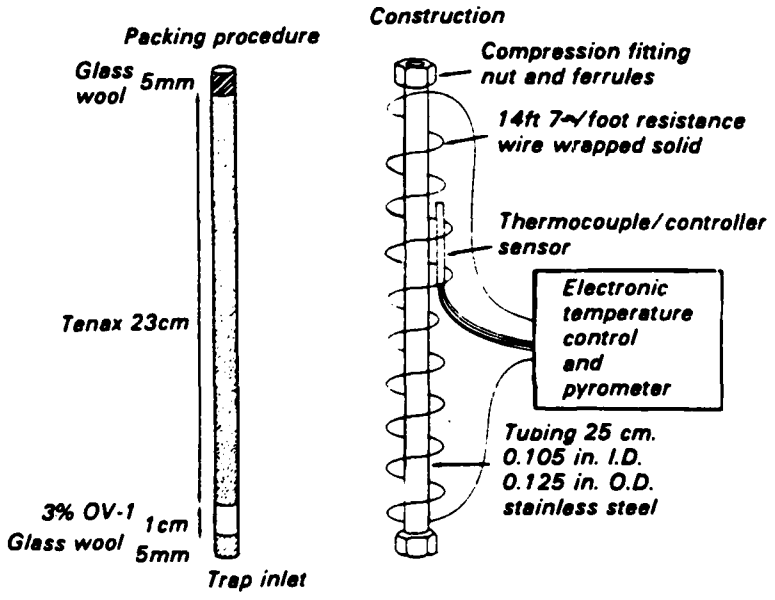


Figure 2. *Trap packings and construction to include desorb capability*

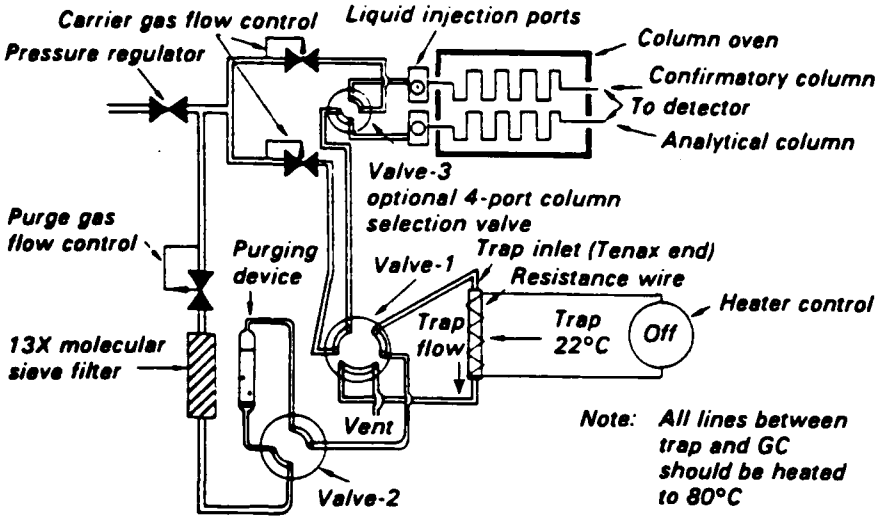


Figure 3. *Purge-trap system (Purge-sorb Mode)*

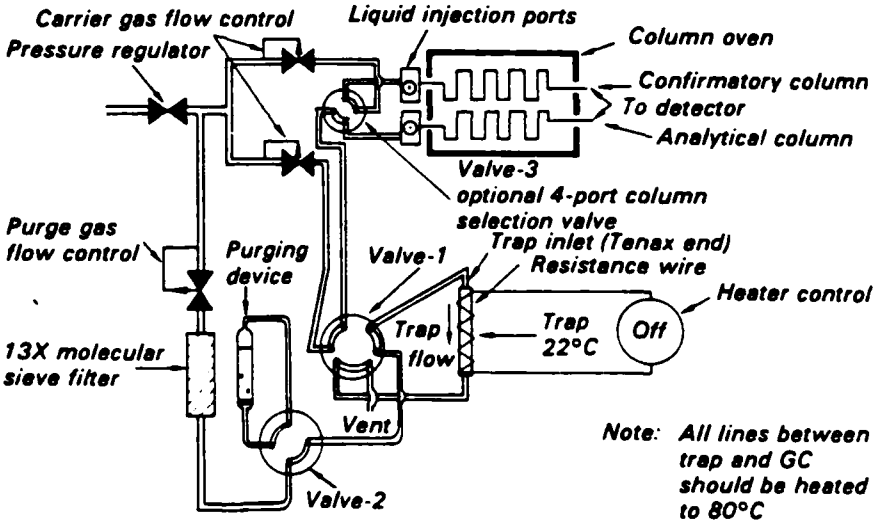


Figure 4. *Purge-trap system (Trap-dry Mode).*

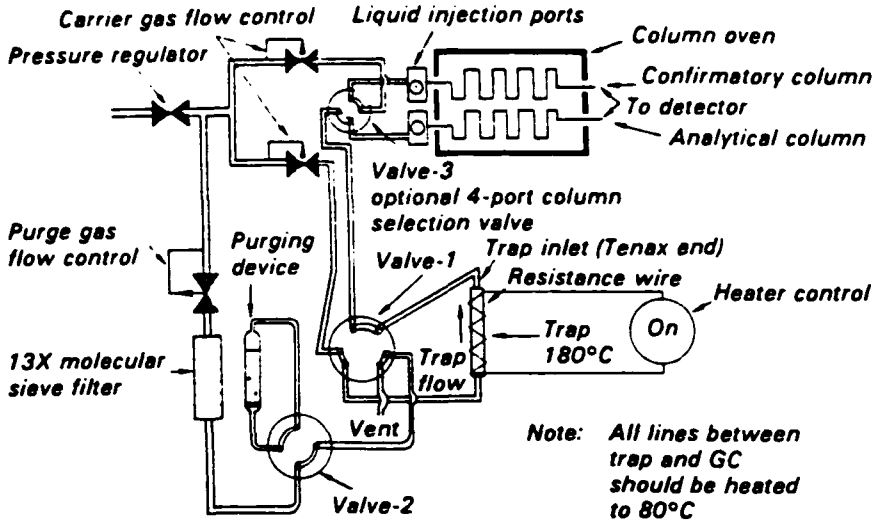


Figure 5. *Purge-trap system (Desorb Mode).*

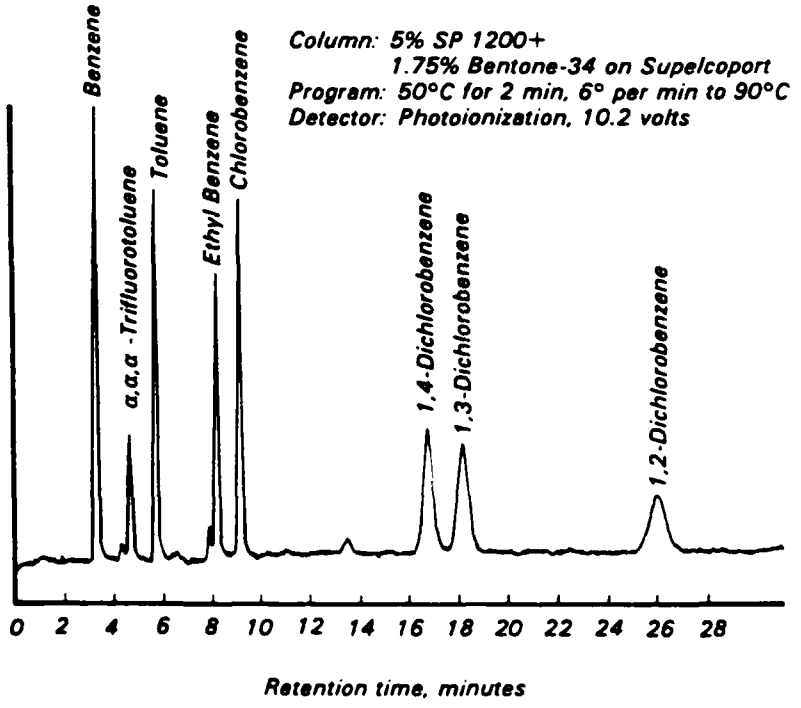


Figure 6. *Gas chromatogram of purgeable aromatics.*

METHOD 5020

HEADSPACE METHOD

1.0 Scope and Application

1.1 Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. It is a simple method that allows large numbers of samples to be analyzed in a relatively short period of time. Because of the large variability and complicated matrices of waste samples in the solid and paste forms, detection limits for this method may vary widely among samples. The method works best for compounds with boiling points of less than 125° C. The sensitivity of this method will depend on the equilibria of the various compounds between the vapor and dissolved phases.

1.2 This method is recommended for use by, or under the supervision of, analysts experienced in the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Summary of Method

The sample is collected in sealed glass containers and allowed to equilibrate at 90° C. A sample of the headspace gas is withdrawn with a gas-tight syringe for analysis by the appropriate gas chromatographic method (8010, 8015, 8020, or 8030).

3.0 Interferences

Refer to Methods 8010, 8015, 8020, or 8030.

4.0 Apparatus and Materials

4.1 Gas-tight syringe: 5-cc with chromatographic needles.

4.2 Headspace standard solutions: Prepare according to procedures in 8010, 8015, 8020, or 8030 at 50 ng/µl and 250 ng/µl concentrations.

4.3 Vials: 125-ml Hypo-Vials (Pierce Chemical Co., #12995, or equivalent).

4.4 Septa: Tuf-Bond (Pierce #12720, or equivalent).

4.5 Seals: Aluminum (Pierce #13214, or equivalent).

4.6 Crimper: Hand (Pierce #13212, or equivalent).

5.0 Reagents

5.1 Refer to Methods 8010, 8015, 8020, or 8030.

6.0 Sample Collection, Preservation, and Handling

6.1 Refer to Methods 8010, 8015, 8020, or 8030.

7.0 Procedure

7.1 Place 10.0 g each of the well-mixed waste sample into three separate 125-ml septum seal vials.

7.2 Dose one sample vial through the septum with 200 µl of a 50-ng/µl methanolic standard of the compounds of interest. Label this "1-ppm spike."

7.3 Dose a separate (empty) 125-ml septum seal vial with 200 µl of the 50 ng/µl standard methanol solution. Label this "1-ppm standard."

7.4 Place the sample, 1-ppm-spike, and 1-ppm-standard vials into a 90° C water bath for 1 hr. Store the remaining sample vial at 4.0° C for possible future analysis.

7.5 While maintaining the vials at 90° C, withdraw 2 ml of the headspace gas with a gas-tight syringe and analyze by injecting into a GC, operating under the appropriate conditions for the GC measurement method being used (8010, 8015, 8020, or 8030).

7.6 Analyze the 1-ppm standard and adjust instrument sensitivity to give a minimum response of at least 2x the background. Record retention times (RT) and peak areas of compounds of interest.

7.7 Analyze the 1-ppm spiked sample in the same manner. Record RT's and peak areas.

7.8 Analyze the undosed sample as in Section 7.7.

8.0 Quality Control

8.1 Before processing any samples, the analyst should demonstrate through the analysis of a distilled water method blank that all glassware and reagents are interference-free. Each time a set of samples is extracted or there is a change in reagents, a method blank should be processed as a

safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified samples do not indicate sufficient sensitivity to detect less than or equal to 1 µg/g of sample, then the sensitivity of the instrument should be increased. Where doubt exists over the identification of a peak on the chromatograph, confirmatory techniques such as mass spectroscopy should be used.

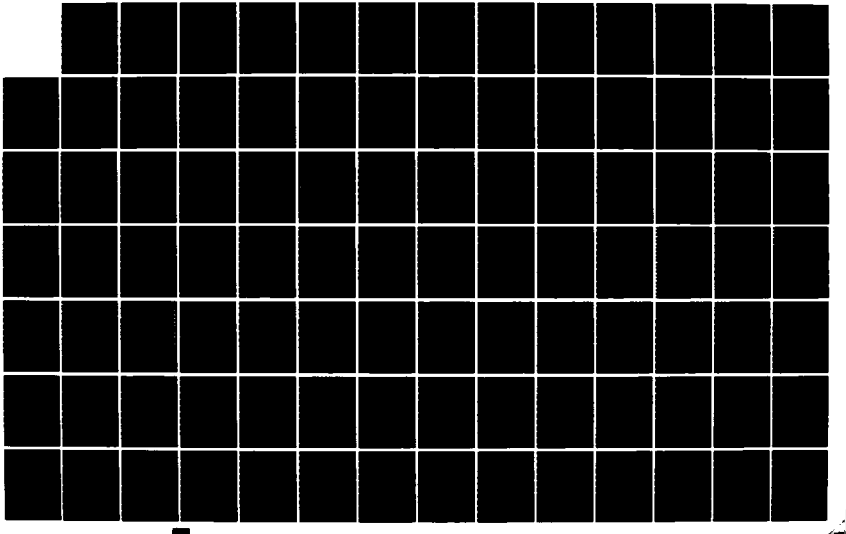
9.0 References

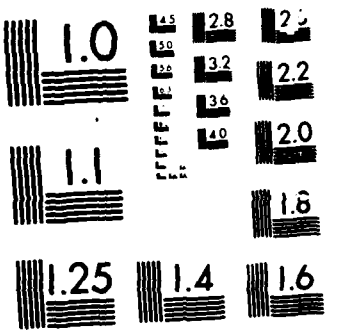
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METHOD 8240

GC/MS METHOD FOR VOLATILE ORGANICS

1.0 Scope and Application

1.1 Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 The detection limit of Method 8240 for an individual compound is approximately 1 µg/g (wet weight) in waste samples. For samples containing more than 1 mg/g of total volatile material, the detection limit is proportionately higher.

1.3 Method 8240 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by or under the supervision of analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 Summary of Method

2.1 The volatile compounds are introduced to the gas chromatograph by direct injection, the Headspace Method (Method 5020), or the Purge-and-Trap Method (Method 5030). Method 5030 should be used for groundwater analysis. The components are separated via the gas chromatograph and detected using a mass spectrometer which is used to provide both qualitative and quantitative information. The chromatographic conditions as well as typical mass spectrometer operating parameters are given.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample can be dispersed in methanol or polyethylene glycol (PEG) to dissolve the volatile organic constituents. A portion of the methanolic or PEG solution is combined with water in a specially designed purging chamber. An inert gas is then bubbled through the solution at ambient temperature and the volatile comnponents are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

2.3 An aliquot of each sample must be spiked with an appropriate standard to determine percent recovery and detection limits for that sample.

2.4 Table 1 lists detection limits that can be obtained in wastewaters in the absence of interferences. Detection limits for a typical waste sample would be significantly higher.

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min) Column 1[a]	Method detection limit (µg/l)
Chloromethane	2.3	ND
Bromomethane	3.1	ND
Vinyl chloride	3.8	ND
Chloroethane	4.6	ND
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	ND
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2-Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloropropane	15.7	6.0
trans-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
cis-1,3-Dichloropropene	17.2	ND
2-Chloroethylvinyl ether	18.6	ND
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1
Toluene	23.5	6.0
Chlorobenzene	24.6	6.0
Ethyl benzene	26.4	7.2
1,3-Dichlorobenzene	33.9	ND
1,2-Dichlorobenzene	35.0	ND
1,4-Dichlorobenzene	35.4	ND

ND = not determined.

[a]Column conditions: Carbopack B (60/80 mesh) coated with 1% SP-1000 packed in a 6-ft by 2-mm I.D. glass column with helium carrier gas at a flow rate of 30 ml/min. Column temperature is isothermal at 45° C for 3 min, then programmed at 8° C per minute to 220° and held for 15 min.

3.0 Interferences

3.1 Interferences coextracted from the samples will vary considerably from source to source, depending upon the particular waste or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences under the conditions of the analysis by running method blanks. Method blanks are run by analyzing organic-free water in the normal manner. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride) through the septum seal into the sample during shipment and storage. A field blank prepared from organic-free water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Cross contamination can occur whenever high-level and low-level samples are sequentially analyzed. To reduce cross contamination, the purging device and sample syringe should be rinsed out twice, between samples, with organic-free water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of organic-free water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high organohalide levels, it may be necessary to wash out the purging device with a soap solution, rinse with distilled water, and then dry in a 105° C oven between analyses.

3.4 Low molecular weight impurities in PEG can be volatilized during the purging procedure. Thus, the PEG employed in this method must be purified before use as described in Section 5.2.

4.0 Apparatus and Materials

4.1 Sampling equipment

4.1.1 Vial: 25-ml capacity or larger, equipped with a screw cap (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry for 1 hr at 105° C before use.

4.1.2 Septum: Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water and dry at 105° C for 1 hr before use.

4.2 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the purging chamber, trap, and the desorber. Several complete devices are now commercially available.

4.2.1 The purging chamber must be designed to accept 5-ml or 25-ml samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging chamber, illustrated in Figure 1, meets these design criteria.

4.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 2.5 mm. The trap must be packed to contain the following minimum lengths-of-adsorbents: 1.0 cm of methyl-silicone-coated packing (Section 5.3.2), 15 cm of 2,6-diphenylene oxide polymer (Section 5.3.1), and 8 cm of silica gel (Section 5.3.3). The minimum specifications for the trap are illustrated in Figure 2.

4.2.3 The desorber must be capable of rapidly heating the trap to 180° C within 30 sec. The polymer section of the trap should not be heated higher than 180° C and the remaining sections should not exceed 220° C. The desorber design, illustrated in Figure 2, meets these criteria.

4.2.4 The purge-and-trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

4.3 Gas chromatograph/mass spectrometer system

4.3.1 Gas chromatograph: An analytical system complete with a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

4.3.2 Column: 2-m x 2-mm I.D. stainless steel or glass, packed with 1% SP-1000 on 60/80 mesh Carbopack B or equivalent.

4.3.3 Mass spectrometer: Capable of scanning from 40 to 250 amu every 3 sec or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria in Table 1 when 50 ng of 4-bromofluoro-benzene (BFB) is injected through the GC inlet or introduced in the purge-and-trap mode.

4.3.4 GC/MS interface: Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria (see Section 9) may be used. GC-to-MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane. The interface must be capable of transporting at least 10 ng of the components of interest from the GC to the MS.

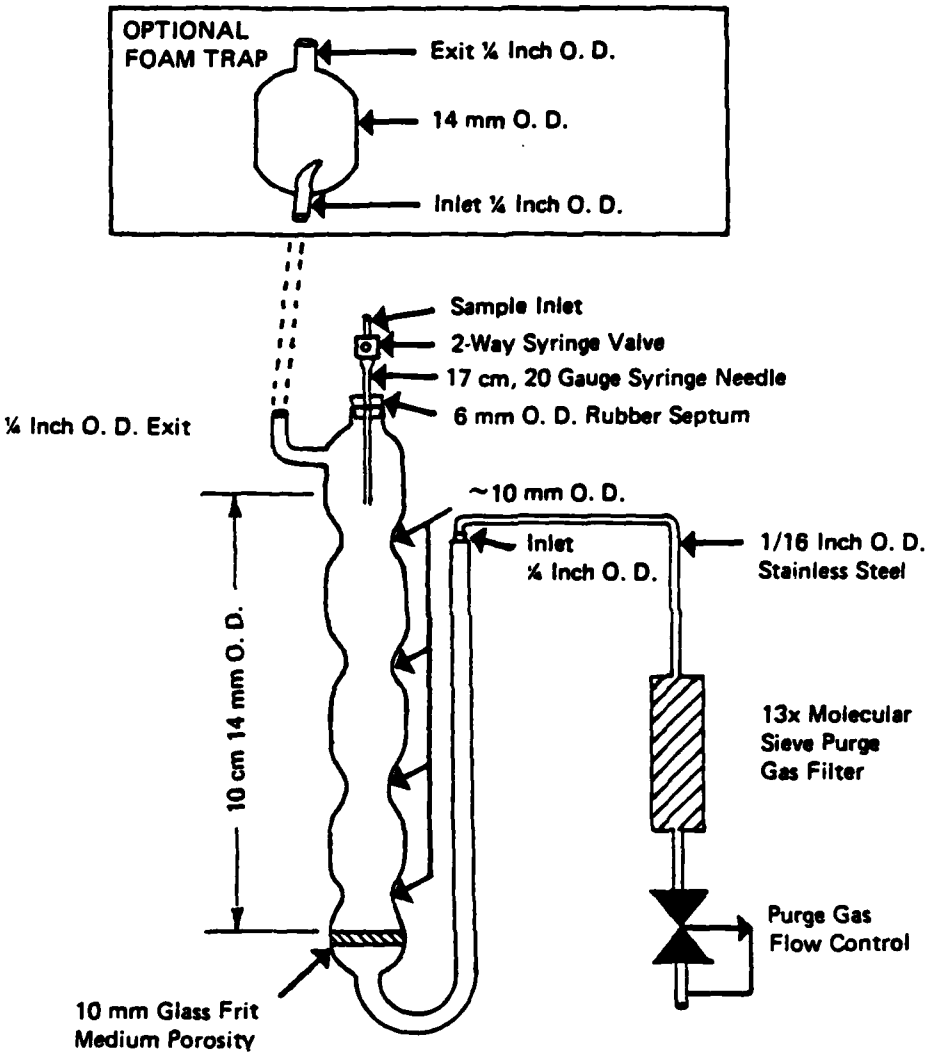


Figure 1. Purging chamber.

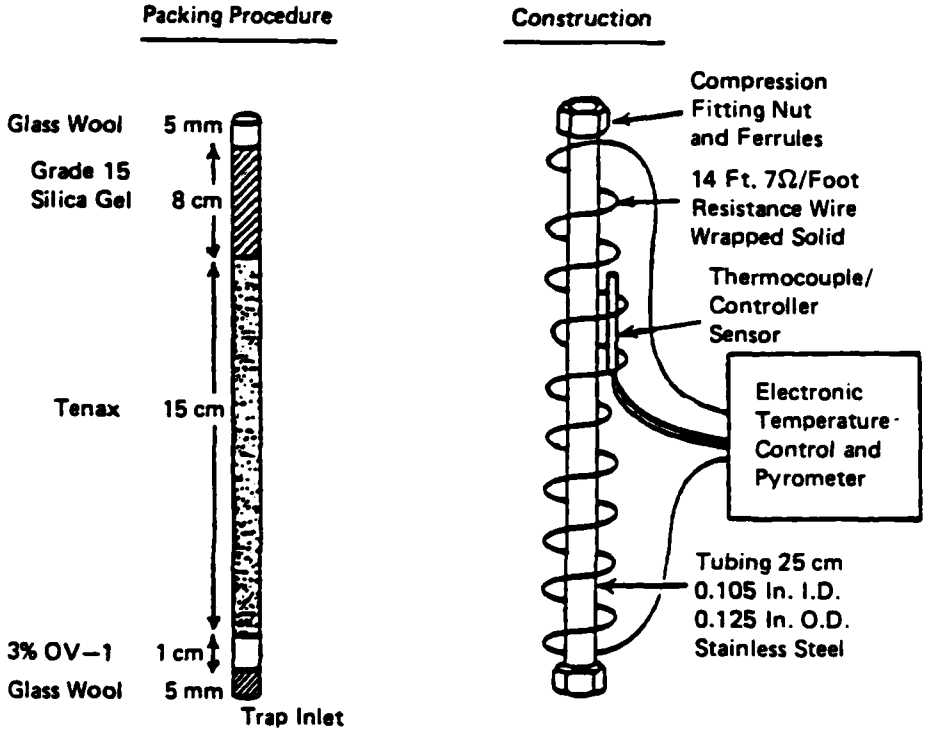


Figure 2. Trap packings and construction to include desorb capability.

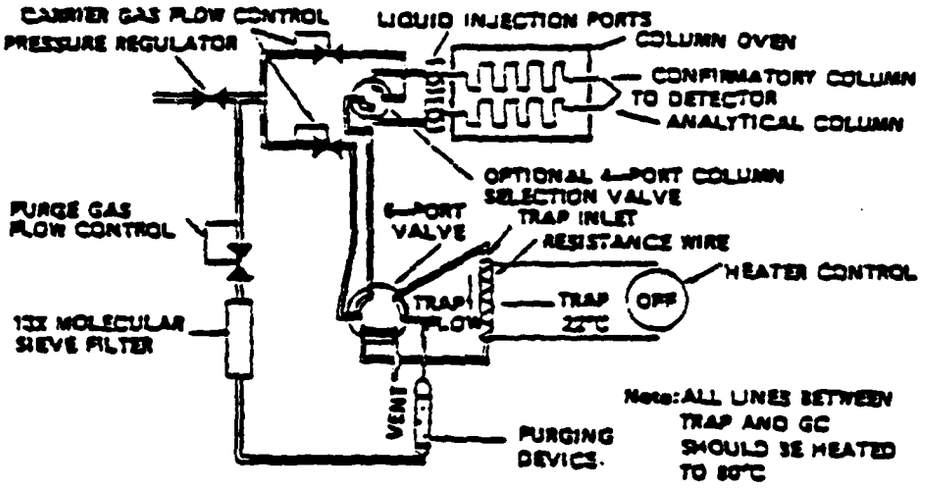


FIGURE 3. Schematic of purge and trap device – purge mode

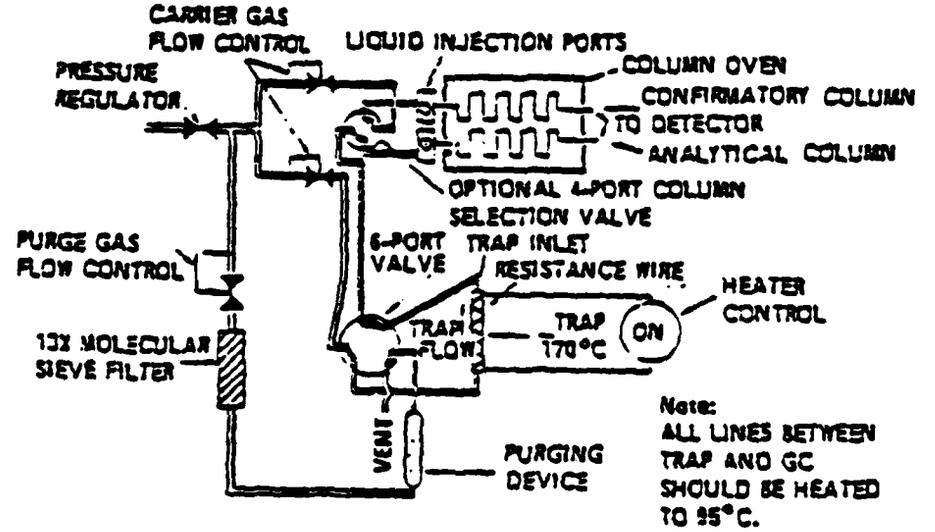


Figure 4. Schematic of purge and trap device - desorb mode

4.3.5 Data system: A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Hardware and software must be available to transform the data into a compatible format. These generally consist of a 9-inch, 800-bpi tape drive and the associated software.

4.4 Sample transfer implements: Implements are required to transfer portions of solid, semisolid, and liquid wastes from sample containers to laboratory glassware. The transfer must be accomplished rapidly to avoid loss of volatile components during the transfer step. Liquids may be transferred using a hypodermic syringe with a wide-bore needle or no needle attached. Samples should be introduced into the syringe by (1) removing the plunger from the syringe, (2) pouring the sample into the barrel, and (3) replacing the barrel and inverting the syringe to remove any air trapped in the syringe. Do not draw the sample up into the syringe. Solids may be transferred using a conventional laboratory spatula, spoon, or coring device. A coring device that is suitable for handling some samples can be made by using a glass tubing saw to cut away the closed end of the barrel of a glass hypodermic syringe.

TABLE 2. BFB KEY ION ABUNDANCE CRITERIA

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 100% of mass 174
177	5 to 9% of mass 176

4.5 Syringes: 5-ml and 25-ml glass hypodermic, equipped with 20-gauge needle, at least 15 cm in length.

4.6 Micro syringes: 10-µl, 25-µl, 100-µl, 250-µl, and 1000-µl. These syringes should be equipped with 20-gauge needles having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device (see Figure 1). The needle length required will depend upon the dimensions of the purging device employed.

4.7 Centrifuge tubes: 50-ml round-bottom glass centrifuge tubes with Teflon-lined screw caps. The tubes must be marked before use to show an approximate 20-ml graduation.

4.8 Centrifuge: Capable of accommodating 50-ml glass tubes.

4.9 Syringe valve: 2-way, with Luer ends (2 each) (Hamilton #86725 valve equipped with one Hamilton #35033 Luer fitting, or equivalent).

4.10 Syringe: 5-ml, gas-tight with shut-off valve.

4.11 Bottle: 15-ml, screw-cap, Teflon cap liner.

4.12 Balance: Analytical, capable of accurately weighing 0.0001 g.

4.13 Rotary evaporator: equipped with Teflon-coated seals (Buchi Rotavapor R-110, or equivalent).

4.14 Vacuum pump: mechanical, two-stage.

5.0 Reagents

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 500 g of activated carbon (Calgon Corp., Filtrasorb-300, or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90° C, bubble a contaminant-free inert gas through the water for 1 hr. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

5.1.4 Reagent water may also be purchased under the name "HPLC water" from several manufacturers (Burdick and Jackson, Baker and Waters, Inc.).

5.2 Reagent PEG: Reagent PEG is defined as PEG having a nominal average molecular weight of 400, and in which interferents are not observed at the method detection limit for compounds of interest.

5.2.1 Reagent PEG is prepared by purification of commercial PEG having a nominal average molecular weight of 400. The PEG is placed in a round-bottom flask equipped with a standard taper joint, and the flask is affixed to a rotary evaporator. The flask is immersed in a water bath at 90-100° C and vacuum is maintained at less than 10 mm Hg for at least 1 hr using a two-stage mechanical pump. The vacuum system is equipped with an all-glass trap, which is maintained in a dry ice/methanol bath.

5.2.2 In order to demonstrate that all interfering volatiles have been removed from the PEG, a reagent water/PEG blank must be analyzed.

5.3 Trap materials

5.3.1 2,6-Diphenylene oxide polymer: 60/80-mesh Tenax, chromatographic grade or equivalent.

5.3.2 Methyl silicone packing: 3 percent OV-1 on 60/80 mesh Chromosorb-W or equivalent.

5.3.3 Silica gel, Davison Chemical (35/60 mesh), grade-15 or equivalent.

5.3.4 Prepared trapping columns may be purchased from several chromatography suppliers.

5.4 Methanol: Distilled-in-glass quality or equivalent.

5.5 Calibration standards; stock solutions (2 mg/ml): Stock solutions of calibration standards may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions of individual compounds in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA-approved toxic gas respirator should be worn by analysts when handling high concentrations of these materials.

5.5.1 Place about 9.8 ml of methanol in 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. Weigh the flask to the nearest 0.1 mg.

5.5.2 Add the assayed reference material as described below.

5.5.2.1 Liquids: Using a 100-µl syringe, immediately add 2 drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.5.2.2 Gases: To prepare standards for any compounds that boil below 30° C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-ml valved gas-tight syringe with a reference standard to the 5.0-ml mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol.

5.5.3 Reweigh, dilute to volume, stopper, then mix by gently inverting the flask several times. Calculate the concentration in µg/µl per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10 to -20° C and protect from light.

5.5.5 Prepare fresh standards weekly for gases or for reactive compounds such as 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

5.6 Calibration standards; secondary dilution solutions: Using stock solutions described in Section 5.5, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the methanol or aqueous PEG calibration solutions prepared as described in Section 6.3.2 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards from them.

5.7 Surrogate standards: Surrogate standards may be added to samples and calibration solutions to assess the effect of the sample matrix on recovery efficiency. The compounds employed for this purpose are 1,2-dibromotetrafluoroethane, bis(perfluoroisopropyl) ketone, fluorobenzene, and m-bromobenzotrifluoride. Prepare methanolic solutions of the surrogate standards using the procedures described in Sections 5.5 and 5.6. The

concentrations prepared and the amount of solution added to each sample should be those required to give an amount of each surrogate in the purging device that is equal to the amount of each internal standard added, assuming a 100% recovery of the surrogate standards.

5.8 Internal standards: In this method, internal standards are employed during analysis of all samples and during all calibration procedures. The analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. However, for general use, D_4-1,2-dichloroethane, D_6-benzene, and D_5-ethylbenzene are recommended as internal standards covering a wide boiling point range.

5.9 4-Bromofluorobenzene (BFB): BFB is added to the internal standard solution or analyzed alone to permit the mass spectrometer tuning for each GC/MS run to be checked.

5.10 Internal standard solution: Using the procedures described in Sections 5.5 and 5.6, prepare a methanolic solution containing each internal standard at a concentration of 12.5 µg/ml.

5.11 Sodium monohydrogen phosphate: 2.0 µ in distilled water.

5.12 n-Nonane and n-dodecane, 98+% purity.

5.13 N-Hexadecane, distilled-in-glass (Burdick and Jackson, or equivalent).

6.0 Sample Collection, Handling, and Preservation

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All samples must be stored in Teflon-lined screw cap vials. Sample containers should be filled as completely as possible so as to minimize headspace or void space. Vials containing liquid sample should be stored in an inverted position.

6.3 All samples must be iced or refrigerated from the time of collection to the time of analysis, and should be protected from light.

7.0 Procedure

7.1 Calibration

7.1.1 Assemble a purge-and-trap device that meets the specifications in Section 4.2 and connect the device to a GC/MS system. Condition the trap overnight at 180° C by backflushing with an inert gas flow of at least 20 ml/min. Prior to use, condition the trap daily for 10 min while backflushing at 180° C.

7.1.2 Operate the gas chromatograph using the conditions described in Section 7.3.5 and operate the mass spectrometer using the conditions described in Section 7.3.2.

7.1.3 Calibration procedure

7.1.3.1 Conduct calibration procedures using a minimum of three concentration levels for each calibration standard. One of the concentration levels should be at a concentration near but above the method detection limit. The remaining two concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

7.1.3.2 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. To the purging device, add 5.0 ml of reagent water or reagent water/PEG solution. This solution is prepared by taking 4.0 ml of reagent water or reagent PEG and diluting to 100 ml with reagent water. The reagent water/PEG solution is added to the purging device using a 5-ml glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of a 20-gauge needle. Next, using a 10-µl or 25-µl micro-syringe equipped with a long needle (see Section 4.6), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards (see Section 5.6). Add the aliquot of calibration solution directly to the reagent water or reagent water/PEG solution in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe be sure that the end of the syringe needle is well beneath the surface of the reagent water or water/PEG solution. Similarly, add 20 µl of the internal standard solution (see Section 5.10). Close the 2-way syringe valve at the sample inlet.

7.1.3.3 Carry out the purge and analysis procedure as described in Section 7.3.4. Tabulate the area response of the primary characteristic ion against concentration for each compound

including the internal standards. Calculate response factors (RF) for each compound as follows:

$$RF = (A_s C_{is})/A_{is} C_s)$$

where:

A_s = Area of the primary characteristic ion for the compound to be measured

A_{is} = Area of the primary characteristic ion of the internal standard

C_{is} = Concentration of the internal standard

C_s = Concentration of the compound to be measured.

The internal standard selected for the calculation of the RF of a compound and subsequent quantification of the compound is generally the internal standard that has a retention time closest to that of the compound. It is assumed that a linear calibration plot will be obtained over the range of concentrations used. If the RF value over the working range is a constant (less than 10% relative standard deviation), the RF can be assumed to be invariant, and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, versus RF.

7.1.3.4 The RF must be verified on each working day. The concentrations selected should be near the midpoint of the working range. The response factors obtained for the calibration standards analyzed immediately before and after a set of samples must be within $\pm$20% of the response factor used for quantification of the sample concentrations.

7.2 Daily GC/MS performance tests

7.2.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for BFB (see Table 2).

7.2.2 The BFB performance test requires the following instrumental parameters:

Electron Energy:	70 volts (nominal)
Mass Range:	40 to 250 amu
Scan Time:	to give approximately 6 scans per peak but not to exceed 3 sec per scan.

7.2.3 Bleed BFB vapor into the mass spectrometer and tune the instrument to achieve all the key ion criteria for the mass spectrum of BFB given in Table 1. A solution containing 20 ng of BFB may be injected onto the gas chromatographic column in order to check the key ion criteria.

7.2.4 The peak intensity of D_6-benzene is used to monitor the mass spectrometer sensitivity. The peak intensity for D_6-benzene observed during each sample analysis must be between 0.7 and 1.4 times the D_6-benzene peak intensity observed during the applicable calibration runs. For example, if the peak intensity of D_6-benzene observed during calibration was 355,000 area counts, then each subsequent sample or blank must give a D_6-benzene peak intensity of between 250,000 and 500,000 area counts. If the D_6-benzene peak intensity is outside the specified range, the sample must be reanalyzed. If the peak intensity is again outside the specified range, the analyst must investigate the cause of the variability in sensitivity and correct the problem.

7.3 Sample extraction and analysis

7.3.1 The analytical procedure involves extracting the non-aqueous sample with methanol or polyethylene glycol (PEG) and analyzing a portion of the extract by a purge-and-trap GC/MS procedure. The amount of the extract to be taken for the GC/MS analysis is based on the estimated total volatile content (TVC) of the sample. The TVC is estimated by extracting the sample with n-hexadecane and analyzing the n-hexadecane extract by gas chromatography.

7.3.2 The estimated TVC is based on the total area response relative to that of n-nonane for all components eluting prior to the retention time of n-dodecane. The response factor for n-nonane and the retention time of n-dodecane are determined by analyzing a 2-µl aliquot of an n-hexadecane solution containing 0.20 mg/ml of n-nonane and n-dodecane.

7.3.2.1 The GC analyses are conducted using a flame ionization detector and a 3-m x 2-mm I.D. glass column packed with 10% OV-101 on 100-200 mesh Chromosorb W-HP. The column temperature is programmed from 80° C to 280° C at 8°/min and held at 280° for 10 min.

7.3.2.2 Determine the area response for n-nonane and divide by 0.2 to obtain the area response factor. Record the retention time of n-dodecane.

7.3.2.3 Add 1.0 g of sample to 20 ml of n-hexadecane and 2 ml of 2.0 M Na_2HPO_4 contained in a 50-ml glass centrifuge tube and cap securely with a Teflon-lined screw cap. Shake the mixture vigorously for one minute. If the sample does not disperse

during the shaking process, sonify the mixture in an ultrasonic bath for 30 min. Allow the mixture to stand until a clear supernatant is obtained. Centrifuge if necessary to facilitate phase separation.

7.3.2.4 Analyze a 2-µl aliquot of the n-hexadecane supernatant using the conditions described in Section 7.3.2.1. Determine the total area response of all components eluting prior to the retention time of n-dodecane and subtract the corresponding area of an n-hexadecane blank. Using the area response factor determined for n-nonane in Section 7.3.2.2, calculate the TVC as follows:

$$TVC = \frac{TAR_{sample} - TAR_{blank}}{\text{n-Nonane Area Response Factor}} \times 20$$

where:

TVC = total volatile content of the sample in mg/g

TAR_{sample} = total area response obtained for the sample

TAR_{blank} = total area response obtained for a blank.

7.3.3 The transfer of an aliquot of the sample for extraction with methanol or PEG should be made as quickly as possible to minimize loss of volatiles from the sample.

7.3.3.1 To a 50-ml glass centrifuge tube with Teflon-lined cap, add 40 ml of reagent methanol or PEG. Weigh the capped centrifuge tube and methanol or PEG on an analytical balance.

7.3.3.2 Using an appropriate implement (see Section 4.4), transfer approximately 2 g of sample to the methanol or PEG in the centrifuge tube in such a fashion that the sample is dissolved in or submerged in the methanol or PEG as quickly as possible. Take care not to touch the sample-transfer implement to the methanol or PEG. Recap the centrifuge tube immediately and weigh on an analytical balance to determine an accurate sample weight.

7.3.3.3 Disperse the sample by vigorous agitation for 1 min. The mixture may be agitated manually or with the aid of a vortex-mixer. If the sample does not disperse during this process, sonify the mixture in an ultrasonic bath for 30 min. Allow the mixture to stand until a clear supernatant is obtained as the sample extract. Centrifuge if necessary to facilitate phase separation.

7.3.3.4 The sample extract may be stored for future analytical needs. If this is desired, transfer the solution to a 10-ml screw cap vial with Teflon cap liner. Store at -10 to -20° C, and protect from light.

7.3.4 Reagent water, internal standard solution, and the sample extract are added to a purging chamber that is connected to the purge-and-trap device and that has been flushed with helium during a 7-min trap reconditioning step (see Section 7.3.4.4). The additions are made using an appropriately sized syringe equipped with a 15-cm 20-gauge needle. Open the syringe valve of the sample inlet (shown in Figure 1) and insert the needle through the valve.

7.3.4.1 Add 5.0 ml of reagent water or aqueous sample to which 20.0 µl of the internal standard solution has been added (see Section 5.10) to the purging chamber. Insert the needle of the syringe well below the surface of the water for the addition of the internal standard solution. If the sample is aqueous go to Section 7.3.5.

7.3.4.2 Add an aliquot of the sample extract from Section 7.3.3.4. The total quantity of volatile components injected should not exceed approximately 10 µg. If the total volatile content (TVC) of the sample as determined in Section 7.3.1.4 is 1.0 mg/g or less, use a 200-µl aliquot of the sample extract. If the TVC is greater than 1.0 mg/g, use an aliquot of the sample extract that contains approximately 10 µg of total volatile components; the volume (in µl) of the aliquot to be taken can be calculated by dividing 200 by the TVC. If the TVC is greater than 20 mg/g, take a 500-µl aliquot of the sample extract and dilute to 10 ml with PEG. In this case calculate the aliquot volume (in µl) of the undiluted extract to be taken by dividing 4,000 by the TVC. If the TVC is less than 1.0 mg/g and greater sensitivity is desired, use a large purging chamber containing 25 ml of reagent water and use a 1.0-ml aliquot of the sample extract.

7.3.4.3 Close the 2-way syringe valve at the sample inlet.

7.3.5 The sample in the purging chamber is purged with helium to transfer the volatile components to the trap. The trap is then heated to desorb the volatile components which are swept by the helium carrier gas onto the GC column for analysis.

7.3.5.1 Adjust the gas (helium) flow rate to 40 $\pm$ 3 ml/min. Set the purging device to purge, and purge the sample for 11.0 $\pm$ 0.1 min at ambient temperature.

7.3.5.2 At the conclusion of the purge time, adjust the device to the desorb mode, and begin the GC/MS analysis and data acquisition using the following GC operating conditions:

Column: 6-ft x 2-mm I.D. glass column of 1% SP-1000 on Carbo-pack B (60-80 mesh).

Temperature: Isothermal at 45° C for 3 min, then increased at 8° C/min to 220° C, and maintained at 220° C for 15 min.

Concurrently, introduce the trapped materials to the GC column by rapidly heating the trap to 180° C while backflushing the trap with helium at a flow rate of 30 ml/min for 4 min. If this rapid heating requirement cannot be met, the GC column must be used as a secondary trap by cooling it to 30° C or lower during the 4-min desorb step and starting the GC program after the desorb step.

7.3.5.3 Return the purge-and-trap device to the purge mode and continue acquiring GC/MS data.

7.3.5.4 Allow the trap to cool for 8 min. Replace the purging chamber with a clean purging chamber. The purging chamber is cleaned after each use by sequential washing with acetone, methanol, detergent solution and distilled water, and then dried at 105° C.

7.3.5.5 Close the syringe valve on the purging chamber after 15 sec to begin gas flow through the trap. Purge the trap at ambient temperature for 4 min. Recondition the trap by heating it to 180° C. Do not allow the trap temperature to exceed 180° C, since the sorption/desorption is adversely affected when the trap is heated to higher temperatures. After heating the trap for approximately 7 min, turn off the trap heater. When cool, the trap is ready for the next sample.

7.3.6 If the response for any ion exceeds the working range of the system, repeat the analysis using a correspondingly smaller aliquot of the sample extract described in Section 7.3.2.3.

7.4 Qualitative identification

7.4.1 Obtain an EICP for the primary characteristic ion and at least two other characteristic ions for each compound when practical. The following criteria must be met to make a qualitative identification.

7.4.1.1 The characteristic ions of each compound of interest must maximize in the same or within one scan of each other.

7.4.1.2 The retention time must fall within ±30 sec of the retention time of the authentic compound.

7.4.1.3 The relative peak heights of the characteristic ions in the EICP's must fall within ±20% of the relative intensities of these ions in a reference mass spectrum. Reference spectra may be generated from the standards analyzed by the analyst or from a reference library. All reference spectra generated from standards must be obtained from an appropriately tuned mass spectrometer.

7.5 Quantitative determination

7.5.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. In general, the primary characteristic ion selected should be a relatively intense ion, as interference-free as possible, and as close as possible in mass to the characteristic ion of the internal standard used. Generally, the base peak of the mass spectrum is used.

8.0 Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of the data that are generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within the accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The laboratory must spike all samples including check samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.1.3 Before processing any samples, the analyst should daily demonstrate, through the analysis of an organic-free water method blank, that the entire analytical system is interference-free. The blank samples should be carried through all stages of the sample preparation and measurement steps.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations using a representative sample as a check sample.

8.2.1 Analyze four aliquots of the unspiked check sample according to the method in Section 7.3.

8.2.2 For each compound to be measured, select a spike concentration representative of twice the level found in the unspiked check sample or a level equal to 10 times the expected detection limit, whichever is greater. Prepare a spiking solution by dissolving the compounds in methanol at the appropriate levels.

8.2.3 Spike a minimum of four aliquots of the check sample with the spiking solution to achieve the selected spike concentrations. Spike the samples by adding the spiking solution to the PEG used for the extraction. Analyze the spiked aliquots according to the method in Section 7.3.

8.2.4 Calculate the average percent recovery, R, and the standard deviation of the percent recovery, s, for all compounds and surrogate standards. Background corrections must be made before R and s calculations are performed. The average percent recovery must be greater than 20 for all compounds to be measured and greater than 60 for all surrogate compounds. The percent relative standard deviation of the percent recovery, s/R x 100, must be less than 20 for all compounds to be measured and all surrogate compounds.

8.3 The analyst must calculate method performance criteria for each of the surrogate standards.

8.3.1 Calculate upper and lower control limits for method performance for each surrogate standard, using the values for R and s calculated in Section 8.2.4:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R - 3s

The UCL and LCL can be used to construct control charts that are useful in observing trends in performance.

8.3.2 For each surrogate standard, the laboratory must maintain a record of the R and s values obtained for each surrogate standard in each waste sample analyzed. An accuracy statement should be prepared from these data and updated regularly.

8.4 The laboratory is required to spike all samples with the surrogate standards to monitor spike recoveries. The spiking level used should be that which will give an amount in the purge apparatus that is equal to the amount of the internal standard assuming a 100% recovery of the surrogate standards. If the recovery for any surrogate standard does not fall within the control limits for method performance, the results reported for that sample must be

qualified as being outside of control limits. The laboratory must monitor the frequency of data so qualified to ensure that it remains at or below 5%. Four surrogate standards, namely 1,2-dibromodifluoroethane, bis(perfluoro-isopropyl) ether, fluorobenzene, and m-bromobenzotrifluoride, are recommended for general use to monitor recovery of volatile compounds varying in volatility and polarity.

8.5 Each day, the analyst must demonstrate through the analysis of a process blank that all glassware and reagent interferences are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field replicates may be analyzed to monitor the precision of the sampling technique. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluatior studies.

8.7 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified waste samples do not indicate sufficient sensitivity to detect less than or equal to 1 µg/g of sample, then the sensitivity of the instrument should be increased or the extract subjected to additional cleanup. Detection limits to be used for groundwater samples are indicated in Table 1. Where doubt exists over the identification of a peak on the chromatograph, confirmatory techniques such as mass spectroscopy should be used.

8.8 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1 were obtained using reagent water. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

8.9 In a single laboratory, using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 3 were obtained. The standard deviation of the measurement in percent recovery is also included in Table 3.

TABLE 3. ACCURACY AND PRECISION FOR PURGEABLE ORGANICS

Parameter	Reagent Water		Wastewater	
	Average percent recovery	Standard deviation (%)	Average percent recovery	Standard deviation (%)
Benzene	99	9	98	10
Bromodichloromethane	102	12	103	10
Bromoform	104	14	105	16
Bromomethane	100	20	88	23
Carbon tetrachloride	102	16	104	15
Chlorobenzene	100	7	102	9
Chloroethane	97	22	103	31
2-Chloroethyl vinyl ether	101	13	95	17
Chloroform	101	10	101	12
Chloromethane	99	19	99	24
Dibromochloromethane	103	11	104	14
1,1-Dichloroethane	101	10	104	15
1,2-Dichloroethane	100	8	102	10
1,1-Dichloroethene	102	17	99	15
trans-1,2-Dichloroethene	99	12	101	10
1,2-Dichloropropane	102	8	103	12
cis-1,3-Dichloropropene	105	15	102	19
trans-1,3-Dichloropropene	104	11	100	18
Ethyl benzene	100	8	103	10
Methylene chloride	96	16	89	28
1,1,2,2-Tetrachloroethane	102	9	104	14
Tetrachloroethene	101	9	100	11
Toluene	101	9	98	14
1,1,1-Trichloroethane	101	11	102	16
1,1,2-Trichloroethane	101	10	104	15
Trichloroethene	101	9	100	12
Trichlorofluoromethane	103	11	107	19
Vinyl chloride	100	13	98	25

Samples were spiked between 10 and 1000 µg/l.

Volatile Organics in Soil by Headspace:
Sampling Protocol

1. Sample containers: 30-mL serum vials with teflon^{R}-lined crimp-top caps.

   Prepared containers are labeled & weighed with caps in place. Weight is recorded directly on the label, caps are taped in place to keep caps with the respective vial and keep the vial clean inside.

2. Approximately 5-10 grams of the soil are placed into the prepared and preweighed 30-mL serum vial. Care is to be taken during sampling not to place pebbles into the vials. A minimum amount of handling of the soil insures a more accurate analysis.

   Note: To obtain approximately 5-10 grams of soil, the vial should only be filled 1/3 to 1/2 full. (NO MORE THAN 1/2 FULL!)

3. After placing the soil into the vial, clean any particles off the lip of the vial with a chem-wipe and crimp the teflon^{R}-lined septum cap firmly in place, with the teflon^{R} liner of the septum towards the soil.

4. Obtain a total of three separate samples from each location to allow for dilutions, spikes, duplicates, or confirmations when needed.

ORGANIC CARBON, TOTAL

Method 415.1 (Combustion or Oxidation)

STORET NO. Total 00680
Dissolved 00681

1. Scope and Application
   1.1 This method includes the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes. Exclusions are noted under Definitions and Interferences.
   1.2 The method is most applicable to measurement of organic carbon above 1 mg/1.
2. Summary of Method
   2.1 Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation. The CO_2 formed can be measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 is directly proportional to the concentration of carbonaceous material in the sample.
3. Definitions
   3.1 The carbonaceous analyzer measures all of the carbon in a sample. Because of various properties of carbon-containing compounds in liquid samples, preliminary treatment of the sample prior to analysis dictates the definition of the carbon as it is measured. Forms of carbon that are measured by the method are:
      A) soluble, nonvolatile organic carbon; for instance, natural sugars.
      B) soluble, volatile organic carbon; for instance, mercaptans.
      C) insoluble, partially volatile carbon; for instance, oils.
      D) insoluble, particulate carbonaceous materials, for instance; cellulose fibers.
      E) soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter; for instance, oily matter adsorbed on silt particles.
   3.2 The final usefulness of the carbon measurement is in assessing the potential oxygen-demanding load of organic material on a receiving stream. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, or on water taken directly from the stream. In this light, carbonate and bicarbonate carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon which are measured. Instrument manufacturer's instructions should be followed.

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4. Sample Handling and Preservation

4.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.
NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

4.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

4.3 In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, the sample is acidified ($pH \leq 2$) with HCl or H_2SO_4.

5. Interferences

5.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

5.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particles which may be included in the sample.

6. Apparatus

6.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

6.2 Apparatus for total and dissolved organic carbon:

6.2.1 A number of companies manufacture systems for measuring carbonaceous material in liquid samples. Considerations should be made as to the types of samples to be analyzed, the expected concentration range, and forms of carbon to be measured.

6.2.2 No specific analyzer is recommended as superior.

7. Reagents

7.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank. Carbon dioxide-free, double distilled water is recommended. Ion exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.

7.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.
NOTE 2: Sodium oxalate and acetic acid are not recommended as stock solutions.

7.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

7.4 Carbonate-bicarbonate, stock solution, 1000 mg carbon/liter: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 ml volumetric flask. Dissolve with distilled water.

7.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to step 7.3.

NOTE 3: This standard is not required by some instruments.

7.6 Blank solution: Use the same distilled water (or similar quality water) used for the preparation of the standard solutions.

8. Procedure

8.1 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

8.2 For calibration of the instrument, it is recommended that a series of standards encompassing the expected concentration range of the samples be used.

9. Precision and Accuracy

9.1 Twenty-eight analysts in twenty-one laboratories analyzed distilled water solutions containing exact increments of oxidizable organic compounds, with the following results:

Increment as TOC mg/liter	Precision as Standard Deviation TOC, mg/liter	Accuracy as Bias, %	Accuracy as Bias, mg/liter
4.9	3.93	+15.27	+0.75
107	8.32	+ 1.01	+1.08

(FWPCA Method Study 3, Demand Analyses)

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2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 532, Method 505, (1975).

METHOD 9020

TOTAL ORGANIC HALIDES (TOX)

1.0 Scope and Application

1.1 Method 9020 determines Total Organic Halides (TOX) as Cl^- in drinking and ground waters. The method uses carbon adsorption with a microcoulometric-titration detector. It requires that all samples be run in duplicate. Under conditions of duplicate analysis, the reliable limit of sensitivity is 5 µg/l.

1.2 Method 9020 detects all organic halides containing chlorine, bromine and iodine that are adsorbed by granular activated carbon under the conditions of the method. Fluorine-containing species are not determined by this method.

1.3 Method 9020 is applicable to samples whose inorganic-halide concentration does not exceed the organic-halide concentration by more than 20,000 times.

1.4 Method 9020 is restricted to use by, or under the supervision of, analysts experienced in the operation of a pyrolysis/microcoulometer and in the interpretation of the results.

1.5 This method is provided as a recommended procedure. It may be used as a reference for comparing the suitability of other methods thought to be appropriate for measurement of TOX (i.e., by comparison of sensitivity, accuracy, and precision data).

2.0 Summary of Method

2.1 A sample of water that has been protected against the loss of volatiles by the elimination of headspace in the sampling container, and that is free of undissolved solids, is passed through a column containing 40 mg of activated carbon. The column is washed to remove any trapped inorganic halides, and is then analyzed to convert the adsorbed organohalides to a titratable species that can be measured by a microcoulometric detector.

3.0 Interferences

3.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be

routinely demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

3.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by treating with chromate cleaning solution. This should be followed by detergent washing in hot water. Rinse with tap water and distilled water, drain dry, and heat in a muffle furnace at 400° C for 15 to 30 min. Volumetric ware should not be heated in a muffle furnace. Glassware should be sealed and stored in a clean environment after drying and cooling to prevent any accumulation of dust or other contaminants.

3.1.2 The use of high purity reagents and gases helps to minimize interference problems.

3.2 Purity of the activated carbon must be verified before use. Only carbon samples that register less than 1000 ng/40 mg should be used. The stock of activated carbon should be stored in its granular form in a glass container with a Teflon seal. Exposure to the air must be minimized, especially during and after milling and sieving the activated carbon. No more than a two-week supply should be prepared in advance. Protect carbon at all times from all sources of halogenated organic vapors. Store prepared carbon and packed columns in glass containers with Teflon seals.

4.0 Apparatus and Materials

4.1 Adsorption system

4.1.1 Dohrmann adsorption module (AD-2), or equivalent, pressurized, sample and nitrate-wash reservoirs.

4.1.2 Adsorption columns: Pyrex, 5-cm-long x 6-mm-O.D. x 2-mm-I.D.

4.2.3 Granular activated carbon (GAC): Filtrasorb-400, Calgon-APC or equivalent, ground or milled, and screened to a 100/200 mesh range. Upon combustion of 40 mg of GAC, the apparent-halide background should be 1000 mg Cl^- equivalent or less.

4.1.4 Cerafelt (available from Johns-Manville), or equivalent: Form this material into plugs using a 2-mm-I.D. stainless-steel borer with ejection rod (available from Dohrmann) to hold 40 mg of GAC in the adsorption columns. CAUTION: Do not touch this material with your fingers.

4.1.5 Column holders (available from Dohrmann).

4.1.6 Volumetric flasks: 100-ml, 50-ml. A general schematic of the adsorption system is shown in Figure 1.

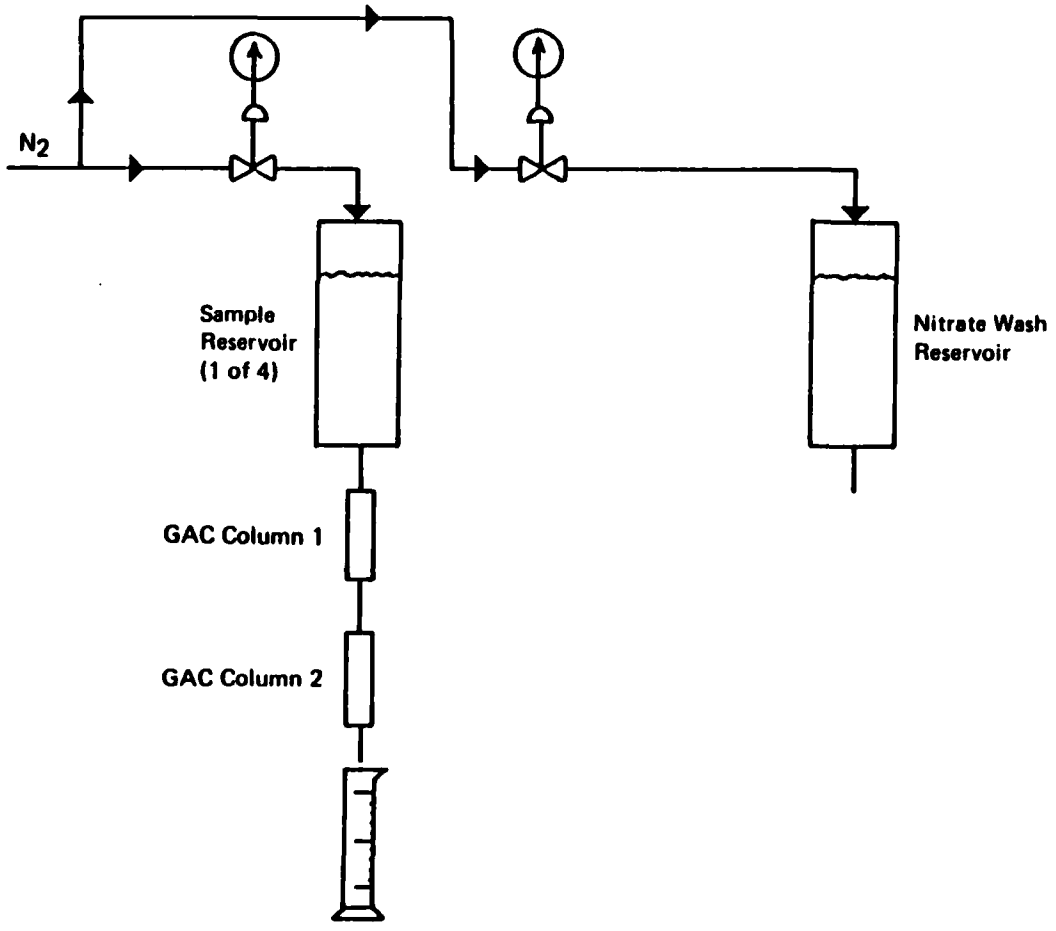


Figure 1. Schematic of Adsorption System.

4.2 Dohrmann microcoulometric-titration system (MCTS-20 or DX-20), or equivalent, containing the following components:

4.2.1 Boat sampler.

4.2.2 Pyrolysis furnace.

4.2.3 Microcoulometer with integrator.

4.2.4 Titration cell: A general description of the analytical system is shown in Figure 2.

4.3 Strip chart recorder.

5.0 Reagents

5.1 Sodium sulfite: 0.1 M, ACS reagent grade (12.6 g/liter).

5.2 Nitric acid: Concentrated.

5.3 Nitrate-wash solution (5000 mg NO_3^-/l): Prepare a nitrate-wash solution by transferring approximately 8.2 g of potassium nitrate into a 1-liter volumetric flask and diluting to volume with reagent water.

5.4 Carbon dioxide: Gas, 99.9% purity.

5.5 Oxygen: 99.9% purity.

5.6 Nitrogen: Prepurified.

5.7 70% acetic acid in water: Dilute 7 volumes of acetic acid with 3 volumes of water.

5.8 Trichlorophenol solution, stock (1 µl = 10 µg Cl^-): Prepare a stock solution by weighing accurately 1.856 g of trichlorophenol into a 100-ml volumetric flask. Dilute to volume with methanol. 10,000 ng/ml

5.9 Trichlorophenol solution, calibration (1 µl = 500 ng Cl^-): = 500 ng/ml
Dilute 5 ml of the trichlorophenol stock solution to 100 ml with methanol.

5.10 Trichlorophenol standard, instrument-calibration: First, nitrate-wash a single column packed with 40 mg of activated carbon as instructed for sample analysis, and then inject the column with 10 µl of the calibration solution.

5.11 Trichlorophenol standard, adsorption-efficiency (100 µg Cl^-/liter): Prepare an adsorption-efficiency standard by injecting 10 µl of stock solution into 1 liter of reagent water.

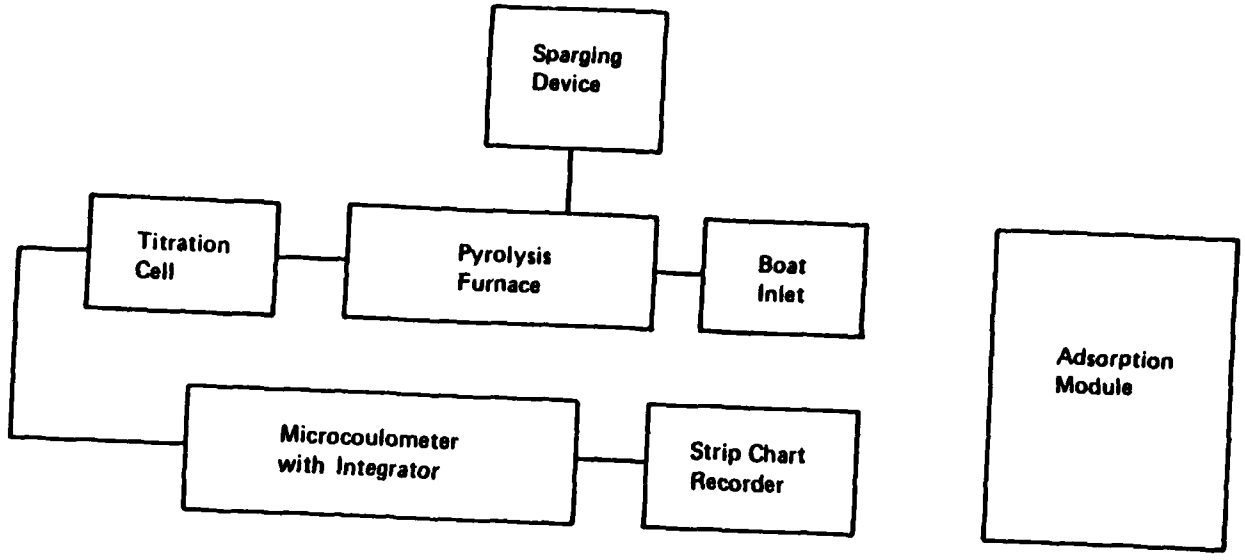


Figure 2. Schematic diagram of CAOX analysis system.

5.12 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

5.13 Blank standard: The reagent water used to prepare the calibration standard should be used as the blank standard.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All samples should be collected in bottles with teflon septa (e.g., Pierce #12722 or equivalent) and be protected from light. If this is not possible, use amber glass, 250-ml, fitted with teflon-lined caps. Foil may be substituted for teflon if the sample is not corrosive. Samples must be protected against loss of volatiles by eliminating headspace in the container. If amber bottles are not available, protect samples from light. The container must be washed and muffled at 400° C before use, to minimize contamination.

6.3 All glassware must be dried prior to use according to the method discussed in 3.1.1.

7.0 Procedure

7.1 Sample preparation

7.1.1 Special care should be taken in handling the sample in order to minimize the loss of volatile organohalides. The adsorption procedure should be performed simultaneously on duplicates.

7.1.2 Reduce residual chlorine by adding sulfite (1 ml of 0.1 M per liter of sample). Sulfite should be added at the time of sampling if the analysis is meant to determine the TOX concentration at the time of sampling. It should be recognized that TOX may increase on storage of the sample. Samples should be stored at 4° C without headspace.

7.1.3 Adjust the pH of the sample to approximately 2 with concentrated HNO_3 just prior to adding the sample to the reservoir.

7.2 Calibration

7.2.1 Check the adsorption efficiency of each newly-prepared batch of carbon by analyzing 100 ml of the adsorption-efficiency standard, in duplicate, along with duplicates of the blank standard. The net recovery should be within 5% of the standard value.

7.2.2 Nitrate-wash blanks (method blanks): Establish the repeatability of the method background each day by first analyzing several nitrate-wash blanks. Monitor this background by spacing nitrate-wash blanks between each group of eight pyrolysis determinations. The nitrate-wash blank values are obtained on single columns packed with 40 mg of activated carbon. Wash with the nitrate solution as instructed for sample analysis, and then pyrolyze the carbon.

7.2.3 Pyrolyze duplicate instrument-calibration standards and the blank standard each day before beginning sample analysis. The net response to the calibration-standard should be within 3% of the calibration-standard value. Repeat analysis of the instrument-calibration standard after each group of eight pyrolysis determinations, and before resuming sample analysis after cleaning or reconditioning the titration cell or pyrolysis system.

7.3 Adsorption procedure

7.3.1 Connect two columns in series, each containing 40 mg of 100/200-mesh activated carbon.

7.3.2 Fill the sample reservoir, and pass a metered amount of sample through the activated-carbon columns at a rate of approximately 3 ml/min. NOTE: 100 ml of sample is the preferred volume for concentrations of TOX between 5 and 500 µg/l; 50 ml for 501 to 1000 µg/l, and 25 ml for 1001 to 2000 µg/l.

7.3.3 Wash the columns-in-series with 2 ml of the 5000-mg/l nitrate solution at a rate of approximately 2 ml/min to displace inorganic chloride ions.

7.4 Pyrolysis procedure

7.4.1 The contents of each column are pyrolyzed separately. After rinsing with the nitrate solution, the columns should be protected from the atmosphere and other sources of contamination until ready for further analysis.

7.4.2 Pyrolysis of the sample is accomplished in two stages. The volatile components are pyrolyzed in a CO_2-rich atmosphere at a low temperature to ensure the conversion of brominated trihalomethanes to a titratable species. The less volatile components are then pyrolyzed at a high temperature in an O_2-rich atmosphere. NOTE: The quartz sampling boat should have been previously muffled at 800° C for at least 2 to 4 min as in a previous analysis, and should be cleaned of any residue by vacuuming.

7.4.3 Transfer the contents of each column to the quartz boat for individual analysis.

7.4.4 If the Dohrmann MC-1 is used for pyrolysis, manual instructions are followed for gas flow regulation. If the MCTS-20 is used, the information on the diagram in Figure 3 is used for gas flow regulation.

7.4.5 Position the sample for 2 min in the 200° C zone of the pyrolysis tube. For the MCTS-20, the boat is positioned just outside the furnace entrance.

7.4.6 After 2 min, advance the boat into the 800° C zone (center) of the pyrolysis furnace. This second and final stage of pyrolysis may require from 6 to 10 min to complete.

7.5 Detection: The effluent gases are directly analyzed in the micro-coulometric-titration cell. Carefully follow manual instructions for optimizing cell performance.

7.6 Breakthrough. The unpredictable nature of the background bias makes it especially difficult to recognize the extent of breakthrough of organohalides from one column to another. All second-column measurements for a properly operating system should not exceed 10% of the two-column total measurement. If the 10% figure is exceeded, one of three events can be happening. Either (1) the first column was overloaded and a legitimate measure of breakthrough was obtained, in which case taking a smaller sample may be necessary; or (2) channeling or some other failure occurred, in which case the sample may need to be rerun; or (3) a high random bias occurred and the result should be rejected and the sample rerun. Because it may not be possible to determine which event occurred, a sample analysis should be repeated often enough to gain confidence in results. As a general rule, any analysis that is rejected should be repeated whenever sample is available. If the second-column measurement is equal to or less than the nitrate-wash blank value, the second-column value should be disregarded.

7.7 Calculations: TOX as Cl^- is calculated using the following formula:

$$\frac{(C_1-C_3) + (C_2 - C_3)}{V} = \mu g/l \text{ Total Organic Halide}$$

where:

C_1 = µg Cl^- on the first column in series

C_2 = µg Cl^- on the second column in series

C_3 = predetermined, daily, average, method-blank value (nitrate-wash blank for a 40-mg carbon column)

V = the sample volume in liters.

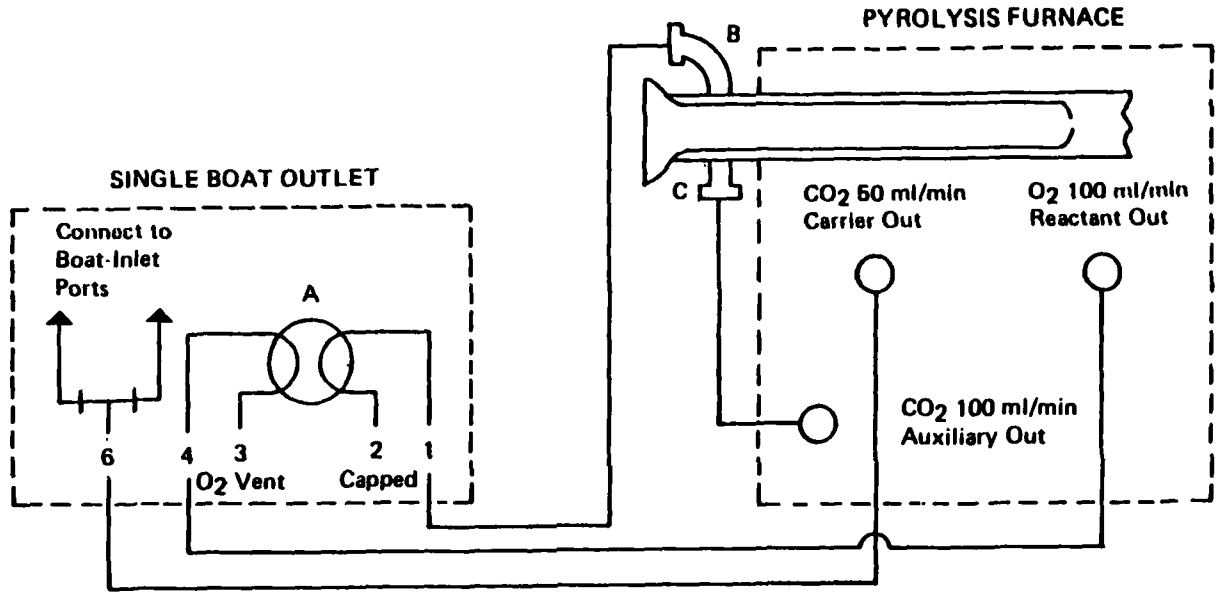


Figure 3. Rear-view plumbing schematic for MCTS–20 System. Valve A is set for first-stage combustion, O_2 venting (push/pull valve out). Port B enters inner combustion tube; Port C enters outer combustion tube.

8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this procedure by analyzing appropriate quality-control check samples.

8.3 The laboratory must develop and maintain a statement of method accuracy for their laboratory. The laboratory should update the accuracy statement regularly as new recovery measurements are made.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Run check standard after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparations process.

8.7 It is recommended that the laboratory adopt additional quality-assurance practices for use with this method. The specific practices that would be most productive will depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance-evaluation studies.

OIL AND GREASE, TOTAL RECOVERABLE

Method 413.2 (Spectrophotometric, Infrared)

STORET NO. 00560

1. Scope and Application
   - 1.1 This method includes the measurement of fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.
   - 1.2 The method is applicable to measurement of most light petroleum fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.
   - 1.3 The method covers the range from 0.2 to 1000 mg/l of extractable material.
   - 1.4 While this method can be used to obtain an estimate of the oil and grease that would be measured gravimetrically, in many cases the estimate more accurately describes the parameter, as it will measure volatiles more effectively and is not susceptible to interferences such as extractable sulfur. It can be used with the Petroleum Hydrocarbon procedure to obtain an oil and grease value and a petroleum hydrocarbon value on the same sample.
2. Summary of Method
   - 2.1 The sample is acidified to a low pH (< 2) and extracted with fluorocarbon-113. The oil and grease is determined by comparison of the infrared absorbance of the sample extract with standards.
3. Definitions
   - 3.1 The definition of oil and grease is based on the procedure used. The source of the oil and/or grease, and the presence of extractable non-oily matter will influence the material measured and interpretation of results.
4. Sampling and Storage
   - 4.1 A representative sample of 1 liter volume should be collected in a glass bottle. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 5 ml HCl (6.1) at the time of collection and refrigerated at 4°C.
   - 4.2 Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentration over an extended period.
5. Apparatus
   - 5.1 Separatory funnel, 2000 ml, with Teflon stopcock.
   - 5.2 Infrared spectrophotometer, scanning. Non-scanning instruments may also be used but can be subject to positive interferences in complex chemical wastewaters.
   - 5.3 Cells, 10 mm, 50 mm, and 100 mm path length, sodium chloride or infrared grade glass.
   - 5.4 Filter paper, Whatman No. 40, 11 cm.

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6. Reagents

6.1 Hydrochloric acid, 1:1. Mix equal volumes of conc. HCl and distilled water.

6.2 Fluorocarbon-113, (1,1,2-trichloro-1,2,2-trifluoroethane), b. p. 48°C.

6.3 Sodium sulfate, anhydrous crystal.

6.4 Calibration mixtures:

6.4.1 Reference oil: Pipet 15.0 ml n-hexadecane, 15.0 ml isooctane, and 10.0 ml chlorobenzene into a 50 ml glass stoppered bottle. Maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

6.4.2 Stock standard: Pipet 1.0 ml reference oil (6.4.1) into a tared 200 ml volumetric flask and immediately stopper. Weigh and dilute to volume with fluorocarbon-113.

6.4.3 Working standards: Pipet appropriate volumes of stock standard (6.4.2) into 100 ml volumetric flasks according to the cell pathlength to be used. Dilute to volume with fluorocarbon-113. Calculate concentration of standards from the stock standard.

7. Procedure

7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 ml hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.

7.2 Pour the sample into a separatory funnel.

7.3 Add 30 ml fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate.

7.4 Filter the solvent layer into a 100 ml volumetric flask through a funnel containing solvent-moistened filter paper.

NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

7.5 Repeat (7.3 and 7.4) twice more with 30 ml portions of fresh solvent, combining all solvent in the volumetric flask.

7.6 Rinse the tip of the separatory funnel, filter paper, and the funnel with a total of 5–10 ml fluorocarbon-113 and collect the rinsings in the flask. Dilute the extract to 100 ml, and stopper the flask.

7.7 Select appropriate working standards and cell pathlength according to the following table of approximate working ranges:

Pathlength	Range
10 mm	2–40 mg
50 mm	0.4–8 mg
100 mm	0.1–4 mg

7.8 Scan standards and samples from 3200 cm^{-1} to 2700 cm^{-1} with fluorocarbon-113 in the reference beam and record the results on absorbance paper. The absorbances of samples

and standards are measured by constructing a straight baseline over the range of the scan and measuring the absorbance of the peak maximum at 2930 cm^{-1} and subtracting the baseline absorbance at that point. For an example of a typical oil spectrum and baseline construction, see Gruenfeld[3]. Non-scanning instruments should be operated according to manufacturer's instructions, although calibration must be performed using the standards described above (6.4). If the absorbance exceeds 0.8 for a sample, select a shorter pathlength or dilute as required.

7.9 Use a calibration plot of absorbance vs. mg oil prepared from the standards to determine the mg oil in the sample solution.

8. Calculation

8.1 $$\text{mg/l total oil and grease} = \frac{R \times D}{V}$$

where:

R = oil in solution, determined from calibration plot, in milligrams.
D = extract dilution factor, if used.
V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

9. Precision and Accuracy

9.1 The two oil and grease methods in this manual were tested by a single laboratory (EMSL) on sewage. This method determined the oil and grease level in the sewage to be 17.5 mg/l. When 1 liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 99% with a standard deviation of ±1.4 mg/l.

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PHENOLICS, TOTAL RECOVERABLE

Method 420.1 (Spectrophotometric, Manual 4-AAP with Distillation)

STORET NO. 32730

1. Scope and Application
   - 1.1 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The method is capable of measuring phenolic materials at the 5 *ug/l* level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.
   - 1.3 The method is capable of measuring phenolic materials that contain more than 50 *ug/l* in the aqueous phase (without solvent extraction) using phenol as a standard.
   - 1.4 It is not possible to use this method to differentiate between different kinds of phenols.
2. Summary of Method
   - 2.1 Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.
3. Comments
   - 3.1 For most samples a preliminary distillation is required to remove interfering materials.
   - 3.2 Color response of phenolic materials with 4-amino antipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.
4. Sample Handling and Preservation
   - 4.1 Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.
5. Interference
   - 5.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4 with H_3PO_4 and aerating briefly by stirring and adding $CuSO_4$.
   - 5.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate (~~6.5~~ 2.5). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

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6. Apparatus
   - 6.1 Distillation apparatus, all glass consisting of a 1 liter pyrex distilling apparatus with Graham condenser.
   - 6.2 pH meter.
   - 6.3 Spectrophotometer, for use at 460 or 510 nm.
   - 6.4 Funnels.
   - 6.5 Filter paper.
   - 6.6 Membrane filters.
   - 6.7 Separatory funnels, 500 or 1,000 ml.
   - 6.8 Nessler tubes, short or long form.
7. Reagents
   - 7.1 Phosphoric acid solution, 1 + 9: Dilute 10 ml of 85% H_3PO_4 to 100 ml with distilled water.
   - 7.2 Copper sulfate solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in distilled water and dilute to 1 liter.
   - 7.3 Buffer solution: Dissolve 16.9 g NH_4Cl in 143 ml conc. NH_4OH and dilute to 250 ml with distilled water. Two ml should adjust 100 ml of distillate to pH 10.
   - 7.4 Aminoantipyrine solution: Dissolve 2 g of 4AAP in distilled water and dilute to 100 ml.
   - 7.5 Potassium ferricyanide solution: Dissolve 8 g of $K_3Fe(CN)_6$ in distilled water and dilute to 100 ml.
   - 7.6 Stock phenol solution: Dissolve ~~1.0 g~~ 1.00g phenol in freshly boiled and cooled distilled water and dilute to 1 liter. 1 ml = 1 mg phenol.
   - 7.7 Working solution A: Dilute 10 ml stock phenol solution to 1 liter with distilled water. 1 ml = 10 *u*g phenol.
   - 7.8 Working solution B: Dilute 100 ml of working solution A to 1000 ml with distilled water. 1 ml = 1 *u*g phenol.
   - 7.9 Chloroform
8. Procedure
   - 8.1 Distillation
     - 8.1.1 Measure 500 ml sample into a beaker. Lower the pH to approximately 4 with 1 + 9 H_3PO_4 (7.1), add 5 ml $CuSO_4$ solution (7.2) and transfer to the distillation apparatus. Omit adding H_3PO_4 and $CuSO_4$ if sample was preserved as described in 4.1.
     - 8.1.2 Distill 450 ml of sample, stop the distillation, and when boiling ceases add 50 ml of warm distilled water to the flask and resume distillation until 500 ml have been collected.
     - 8.1.3 If the distillate is turbid, filter through a prewashed membrane filter.
   - 8.2 Direct photometric method
     - 8.2.1 Using working solution A (7.7), prepare the following standards in 100 ml volumetric flasks.

ml of working solution A	Conc. ug/l
0	0.0
0.5	50.0
1.0	100.0
2.0	200.0
5.0	500.0
8.0	800.0
10.0	1000.0

8.2.2 To 100 ml of distillate or an aliquot diluted to 100 ml and/or standards, add 2 ml of buffer solution (7.3) and mix. The pH of the sample and standards should be 10 ±0.2.

8.2.3 Add 2.0 ml aminoantipyrine solution (7.4) and mix.

8.2.4 Add 2.0 ml potassium ferricyanide solution (7.5) and mix.

8.2.5 After 15 minutes read absorbance at 510 nm.

8.3 Chloroform extraction method

8.3.1 Using working solution B (7.8), prepare the following standards. Standards may be prepared by pipetting the required volumes into the separatory funnels and diluting to 500 ml with distilled water.

ml of working solution B	Conc. ug/l
0.0	0.0
3.0	6.0
5.0	10.0
10.0	20.0
20.0	40.0
25.0	50.0

8.3.2 Place 500 ml of distillate or an aliquot diluted to 500 ml in a separatory funnel. The sample should not contain more than 25 ug phenol.

8.3.3 To sample and standards add 10 ml of buffer solution (7.3) and mix. The pH should be 10 ±0.2.

8.3.4 Add 3.0 ml aminoantipyrine solution (7.4) and mix.

8.3.5 Add 3.0 ml potassium ferricyanide solution (7.5) and mix.

8.3.6 After three minutes, extract with 25 ml of chloroform (7.9). Shake the separatory funnel at least 10 times, let $CHCl_3$ settle, shake again 10 times and let chloroform settle again.

8.3.7 Filter chloroform extracts through filter paper. Do not add more chloroform.

8.3.8 Read the absorbance of the samples and standards against the blank at 460 nm.

9. Calculation

9.1 Prepare a standard curve by plotting the absorbance value of standards versus the corresponding phenol concentrations.

9.2 Obtain concentration value of sample directly from standard curve.

10. Precision and Accuracy

10.1 Using the extraction procedure for concentration of color, six laboratories analyzed samples at concentrations of 9.6, 48.3, and 93.5 *ug*/1. Standard deviations were ±0.99, ±3.1 and ±4.2 *ug*/1, respectively.

10.2 Using the direct photometric procedure, six laboratories analyzed samples at concentrations of 4.7, 48.2 and 97.0 mg/1. Standard deviations were ±0.18, ±0.48 and ±1.58 mg/1, respectively.

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CYANIDE, TOTAL

Method 335.2 (Titrimetric; Spectrophotometric)

STORET NO. 00720

1. Scope and Application
   - 1.1 This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The titration procedure using silver nitrate with p-dimethylamino-benzal-rhodanine indicator is used for measuring concentrations of cyanide exceeding 1 mg/1 (0.25 mg/250 ml of absorbing liquid).
   - 1.3 The colorimetric procedure is used for concentrations below 1 mg/1 of cyanide and is sensitive to about 0.02 mg/1.
2. Summary of Method
   - 2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.
   - 2.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.
   - 2.3 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.
3. Definitions
   - 3.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.
4. Sample Handling and Preservation
   - 4.1 The sample should be collected in plastic or glass bottles of 1 liter or larger size. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.
   - 4.2 Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

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4.3 Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH $\geq$ 12) at the time of collection.

4.4 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C.

5. Interferences

5.1 Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1 through 8.5.

5.2 Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 ml more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide as described in 4.3.

5.3 Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect.

5.3.1 Acidify the sample with acetic acid (1 + 9) to pH 6.0 to 7.0.

Caution: This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed.

5.3.2 Extract with iso-octane, hexane, or chloroform (preference in order named) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.

6. Apparatus

6.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber.

6.2 Microburet, 5.0 ml (for titration).

6.3 Spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.

7. Reagents

7.1 Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.

7.2 Cadmium carbonate: powdered.

7.3 Ascorbic acid: crystals.

7.4 Dilute sodium hydroxide solution, 0.25N: Dilute 200 ml of sodium hydroxide solution (7.1) to 1000 ml with distilled water.

7.5 Sulfuric acid: concentrated.

7.6 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in 1 liter of distilled water. Refrigerate this solution.

7.7 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 liter of distilled water. Standardize with 0.0192 N $AgNO_3$. Dilute to appropriate concentration so that 1 ml = 1 mg CN.

7.8 Standard cyanide solution, intermediate: Dilute 50.0 ml of stock (1 ml = 1 mg CN) to 1000 ml with distilled water (1 ml = 50.0ug).

7.9 Standard cyanide solution: Prepare fresh daily by diluting 100.0 ml of intermediate cyanide solution to 1000 ml with distilled water and store in a glass stoppered bottle. 1 ml = 5.0ug CN (5.0 mg/1).

7.10 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ crystals and drying to constant weight at 40°C. Weigh out 3.2647 g of dried $AgNO_3$, dissolve in distilled water, and dilute to 1000 ml (1 ml = 1mg CN).

7.11 Rhodanine indicator: Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100 ml of acetone.

7.12 Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly.

7.13 Color Reagent — One of the following may be used:

7.13.1 Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCl (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

7.13.2 Pyridine-pyrazolone solution:

7.13.2.1 3-Methyl-1-phenyl-2-pyrazolin-5-one reagent, saturated solution: Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolin-5-one to 50 ml of distilled water, heat to 60°C with stirring. Cool to room temperature.

7.13.2.2 3,3'Dimethyl-1, 1'-diphenyl-[4,4'-bi-2 pyrazoline]-5,5'dione (bispyrazolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine.

7.13.2.3 Pour solution (7.13.2.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (7.13.2.2) collecting the filtrate in the same container as filtrate from (7.13.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.

7.14 Magnesium chloride solution: Weight 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 ml flask, dissolve and dilute to 1 liter with distilled water.

8. Procedure

8.1 Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Add 50 ml of sodium hydroxide (7.1) to the absorbing tube and dilute if necessary with distilled

water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

8.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

Caution: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

8.3 Slowly add 25 ml conc. sulfuric acid (7.5) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride (7.4) into the air inlet and wash down with a stream of water.

8.4 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

8.5 Drain the solution from the absorber into a 250 ml volumetric flask and bring up to volume with distilled water washings from the absorber tube.

8.6 Withdraw 50 ml or less of the solution from the flask and transfer to a 100 ml volumetric flask. If less than 50 ml is taken, dilute to 50 ml with 0.25 N sodium hydroxide solution (7.4). Add 15.0 ml of Sodium phosphate solution (7.6) and mix.

8.6.1 Pyridine – Barbituric Acid Method: Add 2 ml of chloramine T (7.12) and mix. After 1 to 2 minutes, add 5 ml of pyridine – barbituric acid solution (7.13.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

8.6.2 Pyridine – pyrazolone method: Add 0.5 ml of chloramine T (7.12) and mix. After 1 to 2 minutes add 5 ml of pyridine – pyrazolone solution (7.13.1) and mix. Dilute to mark with distilled water and mix again. After 40 minutes read absorbance at 620 nm in a 1 cm cell.

NOTE: More than 0.5 ml of Chloramine T will prevent the color from developing with pyridine-pyrazolone.

8.7 Prepare a series of standards by pipeting suitable volumes of standard solution into 250 ml volumetric flasks. To each standard add 50 ml of 1.25 N sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ML of Standard Solution (1.0 = 5 ug CN)	Conc. mgCN per 250 ml
0	BLANK
1.0	5
2.0	10
5.0	25
10.0	50
15.0	60
20.0	100

8.7.1 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree within ±10% of the undistilled standards the operator should find the cause of the apparent error before proceeding.

8.7.2 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

8.7.3 To check the efficiency of the sample distillation, add an increment of cyanide from either the intermediate standard (7.8) or the working standard (7.9) to insure a level of 20 *ug*/l or a significant increase in absorbance value. Proceed with the analysis as in Procedure (8.8.1) using the same flask and system from which the previous sample was just distilled.

8.8 Alternatively, if the sample contains more than 1 mg of CN transfer the distillate, or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10–12 drops of the benzalrhodanine indicator.

8.9 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

8.10 The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 ml microburet may be conveniently used to obtain a more precise titration.

9. Calculation

9.1 If the colorimetric procedure is used, calculate the cyanide, in *ug*/l, in the original sample as follows:

$$\text{CN, } ug/l = \frac{A \times 1{,}000}{B} \times \frac{50}{C}$$

where:

A = *ug* CN read from standard curve
B = ml of original sample for distillation
C = ml taken for colorimetric analysis

9.2 Using the titrimetric procedure, calculate concentration of CN as follows:

$$\text{CN, mg/l} = \frac{(A - B)1{,}000}{\text{ml orig. sample}} \times \frac{250}{\text{ml of aliquot titrated}}$$

where:

A = volume of $AgNO_3$ for titration of sample.
B = volume of $AgNO_3$ for titration of blank.

10. Precision and Accuracy

10.1 In a single laboratory (EMSL), using mixed industrial and domestic waste samples at concentrations of 0.06, 0.13, 0.28 and 0.62 mg/l CN, the standard deviations were ±0.005, ±0.007, ±0.031 and ±0.094, respectively.

10.2 In a single laboratory (EMSL), using mixed industrial and domestic waste samples at concentrations of 0.28 and 0.62 mg/l CN, recoveries were 85% and 102%, respectively.

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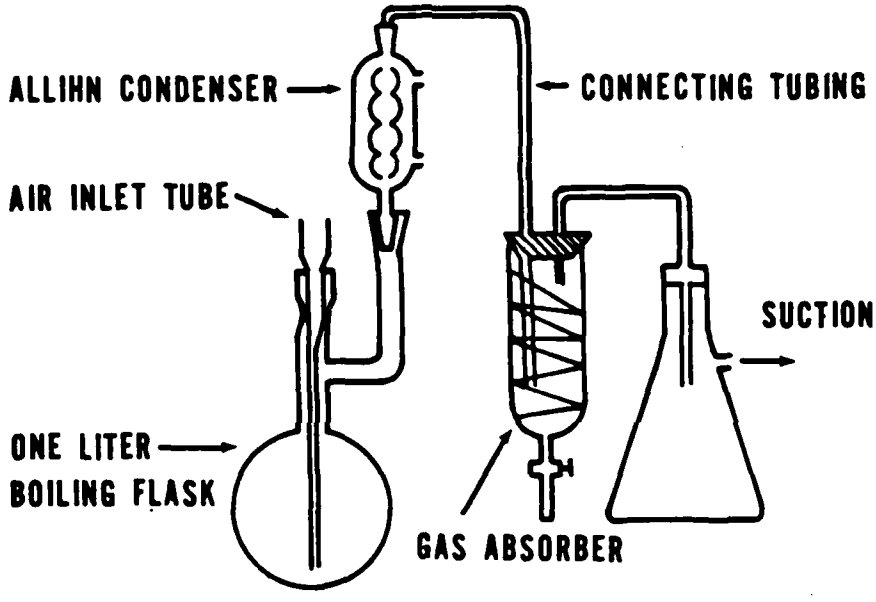


FIGURE 1

CYANIDE DISTILLATION APPARATUS

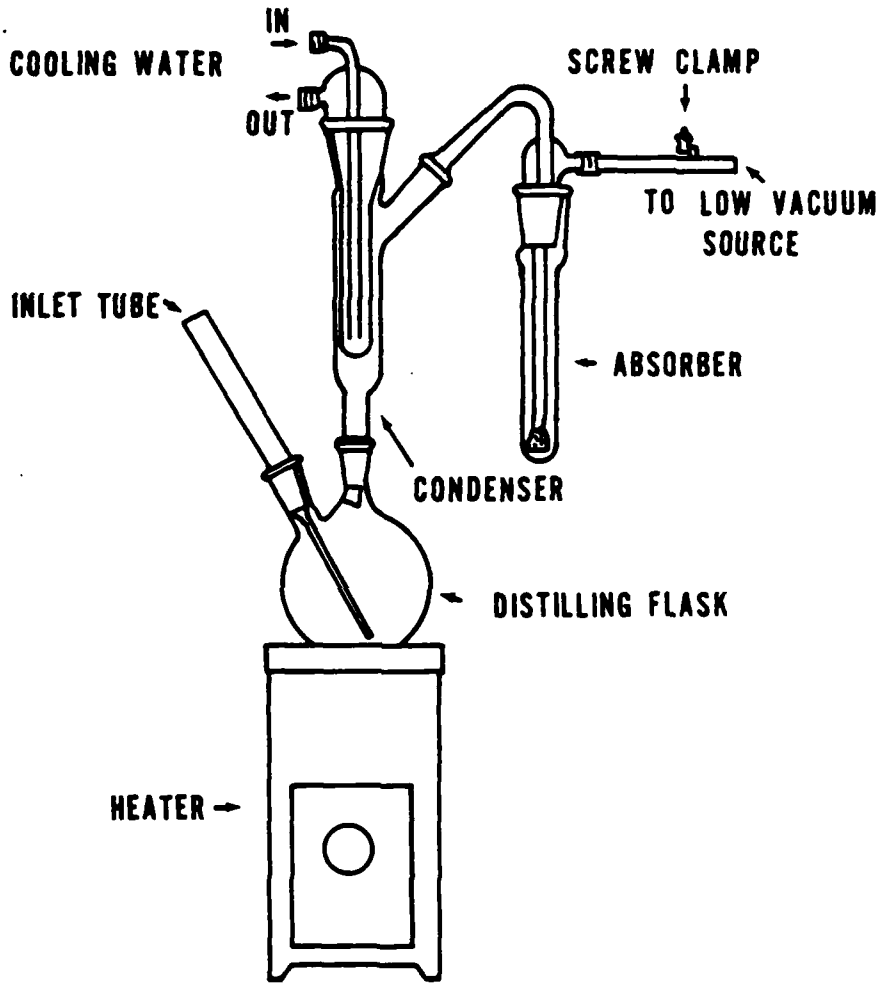


FIGURE 2
CYANIDE DISTILLATION APPARATUS

Appendix IV.—Inductively Coupled Plasma Optical Emission Spectrometric Method (ICP) for Trace Element Analysis of Water and Wastes

Inductively Coupled Plasma (ICP) Optical Emission Spectrometric Method for Trace Element Analysis of Water and Wastes

Interim

U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268

October 1979.

Foreword

This method has been prepared by the staff of the Environmental Monitoring and Support Laboratory—Cincinnati, with the cooperation of the EPA–ICP Users Group. Their cooperation and support is gratefully acknowledged.

This method represents the current state-of-the-art, but as time progresses, improvements are anticipated. Users are encouraged to identify problems and assist in updating the method by contacting the Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

Inductively Coupled Plasma (ICP) Optical Emission Spectrometric Method for Trace Element Analysis of Water and Wastes

1. Scope and Application.

1.1 This method may be used for the determination of dissolved, suspended, or total elements in surface water, drinking water, and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken to ensure that potential interference are taken into account when dissolved solids exceed 1500 mg/l. (See 4.2)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps *must* be taken to correct for potential interference effects.

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

Table 1—*Recommended Wavelengths* [1] and Estimated Instrumental Detection Limits

Element	Wavelength, nm	Estimated detection limit, μg/l [2]
Aluminum	308.2	45
Arsenic	193.7	53
Barium	455.5	2
Beryllium	313.0	0.3
Boron	249.8	5
Cadmium	226.5	4
Calcium	317.9	10
Chromium	267.7	7
Cobalt	228.6	7
Copper	324.7	8
Iron	259.9	7
Lead	220.3	42
Lithium	670.7	4
Magnesium	279.1	30
Manganese	257.6	2
Molybdenum	202.0	8
Nickel	231.6	15
Potassium	766.4	see [3]
Selenium	196.0	75
Silica (SiO_2)	288.1	27
Silver	328.0	7
Sodium	588.0	29
Strontium	407.7	0.5
Vanadium	292.4	8
Zinc	213.8	2

[1] The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can proved the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 4.1.1).

[2] The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Optical Emission Spectroscopy Prominent Lines," EPA–600/4–79–017. Detection limits are sample dependent and as the sample matrix varies, these concentration values may also vary.

[3] Highly dependent on operating conditions and plasma position.

2. Summary of Method.

2.1 The method describes a technique for the simultaneous of sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. Additional interferences named in 4.1 should also be recognized and appropriate corrections made.

3. Definitions.

3.1 *Dissolved*—Those elements which will pass through a 0.45 μm membrane filter.

3.2 *Suspended*—Those elements which are retained by a 0.45 μm membrane filter.

3.3 *Total*—The concentration determined on an unfiltered sample following vigorous digestion (Section 8.3), or the sum of the dissolved plus suspended concentrations (Section 8.1 plus 8.2).

3.4 *Total recoverable*—The concentration determined on an

unfiltered sample following treatment with hot, dilute mineral acid (Section 8.4).

3.5 *Instrumental detection limit*—The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 *Sensitivity*—The slope of the analytical curve, i.e. funtional relationship between emission intensity and concentration.

3.7 *Instrument check standard*—A multielement standard of known concentrations prepared by the analyst. Should be included in the analytical scheme with a frequency of 10%. (See 6.6.1.)

3.8 *Reference standard*—A solution obtained from an outside source having known, verified values. Must be used initially to verify the calibration standards and analyzed thereafter as a blind sample on a weekly frequency. (See 6.6.2.)

3.9 *Calibration standards*—A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 6.4.)

3.10 *Linear dynamic range*—The concentration range over which the analytical curve remains linear.

3.11 *Reagent blank*—A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 6.5.2.)

3.12 *Calibration blank*—A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 6.5.1.)

3.13 *Method of standard addition*—The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 9.6.1.)

4. Interferences.

4.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

4.1.1 *Spectral interferences* can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line.

4.1.2 *Physical interferences* are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. (See Note 1.) If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques.

Note 1.—The use of a peristaltic pump may lessen these interferences.

4.1.3 *Chemical interferences* are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

4.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 4.2.1 through 4.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

4.2.1 *Serial dilution*—If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 percent of the original determination (or within some acceptable control limit (13.3) that has been established for that matrix). If not, a chemical or physical interference effect should be suspected.

4.2.2 *Spike addition*—The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

Caution.—The standard addition technique does not detect coincident spectral overlap. If suspected, use of an alternate wavelength or comparison with an alternate method is recommended (See 4.2.3).

4.2.3 *Comparison with alternate method of analysis*—When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

4.2.4 *Wavelength scanning of analyte line region*—If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

5. Apparatus.

5.1 Inductively Coupled Plasma (ICP) Optical Emission Spectrometer.

5.1.1 Computer controlled atomic emission spectrometer with background correction.

5.1.2 Radiofrequency generator.

5.1.3 Argon gas supply, welding grade or better.

5.2 Operating conditions—Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument.

6. Reagents and standards.

6.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

6.1.1 *Acetic acid*, conc. (sp gr 1.06).

6.1.2 *Aqua regia:* Mix cautiously 3 parts conc. HCl (sp gr 1.19) and 1 part conc. HNO_3 (sp gr 1.41) just before use.

6.1.3 *Hydrochloric acid*, conc. (sp gr 1.19).

6.1.4 *Hydrochloric acid*, (1+1): Add 500 ml conc. HCl (sp gr 1.19) to 400 ml deionized, distilled water and dilute to 1 liter.

6.1.5 *Nitric acid*, conc. (sp gr 1.41).

6.1.6 *Nitric acid*, (1+1): Add 500 ml conc. HNO_3 (sp gr 1.41) to 400 ml deionized, distilled water and dilute to 1 liter.

6.2 *Deionized, distilled water*—Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water.

6.3 *Standard stock solutions* may be purchased or prepared from ultra high purity grade chemicals or metals

(Caution: See Note 2). All salts must be dried for 1 h at 105° C unless otherwise specified.

Note 2.—Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow:

6.3.1 *Aluminum solution, stock.* 1 ml = 100 μg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 ml of (1+1) HCl and 1 ml of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 ml of (1+1) HCl and dilute to 1,000 ml with deionized, distilled water.

6.3.2 *Arsenic solution, stock.* 1 ml = 100 μg As: Dissolve 0.1320 g of As_2O_3 in 100 ml of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 ml conc. NHO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.3 *Barium solution, stock.* 1 ml = 100 μg Ba: Dissolve 0.1516 g $BaCl_2$ in 10 ml deionized, distilled water with 1 ml (1+1) HCl. Add 10.0 ml (1+1) HCl and dilute to 1,000 ml with deionized, distilled water.

6.3.4 *Beryllium solution, stock.* 1 ml = 100 μg Be: Dissolve 1.127 g $Be_4O(C_2H_3O_2)_6$, beryllium acetate basic, in a minimum amount of conc. acetic acid. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.5 *Boron solution, stock.* 1 ml = 100 μg B: Dissolve 0.5716 g anhydrous H_3BO_3 in deionized, distilled water and dilute to 1,000 ml. Because H_3BO_3 loses weight on drying at 105° C, use a reagent meeting ACS specifications and keep the bottle tightly stoppered to prevent the entrance of atmospheric moisture.

6.3.6 *Cadmium solution, stock.* 1 ml = 100 μg Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.7 *Calcium solution, stock.* 1 ml = 100 μg Ca: Suspend 0.2498 g $CaCO_3$ dried at 180° C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.8 *Chromium solution, stock.* 1 ml = 100 μg Cr: Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete, acidify with 10 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.9 *Cobalt solution, stock.* 1 ml = 100 μg Co: Dissolve 0.1407 g Co_3O_4 in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.10 *Copper solution, stock.* 1 ml = 100 μg Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.11 *Iron solution, stock.* 1 ml = 100 μg Fe: Dissolve 0.1430 g Fe_2O_3 in 10 ml deionized, distilled water with 1 ml (1+1) HCl. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.12 *Lead solution, stock.* 1 ml = 100 μg Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.13 *Lithium solution, stock.* 1 ml = 100 μg Li: Dissolve 0.5323 g Li_2CO_3 slowly in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.14 *Magnesium solution, stock.* 1 ml = 100 μg Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.15 *Manganese solution, stock.* 1 ml = 100 μg Mn: Dissolve 0.5225 g $Mn(NO_3)_2 < 6H_2O$ (do not dry) in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.16 *Molybdenum solution, stock.* 1 ml = 100 μg Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 ml.

6.3.17 *Nickel solution, stock.* 1 ml = 100 μg Ni: Dissolve 0.4953 g $Ni(NO_3)_2 < 6H_2O$ in deionized, distilled water. Add 10 ml of conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.18 *Potassium solution, stock.* 1 ml = 100 μg K: Dissolve 0.1907 g KCl, dried at 110° C, in deionized, distilled water dilute to 1,000 ml.

6.3.19 *Selenium solution, stock.* 1 ml = 100 μg Se: Dissolve 0.1727 g H_2SeO_3 in deionized, distilled water and dilute to 1,000 ml.

6.3.20 *Silica solution, stock.* 1 ml = 100 μg SiO_2: *Do not dry.* Dissolve 0.4730 g $Na_2SiO_3 < 9H_2O$ in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.21 *Silver solution, stock.* 1 ml = 1 μg Ag: Dissolve 0.1575 g $AgNO_3$ in 100 ml of deionized, distilled water and 10 ml conc. HNO_3. Dilute to 1,000 ml with deionized, distilled water.

6.3.22 *Sodium solution, stock.* 1 ml = 100 μg Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.23 *Strontium solution, stock.* 1 ml = 100 μg Sr: Dissolve 0.2416 g $Sr(NO_3)_2$ in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.24 *Vanadium solution, stock.* 1 ml = 100 μg V: Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.25 *Zinc solution, stock.* 1 ml = 100 μg Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.4 *Mixed calibration standard solutions*—Prepared mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. (See 6.4.1 thru 6.4.6) Add 2 ml of (1+1) HNO_3 and 2 ml of (1+1) HCl and dilute to 100 ml with deionized, distilled water. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a TFE fluorocarbon bottle for storage. Fresh mixed standards should be prepared weekly. Some typical combinations follow:

6.4.1 *Mixed standard solution I*—Iron, manganese, cadmium, lead, and zinc.

6.4.2 *Mixed standard solution II*—Beryllium, copper, strontium, vanadium, and cobalt.

6.4.3 *Mixed standard solution III*—Molybdenum, silica, lithium, and barium.

6.4.4 *Mixed standard solution IV*—Calcium, magnesium, sodium, and potassium.

6.4.5 *Mixed standard solution V*—Aluminum, arsenic, boron, chromium, nickel, and selenium.

6.4.6 *Mixed standard solution Vi*—Silver.

6.5 Two types of blanks are required for the analysis. The calibration blank (3.12) is used in establishing the analytical curve while the reagent blank (3.11) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

6.5.1 *The calibration blank* is prepared by diluting 2 ml of (1 + 1) HNO_3 and 2 ml of (1 + 1) HCl to 100 ml with deionized, distilled water. Prepare a sufficient quantity to be used to flush the system between standards and samples.

6.5.2 *The reagent blank* must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

6.6 In addition to the calibration standards, an instrument check standard (3.7) and a reference standard (3.8) are also required for the analyses.

6.6.1 *The instrument check standard* is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. This standard should be included in the analytical scheme with a frequency of 10%.

6.6.2 *The reference standard* should be prepared according to the instructions provided by the supplier. Following initial verification of the calibration standards, analyze weekly.

7. Sample handling and preservation.

7.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether linear polyethylene, polypropylene or TFE-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order. (See Notes 3 and 4).

Note 3.—*Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chromic acid should not be used with plastic bottles.*

Note 4.—If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

7.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter.

7.2.1 For the determination of dissolved elements the sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus is recommended to avoid possible contamination.) Use the first 50–100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

7.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

7.2.3 For the determination of total or total recoverable elements, the sample is acidified with 5 ml conc. HNO_3 per liter (pH 2) as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

8. Sample Preparation.

8.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 8.3.

8.2 For the determinaton of suspended elements, transfer the membrane filter containing the insoluble material to a 250-ml Griffin beaker and add 3 ml conc. HNO_3. Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane. Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 ml of conc. HNO_3. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (DO NOT BAKE), cool, add 2 ml of (1+1) HNO_3 and 2 ml HCl (1+1) per 100 ml dilution and warm the beaker gently to dissolve any soluble material. Wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined. The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended."

8.3 For the determination of total elements, choose a measured, volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 3 ml of conc. HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil. (DO NOT BAKE.) Cool the beaker and add another 3 ml portion of conc. HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 2 ml of 1+1 HNO_3 and 2 ml of 1+1 HCl per 100 ml of final solution and warm the beaker to dissolve any precipitate or residue resulting from evaporation. Wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. Adjust the volume based on the expected concentrations of elements present. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

Note 5.—If low determinations of boron are critical, quartz glassware should be used.

8.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 1 ml of HNO_3 (1+1) and 2 ml of HCl (1+1) to the sample and heat on a steam bath or hot plate until the volume has been reduced to 15–20 ml making certain the sample does not boil. After this treatment the sample is filtered to remove insoluble material that could clog the nebulizer, and the volume adjusted to 100 ml. The sample is then ready for analysis. Concentrations so determined shall be reported as "total."

9. Procedure.

9.1 Set up instrument with proper operating parameters established in Section 5.2. Instrument must be allowed to stabilize for at least 30 min prior to operations.

9.2 Initiate appropriaate operating configuration of computer.

9.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 6.4. Flush the system with the calibration blank (6.5.1) between each standard. (See note 6.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

NOTE 6.—For boron concentrations greater than 500 μg/l extended flush times of 1 to 2 minutes may be required.

9.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 2 percent (or the established control limits). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

9.5 Begin the sample run flushing the system with the calibration blank (6.5.1) between each sample. (See Note 6.) Analyze an instrument check standard (6.6.1) each 10 samples.

9.6 If it has been found that methods of standard addition are required, the following procedure is recommended.

9.6.1 The standard addition technique (13.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive intererence which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s of a standared analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$C_x = \frac{S_B V_s c_s}{(S_A - S_B)\ V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

10. Calculation.

10.1 Reagent blanks (6.5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

10.2 If dilutions were performed, the appropriate factor must be applied to sample values.

10.3 Results should be reported to the nearest μg/l, up to three significant figures, except calcium, magnesium, sodium, and potassium which are reported to the nearest 0.1 mg/l.

11. Quality Control (Instrumental).

11.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow:

11.1.1 Analyze the instrument check standard (6.6.1) made up of all the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ± 2% of the expected values or within the established control limits, the analysis is out of control.

11.1.2 For the purpose of verifying interelement and/or background correction factors, analyze a second check standard, prepared in the following manner. Select a representative sample which contains minimal concentrations of the elements of interest. Spike this sample with the analytes of interest at or near 100 μg/l. (For effluent samples of expected high concentrations, spike at an appropriate level.) Values should fall within the established control levels of 1.5 times the standard deviation of the mean value of the check standard. If not, repeat the standardization.

11.1.3 A reference standard (6.6.2) from an outside source, but having known concentration values, should be analyzed as a blind sample on a weekly frequency. Values should be within the established quality control limits. If not, prepare new stock standards.

12. *Precision and Accuracy.*

12.1 *In an EPA round phase 1 study,* seven laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table II lists the true value, the mean reported value and the mean % relative standard deviation.

Table II.—*ICP Precision and Accuracy Data*

Element	Sample No. 1			Sample No. 2			Sample No. 3		
	True value μg/l	Mean reported value μg/l	Mean percent RSD	True value μg/l	Mean reported value μg/l	Mean percent RSD	True value μg/l	Mean reported value μg/l	Mean percent RSD
Be	750	733	0.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	748	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	80	83	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	584	3.0	20	19	15	180	178	8.0
Al	700	686	5.6	60	62	33	180	181	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	500	512	10	20	20	41	120	109	21
Ni	250	245	5.8	30	28	11	80	55	14
Pb	250	238	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se	40	32	21.9	6	8.5	42	10	8.5	8.3

Not all elements were analyzed by all laboratories.

13. References.

13.1 Winge. R. K.. V. J. Peterson. and V. A. Fassel. "Inductively Coupled Plasma-Optical Emission Spectroscopy: Prominent Lines. EPA-600/4-79-017.

13.2 Winefordner. J. D.. "Trace Analysis: Spectroscopic Methods for Elements." *Chemical Analysis.* Vol. 46. pp. 41-42.

13.3 Handbook for Analytical Quality Control in Water and Wastewater Laboratories. EPA-600/4-79-019.

13.4 Garbarino. J. R. and Taylor. H. E.. "An Inductively-Coupled Plasma Optical Emission Spectrometric Method for Routine Water Quality Testing." Applied Spectroscopy 33. No. 3 (1979).

13.5 "Methods for Chemical Analysis of Water and Wastes." EPA-600/4-79-020.

APPENDIX G

FIELD SAMPLE LOG SHEETS

WESTON DESIGNERS CONSULTANTS

Field Sample Log

Project Norton

Site	Date	Type	Sampler
MW #D	7,9,84	Pumped (O+G) Grab	NR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	0612	0635	0635	

	Before Purge	Other Total	Notes
Water Levels	34.39	56.98	

	Calculated	Other
Purge Volume	3.8 x 3 = 11.4 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	0800	7.3	20.7	659	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0098
Bailer jams in well about 3-4 ft. down.
did not attempt to lower pump 7-12-84 straightened well
So pump would lower properly Sampled 7-13-84

Site	Date	Type	Sampler
MW 9	7 [illegible] 84	[illegible]	NR

	Start Purge	Stop Purge	Sample Taken	Notes
Times	0642		0653	

	Before Purge	Other Total	Notes
Water Levels	38.62	59.87	

	Calculated	Other
Purge Volume	3.5 x 3 = 10.5 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions	1500	[illegible]	20.5	684	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC TOX UOA, O+G, Pb + other metals
#0096

WESTON DESIGNERS CONSULTANTS

Field Sample Log

Project Norton A + B

Site	Date	Type (Pumped)	(O+G / Grab)	Sampler
MW17	7/11/84		O+G	N/B

Times	Start Purge	Stop Purge	Sample Taken	Notes
	0859		0908	

Water Levels	Before Purge	Other ~~total~~	Notes
	40.86	56.16	

Purge Volume	Calculated	Other
	2.7 x 3 = 8.1	

Water Conditions	Time	PH	Temp	Conduct	Notes
		6.65	20.5	782	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0092

Site	Date	Type (Pumped)	(O+G / Grab)	Sampler
MW18	[illegible]	[illegible]	O+G	U/B

Times	Start Purge	Stop Purge	Sample Taken	Notes
	0924	0932		

Water Levels	Before Purge	Other ~~total~~	Notes
	41.59	57.11	

Purge Volume	Calculated	Other
	2.7 x 3 = 8.1	

Water Conditions	Time	PH	Temp	Conduct	Notes
		6.59	20.1	627	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0093

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type		Sampler
MW4	7.9.84	Pumped	O+G / Grab	NIB

Times	Start Purge	Stop Purge	Sample Taken	Notes
	1930	1944	19	

Water Levels	Before Purge	Other ~~total~~	Notes
	32.66	58.93	

Purge Volume	Calculated	Other
	4.4 x 3 = 13.2 gal	

Water Conditions	Time	PH	Temp	Conduct	Notes
		7.21	22.2	514	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	
	X	Yes	No

Notes TOC, TOX, UOA-MEK, O+G, Phenol
Pb + other metals

#0082

Site	Date	Type		Sampler
MW10	7.10.84	Pumped	O+G / Grab	NIB

Times	Start Purge	Stop Purge	Sample Taken	Notes
	2026		2034	

Water Levels	Before Purge	~~Other~~ total	Notes
	18.47	31.26	

Purge Volume	Calculated	Other
	2.2 x 3 = 6.6 gal	

Water Conditions	Time	PH	Temp	Conduct	Notes
		~~6.74~~ 7.01	18.3	525	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken		
		Yes	No	

Notes TOC, TOX, UOA ~~Phenol~~, O+G, Pb + other metals

#0097

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type		Sampler
MW 11	7,10,84	Pumped	O+G Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1920	~~19~~	1927	

	Before Purge	Other	Notes
Water Levels	45.83	Other Total 57.82	

	Calculated	Other
Purge Volume	2.1 x 3 = 6.3 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		6.88	20.2	342	

	Duplicate Sample Number	Cleaning Water Sample Taken	
Special Items	#0063 MW23	Yes	No

Notes TOC, TOX, UOA, O+G, Li Pb + other metals

#0062

Site	Date	Type		Sampler
MW12	7.10 84	Pumped	O+G Grab	N/B

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1741		1749	

	Before Purge	Other	Notes
Water Levels	44.78	Other Total 58.43	

	Calculated	Other
Purge Volume	2 x 3 = 7.2 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		6.77	20.0	251	

	Duplicate Sample Number	Cleaning Water Sample Taken	
Special Items		Yes	No

Notes TOC, TOX, UOA, O+G, (Li, Pb + other metals)

0086

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type	Sampler
MW 2	7.9.84	~~Pumped~~ O+G Grab	NLB

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1050		1113	

	Before Purge	Other ~~Total~~	Notes
Water Levels	41.50	87.46	

	Calculated	Other
Purge Volume	7.6 x 3 = 22.8 gal	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.17	20.8	510	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items				

Notes TOC, TOX, UOA-MEK, O+G, Phenols, Pb + other metals
#0077

Site	Date	Type	Sampler
MW 5	7.9.84	~~Pumped~~ O+G Grab	NLB

	Start Purge	Stop Purge	Sample Taken	Notes
Times	1355	1408		

	Before Purge	Other	Notes
Water Levels	32.37	57.26	

	Calculated	Other
Purge Volume	4 1/2 3 = 12.3	

	Time	PH	Temp	Conduct	Notes
Water Conditions		7.11	20.4	584	

	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No
Special Items	~~#0078~~ #0078		☒	☐

Notes TOC, TOX, UOA, O+G, Pb + other metals
~~#0078~~ #0078

WESTON

Field Sample Log

Project Norton AFB

Site MW #3 Norton AFB	**Date** 7,8,84	**Type** ~~Grab~~ Pumped	O+G Grab	**Sampler** N/B	
Times	**Start Purge** 17:55	**Stop Purge** 18:00 →	**Sample Taken**	**Notes**	
Water Levels	**Before Purge** 18.40	**Other** 27.29 Total Depth		**Notes**	
Purge Volume	**Calculated** 1.5 x 3 = 45 gal.			**Other**	
Water Conditions	**Time** 18:00	**PH** 7.81	**Temp** 31°C	**Conduct** 1480	**Notes**
Special Items	**Duplicate Sample Number** no	**Cleaning Water Sample Taken** Yes	(No)		

Notes TOC, TOX, UOA - MEK, O+G, Phenol
Pb + other metals
#0056

Site Norton MW #1	**Date** 7.8.84	**Type** Pumped	O+G Grab	**Sampler** N/B	
Times	**Start Purge** 1845	**Stop Purge** 18:54 →	**Sample Taken**	**Notes**	
Water Levels	**Before Purge** 25.53	**Other**		**Notes** Total Depth 42.55	
Purge Volume	**Calculated** ~~7.932~~ 3 x 2.9 = 8.7			**Other**	
Water Conditions	**Time**	**PH** 6.81	**Temp** 20.8	**Conduct** 674	**Notes**
Special Items	**Duplicate Sample Number**	**Cleaning Water Sample Taken**	Yes	No	

Notes TOC, TOX, UOA - MEK, O+G, Phenol
Pb + other metals
#0061

WESTON

Field Sample Log

Project Norton AFB

Site MW 6		**Date** 7.19.84	**Type** Pumped / (O+G) Grab		**Sampler** N/R
Times	**Start Purge** 1802	**Stop Purge**	**Sample Taken** 1810	**Notes**	
Water Levels	**Before Purge** 39.75	**Other** Total 52.26		**Notes**	
Purge Volume	**Calculated** 2.2 X 3 = 6.6 gal			**Other**	
Water Conditions	**Time**	**PH** 7.41	**Temp** 228	**Conduct** 714	**Notes**
Special Items	**Duplicate Sample Number** #0079 MW24 merged in UOA		**Cleaning Water Sample Taken** Yes / (No)		

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0080

Site MW 8		**Date** 7.19.84	**Type** Pumped / (O+G) Grab		**Sampler** N/R
Times	**Start Purge** 1855	**Stop Purge** 1908 →	**Sample Taken**	**Notes**	
Water Levels	**Before Purge** 32.87	**Other** Total 57.80		**Notes**	
Purge Volume	**Calculated** 4.1 X 3 = 12.3 gal ~~3 8 X 3 = 11.4 gal~~			**Other**	
Water Conditions	**Time**	**PH** 7.27	**Temp** 22.5	**Conduct** 5700	**Notes**
Special Items	**Duplicate Sample Number**		**Cleaning Water Sample Taken** Yes / No		

Notes TOC, TOX, UOA, O+G, Phenols, Cn
Pb + other metals

WESTON DESIGNERS CONSULTANTS

Field Sample Log

Project North AFS

Site	Date	Type	Sampler
Norton MW #20	7.19.84	Pumped (Grab)	NR

Times	Start Purge 9:39	Stop Purge 9:55	Sample Taken 9:48	Notes
Water Levels	Before Purge 12.30	~~Other~~ Total 28.14		Notes
Purge Volume	Calculated 2.7 x 3 = 8.1			Other

Water Conditions	Time	PH	Temp	Conduct	Notes
		7.24	21.2	464	

Special Items	Duplicate Sample Number ✓	Cleaning Water Sample Taken Yes ~~No~~	No

Notes TOC, TOX, UOA, O+G, Pb + other metals #0075
Nitrogen being sprayed — meter box filled with water

Site	Date	Type	Sampler
MW #21	7.9.84	Pumped (O+G 3.10)	KR

Times	Start Purge 1012	Stop Purge ~~1020~~	Sample Taken 1020	Notes
Water Levels	Before Purge 14.57	~~Other~~ Total 29.25		Notes
Purge Volume	Calculated 2.5 x 3 = 7.5 gal			Other

Water Conditions	Time	PH	Temp	Conduct	Notes
		7.40	21.3	366	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken Yes ☐	No ☐

Notes TOC, TOX, UOA, O+G, Pb + other metals
#0076

WESTON

Field Sample Log

Project Norton AFB

Site	Date	Type (Pumped)	(O+G Grab)	Sampler
MW19	7,11,84		O+G	N/B

Times	Start Purge	Stop Purge	Sample Taken	Notes
	0801		0809	

Water Levels	Before Purge	~~Other~~ total	Notes
	40.87	53.94	

Purge Volume	Calculated	Other
	2.4 x 3 = 7.2 gal	

Water Conditions	Time	PH	Temp	Conduct	Notes
	1500	6.47	20.3	423	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No

Notes TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0094

Site	Date	Type (Pumped)	(O+G Grab)	Sampler
MW22	7 11 84		O+G	N/B

Times	Start Purge	Stop Purge	Sample Taken	Notes
	606		615	

Water Levels	Before Purge	~~Other~~ total	Notes
	22.62	37.71	

Purge Volume	Calculated	Other
	2.7 x 3 = 8.1	

Water Conditions	Time	PH	Temp	Conduct	Notes
	1500	6.53	20.0	802	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken	Yes	No

Notes TOC, TOX, UOA, O+G, Pb + other metals

#0095

WESTON DESIGNERS CONSULTANTS

Field Sample Log

Project _____

Site	Date	Type		Sampler
MW15	/ /	(Pumped)	(O+G Grab)	N/B

Times	Start Purge: 1117	Stop Purge:	Sample Taken: 1120	Notes:
Water Levels	Before Purge: 33.50	~~Other~~ total: 49.26		Notes:
Purge Volume	Calculated: 2.7 x 3 = 8.1 gal			Other:

Water Conditions	Time	PH	Temp	Conduct	Notes
	1500	7.02	21.6	1177	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken: Yes ☐ No ☐

Notes: TOC, TOX, UOA, O+G, Pb

#0089

Site	Date	Type		Sampler
MW16	7/11/89	~~Type~~ (Pumped)	(O+G ~~Grab~~)	N/B

Times	Start Purge: 0831	Stop Purge: 0837	Sample Taken: →	Notes:
Water Levels	Before Purge: 40.15	Other total: 50.65		Notes:
Purge Volume	Calculated: 1.9 x 3 = 5.7 gal			Other:

Water Conditions	Time	PH	Temp	Conduct	Notes
	1500	6.53	21.4	1637	

Special Items	Duplicate Sample Number: #0091 MW25	Cleaning Water Sample Taken: Yes ☐ No ☐

Notes: TOC, TOX, UOA, O+G, Cn, Pb + other metals

#0090

WESTON

Field Sample Log

Project Norton

Site	Date	Type: Pumped / O+G Grab	Sampler
MW13	7/10/84	O+G Grab	N/B

Times	Start Purge: 1828	Stop Purge:	Sample Taken: 1833	Notes:
Water Levels	Before Purge: 48.96	Other: Tot 57.00		Notes:
Purge Volume	Calculated: 1.4 x 3 = 4.2 gal			Other:

Water Conditions	Time	PH	Temp	Conduct	Notes
		6.94	18.2	365	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken: Yes / No

Notes TOC, TOX, UOA, O+G, Pb + other metals

#0087

Site	Date	Type: Pumped / O+G Grab	Sampler
MW 14	7/11/84	O+G Grab	N/R

Times	Start Purge: 1715	Stop Purge:	Sample Taken: 1035	Notes:
Water Levels	Before Purge: 27.12	Other: Tot 66.65		Notes:
Purge Volume	Calculated: 6.6 x 3 = 19.8 gal			Other:

Water Conditions	Time	PH	Temp	Conduct	Notes
	1500	6.75	21.8	1053	

Special Items	Duplicate Sample Number	Cleaning Water Sample Taken: Yes / No

Notes TOC, TOX, UOA, O+G, Pb + other metals

#0088

APPENDIX H

SAMPLE CHAIN-OF-CUSTODY RECORDS

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone:

SHIP TO: Weston tWestch Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sampler

Address

Date Shipped 2-11-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0062-UOA	MW11	2-10-84	UOA	
0063-UOA	MW23			
0086	MW12			
0087	MW13			
0097	MW10	✓		
0088	MW14	2-11-84		
0089	MW15			
0090	MW16			
0091	MW25			
0092	MW17			
0093	MW18			
0094	MW19			
0095	MW22			
0096	MW9			
0099	S-0637			
0100	S-0635			

White · Sampler Canary · Return Copy To Shipper Pink · Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: *(Signature)* Maggie Neckels

Phone: (209) 957-3405

SHIP TO:
Roy F. Weston (Stkn)
7220 Lorraine Ave #102
Stockton Ca 95336

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sample

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
John D. Bognar		
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Receive for laboratory by: *(Signature)*	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	Oil + Grease	
0097	MW 10	7-10-84		
0062	MW 11	7-10-84		
0086	MW 12	7-10-84		
0087	MW 13	7-10-84		
0088	MW 14	7-11-84		
0089	MW 15	7-11-84		
0090	MW 16	7-11-84		

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Nackels

Phone: (209) 957-3405

SHIP TO:

Roy F. Weston
7220 Lorraine Ave # 103
Stockton Ca 95210

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bogner		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	TOC	
0097	MW 10	7-10-84		
2062	MW 11			
0086	MW 12			
0087	MW 13			
0088	MW 14	7-11-84		
0089	MW 15			
2096	MW 9		TOX	
0097	MW 10	7-10-84		
2062	MW 11			
0086	MW 12			
0087	MW 13			
0088	MW 14	7-11-84		
0089	MW 15			
0090	MW 16		TOC	
0092	MW 17			
0090	MW 16		TOX	
0092	MW 17			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) Maggie Neckels
Phone: (209) 957-3405

SHIP TO:
Roy F. Weston
7720 Lorraine Ave #102
Stockton Ca 95210

ATTENTION:
Phone No.

SHIPPING INFORMATION

Site Norton AFB
Shipper Samples
Address
Date Shipped 7-11-84
Shipment Service Fed Exp.
Airbill No.
Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Conditi Upon Receipt
0093	MW18	7-11-84	TOC	
0094	MW19	7-11-84		
0095	MW22	7-11-84		
0063	MW23	7-10-84		
0091	MW25	7-11-84		
0093	MW18	7-11-84	TOX	
0094	MW19	7-11-84		
0095	MW22	7-11-84		
0063	MW23	7-10-84		
0091	MW25	7-11-84		

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F. Weston
2720 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site: Norton AFB

Shipper: Samples

Address:

Date Shipped: 7-11-84

Shipment Service: Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
2081	MW 8	7-9-84	Cyanides	
0090	MW 16	7-11-84		
2092	MW 17			
2093	MW 18			
0094	MW 19			
2091	MW 25			

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F Weston
Weston Way
Weschester PA

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0092	MW17	7-11-84	Disolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0093	MW18	↓	↓	
0094	MW19	↓	↓	
0095	MW22	↓	↓	
0063	MW23	7-10-84	Disolved Metals Li, Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0091	MW25	↓	Disolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F Weston

Weston Way

Weschester PA.

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bagnar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0096	MW 9	7-11-84	Dissolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0097	MW 10	7-10-84	↓	
0062	MW 11	↓	Disolved Metals Li, Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0086	MW 12		↓	
0087	MW 13	↓		
0088	MW 14	7-11-84	Dissolved Metals Pb, Cr, Ni Cd, As, Zn, Cu, Hg	
0089	MW 15	↓	↓	
0090	MW 16	↓		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) Maggie Neckels

Phone: (209) 957-3405

SHIP TO:

Roy F Weston (Stkn)
1720 Lorraine Ave # 102
Stockton Ca 95210

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-11-84

Shipment Service Fed Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D. Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0092	MW17	7-11-84	Oil + Grease	
0093	MW 18	7-11-84		
0094	MW 19	7-11-84		
0095	MW22	7-11-84		
0063	MW 23	7-10-84		
0091	MW 25	7-11-84		

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) John Bognar

Phone: _____

SHIP TO:
Ray F Weston
Weston Way
Westchester PA

ATTENTION: _____

Phone No. _____

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address _____

Date Shipped 7-13-84

Shipment Service Fed Exp.

Airbill No. _____

Cooler No. _____

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John De Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0098	MW7	7-13-84	TOC	
↓	↓	↓	TOX	
			Dissolved Metals Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) John D Bognar

Phone:

SHIP TO:
Roy F Weston
7720 Lorraine Ave #102
Stockton Ca.

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Samples

Address

Date Shipped 7-13-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
John D Bognar		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condit. Upon Receipt
0098	MW7	7-13-84	VOA	
↓	↓	↓	Oil + Grease	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON ENGINEERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone: (209) 957-7405

SHIP TO: Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sample

Address

Date Shipped 2-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075 (x3)	MW20	2-9-84	UOA	
0076	MW21			
0078	MW5			
0080	MW6			
0077	MW2		UOA + MEK	
0081	MW8			
0082	MW4			
0084	S-0630			
0085	S-0625			

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nebb

Phone:

SHIP TO: Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sampler

Address

Date Shipped 7-9-84

Shipment Service Fed. Exp

Airbill No.

Cooler No. 272

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nebb		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0053W (x3)	Pond 1-W	7-6-84	VOA + MEK	
0054	Pond 1-S	↓		
0055	Pond 1-S1			
0056	MW-3	7-8-84		
0057	Pond 2-S	7-2-84		
0058	Pond 3-S			
0059	Pond 2-W			
0060	Pond 3-W			
0061	MW-1	7-8-84		
0071	EB-1	↓		
0072 ①	TB#S-0633	X		
0073 ↓	TB#S-0646			

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nichols

Phone: (209) 957-3405

SHIP TO:

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sampler

Address

Date Shipped 7-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075	MW20	7-9-84	Oil and Grease	
0076	21	"		
0077	2	"		
0078	~~25~~	"		
0079	24	"		
0080	6	"		
0081	8	"		
0082	4	"		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Nechaeb

Phone: (209) 957-3405

SHIP TO:
Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sampler

Address

Date Shipped 7-10-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nechaeb		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper.

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0077-Phenols	MW2	7-9-84	Phenols (preserved H_3PO_4-$CuSO_4$)	
0081-Phenols	MW8		↓	
0082-Phenols	MW4		↓	
0075-TOC	MW20		as lbld	
0075-TOX	↓			
0076-TOC	MW21			
0076-TOX	↓			
0077-TOC	MW2			
0077-TOX	↓			
0078-TOC	MW5			
0078-TOX	↓			
0079-TOC	MW24			
0079-TOX	↓			
0080-TOC	MW6			
0080-TOX	↓			
0081-TOC	MW8			
0081-TOX	↓			
0082-TOC	MW4			
0082-TOX	↓	X		

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON ENGINEERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone: (209) 9573405

SHIP TO:
Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper sampler

Address

Date Shipped 7-10-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0075-Metals	MW20	7-9-84	Dissolved Metals	
0076 Metals	MW21		Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
0077 Metals	MW2			
0078 Metals	MW5			
0079 Metals	MW24			
0080 Metals	MW6			
0081 Metals	MW8			
0082 Metals	MW4			

samples filtered and acidified (HNO_3) in field

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

WESTON DESIGNERS CONSULTANTS

CHAIN OF CUSTODY RECORD

SAMPLERS: (Signature) M. Neebels

Phone:

SHIP TO: Weston - Stockton

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper Sample

Address

Date Shipped 7-9-84

Shipment Service Fed Exp

Airbill No.

Cooler No. 1 of 2

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neebels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0053W	Pond 1-W	7-6-84	Oil + Grease	
0059	Pond 2-W	7-7-84	↓	
0060	Pond 3-W	↓		
0056	MW3	7-8-84		
0061	MW1	↓		
0071	FB1			

White - Sampler Canary - Return Copy To Shipper Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON SCIENCE CONSULTANTS

SAMPLERS: (Signature) M. Neckels

Phone: (209) 957-3405

SHIP TO:

Weston - WestChester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site: Norton AFB

Shipper: Sampler

Address:

Date Shipped: 7-9-84

Shipment Service: Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neckels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0058W-Metals	Pond1-W	7-6-84	Disolved Metals Pb, Ni, Cd, As, Zn, Cr, Cu, Hg	
0058W-Phenols	↓	↓		
0058W-TOC				
0058W-TOX				
0059-Metals	Pond2-W	7-7-84		
0059-Phenols	↓			
0059-TOC				
0059-TOX	↓			
0060-Metals	Pond3-W			
0060-Phenols	↓			
0060-TOC				
0060-TOX	↓	↓		
			metals filtered and acidified (HNO_3) in field	

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Nichols

Phone: (209) 957-3405

SHIP TO:

Weston - Westchester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton AFB

Shipper mgm

Address Weston - Sth.

Date Shipped 7-9-84

Shipment Service Fed. Exp.

Airbill No.

Cooler No. X

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Nichols		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
0054	Pond 1-S	7-6-84	Phenols	
0055	Pond 1-SI	↓	↓	
0057	Pond 2-S	7-7-84		
0058	Pond 3-S	↓		

please return our check to Blue sin to Stockton Off

White - Sampler | Canary - Return Copy To Shipper | Pink - Lab Copy

CHAIN OF CUSTODY RECORD

WESTON DESIGNERS CONSULTANTS

SAMPLERS: (Signature) M. Neckels

Phone: (209) 9573405

SHIP TO: Weston - West Chester

ATTENTION:

Phone No.

SHIPPING INFORMATION

Site Norton

Shipper Sampler

Address

Date Shipped 7-9-84

Shipment Service Fed Exp

Airbill No.

Cooler No.

Relinquished by: (Signature)	Received by: (Signature)	Date/Time
M. Neckels		
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Receive for laboratory by: (Signature)	Date/Time

Analysis laboratory should complete "sample condition upon receipt" section below, sign and return top copy to Shipper

Sample Number	Site Identification	Date Sampled	Analysis Requested	Sample Condition Upon Receipt
2056-Metals	MW#3	7-8-84	Dsolved Pb, Cr, Ni, Cd, As, Zn, Cu, Hg	
2056-TOC	↓	↓		
2056-TOX	↓	↓		
0061-Metals	MW#1	↓		
0061-TOC	↓	↓		
2061-TOX	↓	↓		
0071-Metals	FB-1	↓		
0071-TOC	↓	↓		
0071-TOX	↓	↓		
2056-Phenols	MW#3	↓	(metals filtered and acidified (HNO_3) in field	
2061-Phenols	MW#1	↓		
2071-Phenols	FB-1	↓		

White · Sampler Canary · Return Copy To Shipper Pink · Lab Copy

APPENDIX I

LABORATORY ANALYTICAL METHODS

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Purgeable Halocarbons—Method 601

1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Bromodichloromethane	32101	75-27-4
Bromoform	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	100-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Dichlorodifluoromethane	34668	75-71-8
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethene	34501	75-35-4
trans-1,2-Dichloroethene	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropene	34704	10061-01-5
trans-1,3-Dichloropropene	34699	10061-02-6
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethane	34511	79-00-5
Trichloroethene	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39175	75-01-4

1.2 This is a purge and trap gas chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identification should be supported by at least one additional qualitative

technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse cap with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15-mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated backing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel, 7.7 gm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should

not be heated higher than 180 °C and the remaining sections should not exceed 220 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long × 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long × 0.1 in ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120) mesh or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope. The electrolytic conductivity detector was used to develop the method performance statements and MDL listed in Tables 1 and 2. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25 μL, 0.006 in ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon (Filtrasorb-300 or equivalent (Calgon Corp.)).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials

6.3.1 Coconut charcoal (6/10 mesh sieved to 26 mesh), (Barnaby Chaney, CA-580-26 lot # M-2649 or equivalent).

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on 60/80 mesh Chromosorb-W or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methyl Alcohol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methyl alcohol using assayed liquids or gas cylinders as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methyl alcohol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methyl alcohol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methyl alcohol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methyl alcohol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, in Cincinnati, Ohio.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-µL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the method detection limit (See Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 hours, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after one hour.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (≤10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.4.3 Analyze each calibration standard, according to Section 10, adding 10 µL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1 } RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard.
C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (≤10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in methyl alcohol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 µL of the check sample concentrate to each of a minimum of four 5-mL aliquots of reagent water. A representative waste-

water may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3.1 Calculate upper and lower control limts for method performance:

$$\text{Upper Control Limit (UCL)} = R + 3s$$
$$\text{Lower Control Limit (LCL)} = R - 3s$$

where R and s are calculated as in Section 8.2.3. The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as $R \pm s$. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly.[7]

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 11.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should maintain constant surveillance of both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as above, add a volume to give 7500 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume (15 ng/μL). If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2). Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. USEPA methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.

10. Sample Extraction and Gas Chromatography

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this Table are estimated retention times and method detection limits that can be achieved by this method. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the

syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0 ± .1 minutes at ambient temperature.

10.7 After the 11-minute purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If rapid heating of the trap cannot be achieved, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C.

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for four minutes recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately seven minutes turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool the trap is ready for the next sample.

10.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for the peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of material from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration $\mu g/L = (A_s C_{is})/(A_{is})(RF)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_s = Concentration of the internal standard.

11.2 Report results in micrograms per liter. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

11.3 For samples processed as part of a set where the spiked sample recovery falls outside of the control limits which were established according to Section 8.3, data for the affected parameters must be labeled as suspect.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL up to 1000 × MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000 × MDL.

12.3 In a single laboratory (Monsanto Research), using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 2 were obtained.[9] The standard deviation of the measurement in percent recovery is also included in Table 2.[9]

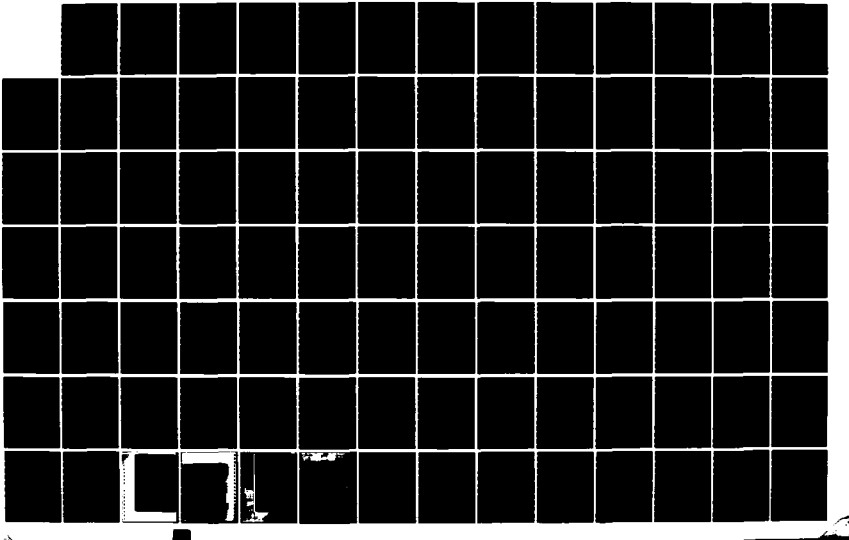
12.4 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

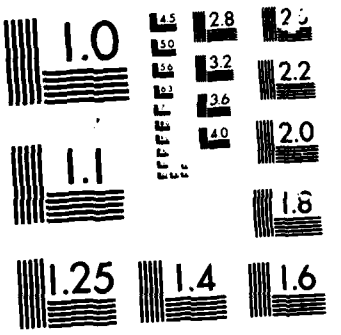
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AD-A165 514 INSTALLATION RESTORATION PROGRAM FINAL REPORT PHASE II 4/5
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MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

Environmental Monitoring and Support Laboratory—Cincinnati, Ohio 45268, March 1979.
9. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (In preparation).

Table 1. Chromatographic Conditions and Method Detection Limits

Parameter	Retention Time (min.) Column 1	Retention Time (min.) Column 2	Method Detection Limit µg/L
Chloromethane	1.50	5.28	0.08
Bromomethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.67	5.28	0.18
Chloroethane	3.33	8.68	0.52
Methylene chloride	5.25	10.1	0.25
Trichlorofluoromethane	7.18	nd	nd
1,1-Dichloroethene	7.93	7.72	0.13
1,1-Dichloroethane	9.30	12.6	0.07
trans-1,2-Dichloroethene	10.1	9.38	0.10
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
trans-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethene	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
cis-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13
Bromoform	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethene	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3-Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15
1,4-Dichlorobenzene	35.4	22.3	0.24

nd = not determined

Column 1 conditions: Carbopack B 60/80 mesh coated with 1% SP-1000 packed in an 8 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45°C for 3 min. then programmed at 8°C/min. to 220° and held for 15 min.

Column 2 conditions: Porasil-C 100/120 mesh coated with n-octane packed in a 6 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50°C for 3 min then programmed at 6°C/min to 170° and held for 4 min.

Table 2. *Single Operator Accuracy and Precision*

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (µg/L)	Number of Analyses	Matrix Types
Bromodichloromethane	100.9	5.0	0.43-46.7	21	3
Bromoform	89.5	9.0	1.45-50	20	3
Bromomethane	105.0	17.3	3.39-49.2	21	3
Carbon tetrachloride	82.5	25.6	0.55-50	19	3
Chlorobenzene	93.9	8.9	2.21-50	20	3
Chloroethane	91.5	22.4	3.95-50	21	3
2-Chloroethylvinyl ether	96.3	9.9	4.39-133	20	3
Chloroform	101.7	20.6	0.44-50	20	3
Chloromethane	91.4	13.4	0.55-23.9	21	3
Dibromochloromethane	98.3	6.5	0.75-93.0	21	3
1,2-Dichlorobenzene	10.20	2.0	4.89-154	21	3
1,3-Dichlorobenzene	91.6	4.3	2.94-46.7	21	3
1,4-Dichlorobenzene	97.5	9.3	2.99-51.6	21	3
Dichlorodifluoromethane	87.8	18.0	2.18-43.4	21	3
1,1-Dichloroethane	102.3	5.5	0.44-46.7	21	3
1,2-Dichloroethane	97.8	4.8	0.44-46.7	21	3
1,1-Dichloroethene	101.1	21.7	0.37-50	19	3
trans-1,2-Dichloroethene	91.0	19.3	0.44-98.0	20	3
1,2-Dichloropropane	97.7	8.8	0.29-39.0	21	3
cis-1,3-Dichloropropene	86.7	6.0	0.44-46.7	21	3
trans-1,3-Dichloropropene	73.5	17.2	0.43-50	20	3
Methylene chloride	97.9	2.6	0.73-46.7	21	3
1,1,2,2-Tetrachloroethane	91.9	15.0	0.46-46.7	21	3
Tetrachloroethene	94.1	18.1	0.50-35.0	21	3
1,1,1-Trichloroethane	75.1	12.5	0.37-29.0	21	3
1,1,2-Trichloroethane	91.0	25.1	0.45-50	21	3
Trichloroethene	106.1	7.4	0.38-46.7	21	3
Trichlorofluoromethane	89.3	13.9	149	14	2
Vinyl chloride	101.9	11.4	0.82-32.3	21	3

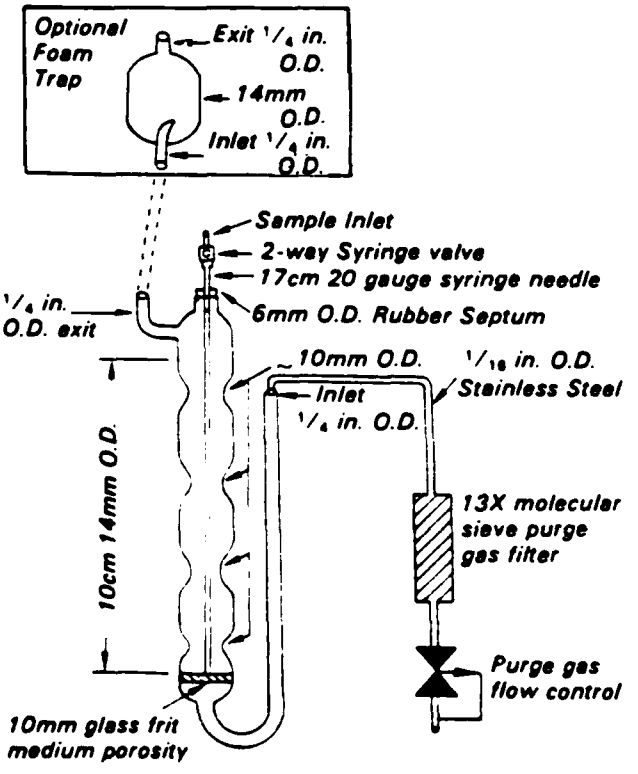


Figure 1. *Purging device*

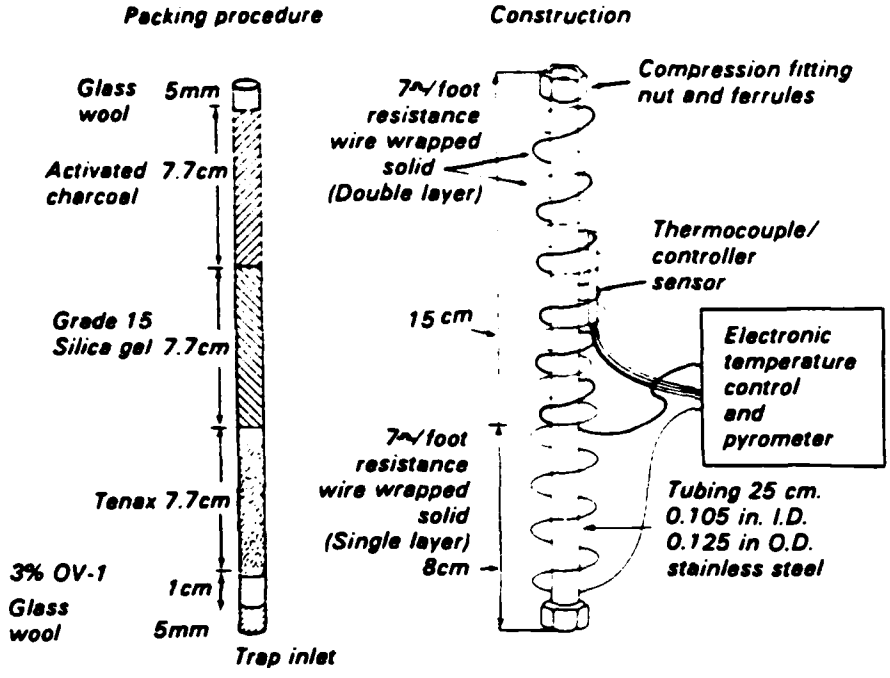


Figure 2. *Trap packings and construction to include desorb capability*

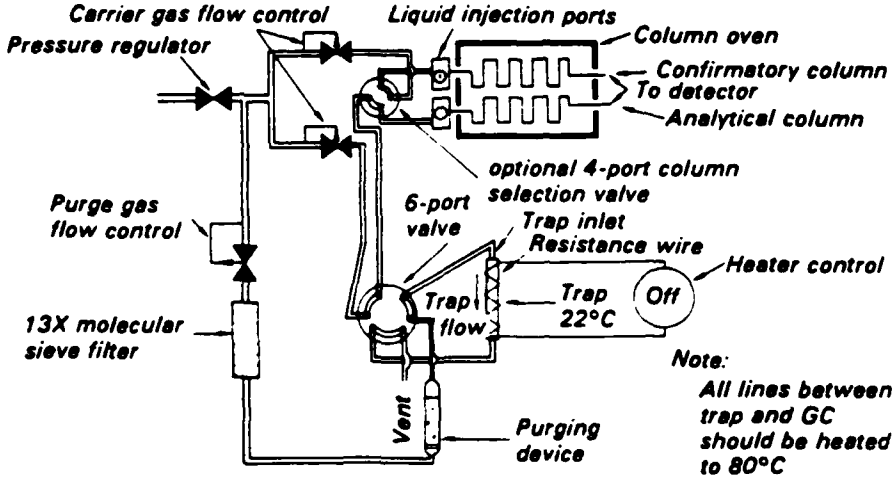


Figure 3. *Schematic of purge and trap device — purge mode*

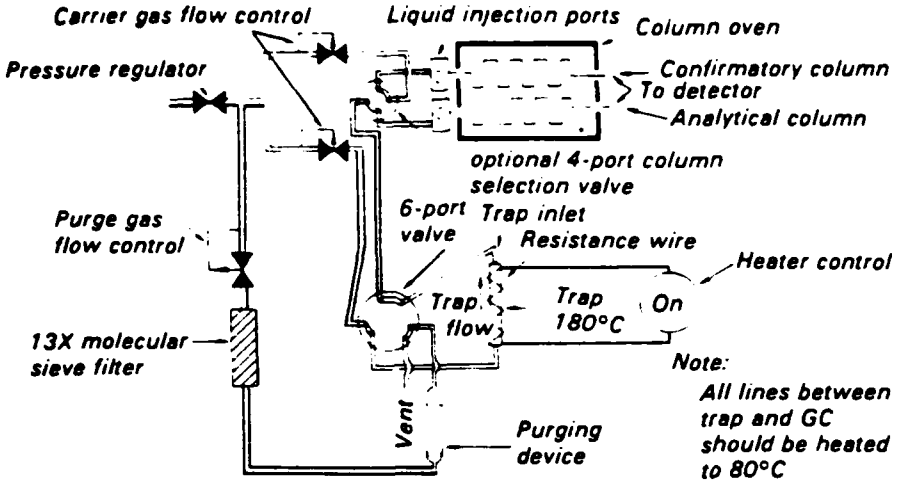


Figure 4. *Schematic of purge and trap device — desorb mode*

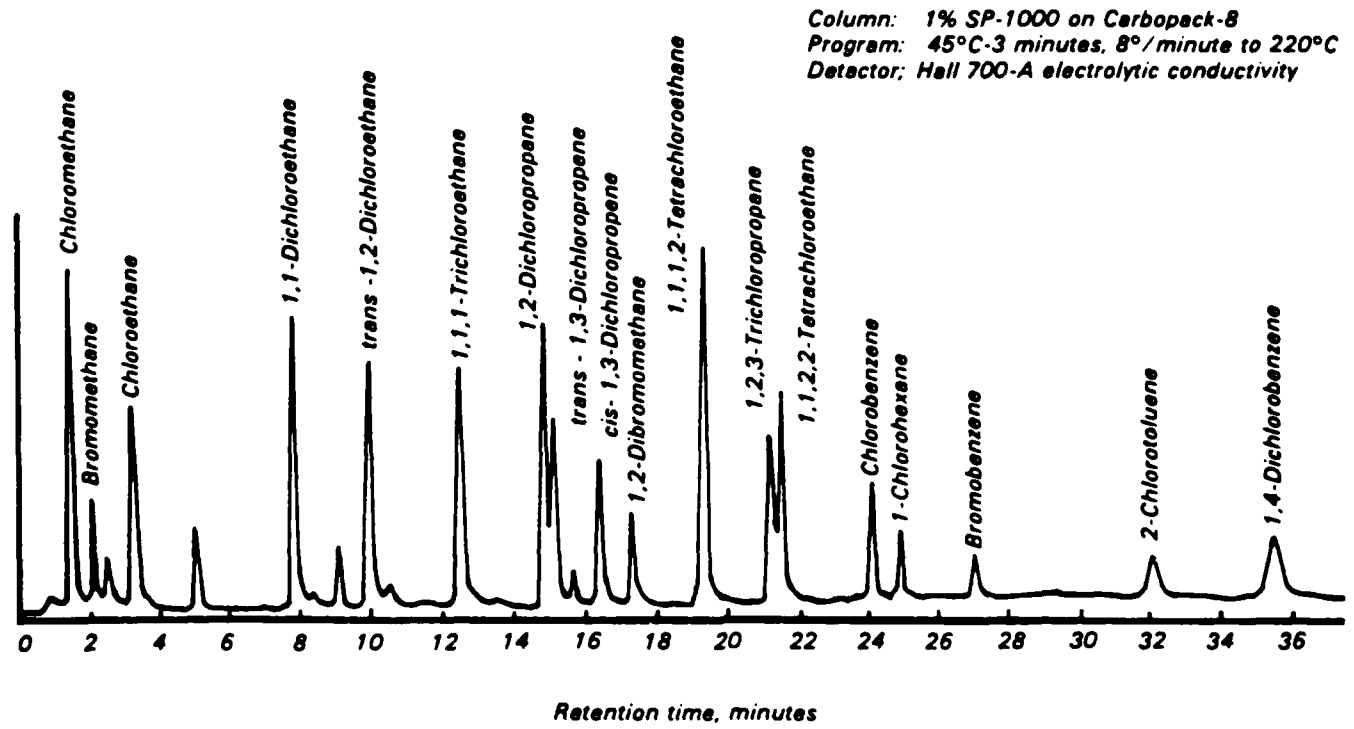


Figure 5. *Gas chromatogram of purgeable halocarbons*

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Purgeable Aromatics—Method 602

1. Scope and Application

1.1 This method covers the determination of various purgeable aromatics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Benzene	34030	71-43-2
Chlorobenzene	34301	108-90-7
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Ethylbenzene	34371	100-41-4
Toluene	34010	108-88-3

1.2 This is a purge and trap gas chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1[1]) for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from these listed depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval for alternate test procedures under 40 CFR 136.4 and 136.5

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The aromatics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After

purging is completed, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected with a photoionization detector[2,3].

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high aromatic levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene and 1,4-dichlorobenzene. Primary standards of these toxic compounds should be prepared in a hood. An NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch.

5.2.2.1 The trap is packed with 1 cm of methyl silicone and 23 cm 2,6-diphenylene oxide polymer as shown in Figure 2. This trap was used to develop the method performance statements in Section 12.

5.2.2.2 Alternatively, either of the two traps described in Method 601 may be used, although water vapor will preclude the measurement of low concentrations of benzene.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3, 4, and 5.

5.3 Gas chromatograph—Analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and stripchart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—6 ft long × 0.082 in ID stainless steel or glass, packed with 5% SP-1200 and 1.75% Bentone-34 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements and the MDLs listed in Tables 1 and 2. Guidelines for the use of alternate column packings are provided in Section 10.1. .

5.3.2 Column 2—8 ft long × 0.1 in ID stainless steel or glass, packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on Chromosorb W-AW (60/80 mesh) or equivalent.

5.3.3 Detector—Photoionization detector (h-nu Systems, Inc. Model PI-51-02 or equivalent). This type of detector has been proven effective in the analysis of wastewaters for the parameters listed in the scope, and was used to develop the performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purge device.

5.5 Micro syringes—25 μL, 0.006 in ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Bottle—15-mL screw-cap with Teflon cap liner.

5.8 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon. (Filtrasorb-300 or equivalent (Calgon Corp.)).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Hydrochloric acid (1 + 1)—Add 50 mL of concentrated HCl to 50 mL of reagent water.

6.4 Trap Materials

6.4.1 2,6-Diphenylene oxide polymer-Tenax, (60/80 mesh) chromatographic grade or equivalent.

6.4.2 Methyl silicone—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.5 Methyl alcohol—Pesticide quality or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methyl alcohol using assayed liquids. Because benzene and 1,4-dichlorobenzene are suspected carcinogens, primary dilutions of these materials should be prepared in a hood.

6.6.1 Place about 9.8 mL of methyl alcohol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.6.2 Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter 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. Commercially prepared stock standards can be used, at any concentration, if they are certified by the manufacturer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 All standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methyl alcohol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary solution standards must be stored with zero headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, in Cincinnati, Ohio.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4.).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (see Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range ($\leq$10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that

is applicable to all samples. The compound, α,α,α-trifluorotoluene, recommended as a surrogate spiking compound in Section 8.7 has been used successfully as an internal standard.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard, according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe as indicated in Section 10.4. Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1} \quad RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard.
C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in methyl alcohol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 μL of the check sample concentrate to each of a minimum of four 5-mL aliquots of reagent water. A representative wastewater may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.3.1 Calculate upper and lower control limits for method performance:

$$\text{Upper Control Limit (UCL)} = R + 3s$$
$$\text{Lower Control Limit (LCL)} = R - 3s$$

where R and s are calculated as in Section 8.2.3

The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly[7].

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results

reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 11.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should maintain constant surveillance of both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and blank with surrogate compounds (e.g. α,α,α-trifluorotoluene). From stock standard solutions prepared as above, add a volume to give 7500 μg of each surrogate to 45 mL of organic-free water contained in a 50-mL volumetric flask, mix and dilute to volume (15 ng/μL). If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2). Dose 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis.

9. Sample Collection, Preservation, and Handling

9.1 The samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottles just prior to shipping to the sampling site. USEPA Methods 330.4 or 330.5 may be used to measure residual chlorine[8]. Field Test Kits are available for this purpose.

9.2 Collect about 500 mL sample in a clean container. Adjust the pH of the sample to about 2 by adding 1 + 1 HCl while stirring gently. Fill the sample bottle in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Sample Extraction and Gas Chromatography

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and method detection limits that can be achieved by this method. An example of the separations achieved by Column 1 is shown in Figure 6. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow sample to come to ambient temperature prior to introducing it into the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (Section 8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 12.0 ± 0.1 minutes at ambient temperature.

10.7 After the 12-minute purge time, disconnect the purge chamber from the trap. Dry the trap by maintaining a flow of 40 mL/min of dry purge gas through it for six minutes. See Figure 4. A dry purger should be inserted into the device to minimize moisture in the gas. Attach the trap to the chromatograph, adjust the device to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If rapid heating cannot be achieved, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry and random retention time problems persist) instead of the initial program temperature of 50 °C.

10.8 While the trap is being desorbed onto the GC column, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

10.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for the peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of material from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration $\mu g/L = (A_s C_{is})/(A_{is})(RF)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard.

11.2 Report results in micrograms per liter. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

11.3 For samples processed as part of a set where the spiked sample recovery falls outside of the control limits which were described in Section 8.3, data for the affected parameters must be labeled as suspect.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero[1]. The MDL concentrations listed in Table 1 were obtained using reagent water[9]. Similar results were achieved using representative wastewaters.

12.2 This method has been demonstrated to be applicable for the concentration range from the MDL up to 1000 × MDL[9]. Direct aqueous injection techniques should be used to measure concentration levels above 1000 × MDL.

12.3 In a single laboratory (Monsanto Research), using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 2 were obtained[9]. The standard deviation of the measurement in percent recovery is also included in Table 2.

12.4 The Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1 See Appendix A

2. Bellar, T.A., and Lichtenberg, J.J. *Journal American Water Works Association, 66,* 739, (1974).

3. Bellar, T.A., and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compunds," Proceedings of Symposium on Measurement of Organic Pollutants in Water and Wastewater. American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.

4. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Safety, 3rd Edition, 1979.

7. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.

8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.

9. "EPA Method Validation Study 24, Method 602 (Purgeable Aromatics)," Report for EPA Contract 68-03-2856 (In preparation).

Table 1. *Chromatographic Conditions and Method Detection Limits*

Parameter	Retention Time (min.) Column 1	Retention Time (min.) Column 2	Method Detection Limit µg/L
Benzene	3.33	2.75	0.2
Toluene	5.75	4.25	0.2
Ethylbenzene	8.25	6.25	0.2
Chlorobenzene	9.17	8.02	0.2
1,4-Dichlorobenzene	16.8	16.2	0.3
1,3-Dichlorobenzene	18.2	15.0	0.4
1,2-Dichlorobenzene	25.9	19.4	0.4

Column 1 conditions: Supelcoport 100/120 mesh coated with 5% SP-1200 and 1.75% Bentone-34 packed in a 6 ft. × 0.085 in ID stainless steel column with helium carrier gas at 36 cc/min flow rate. Column temperature held at 50°C for 2 min. then programmed at 6°C/min to 90°C for a final hold.

Column 2 conditions: Chromosorb W-AW 60/80 mesh coated with 5% 1,2,3-Tris(2-cyanoethyoxy)propane packed in a 6 ft. × 0.085 in ID stainless steel column with helium carrier gas at 30 cc/min flow rate. Column temperature held at 40°C for 2 min then programmed at 2°C/min to 100°C for a final hold.

Table 2. *Single Operator Accuracy and Precision*

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (µg/L)	Number of Analyses	Matrix Types
Benzene	91	10.0	0.5-9.7	21	3
Chlorobenzene	97	9.4	0.5-100	21	3
1,2-Dichlorobenzene	104	27.7	0.5-10.0	21	3
1,3-Dichlorobenzene	97	20.0	0.5-4.8	21	3
1,4-Dichlorobenzene	120	20.4	0.5-10.0	21	3
Ethylbenzene	98	12.4	0.5-9.9	21	3
Toluene	77	12.1	0.5-100	21	3

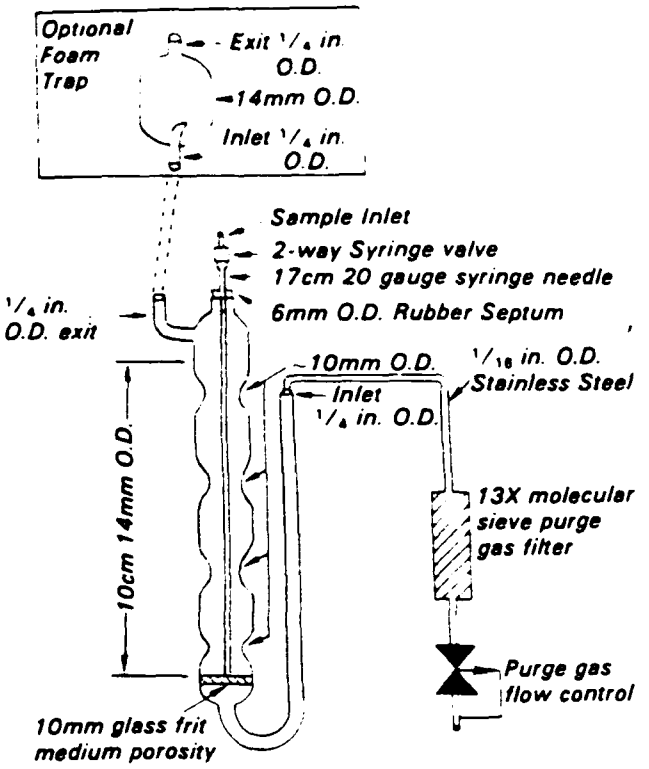


Figure 1. *Purging device*

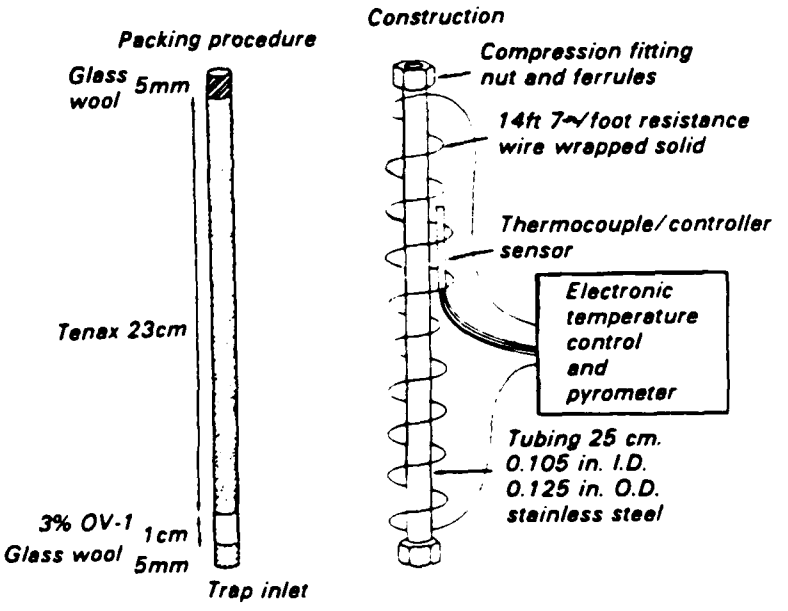


Figure 2. *Trap packings and construction to include desorb capability*

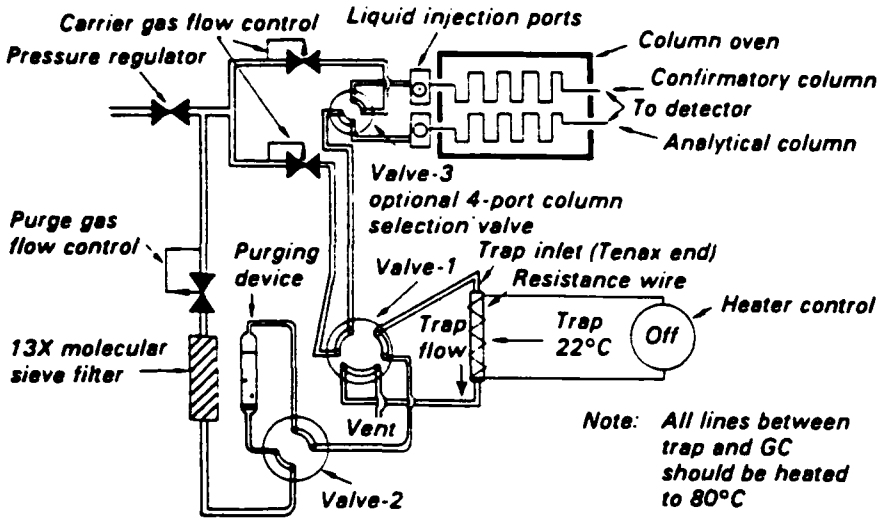


Figure 3. *Purge-trap system (Purge-sorb Mode)*

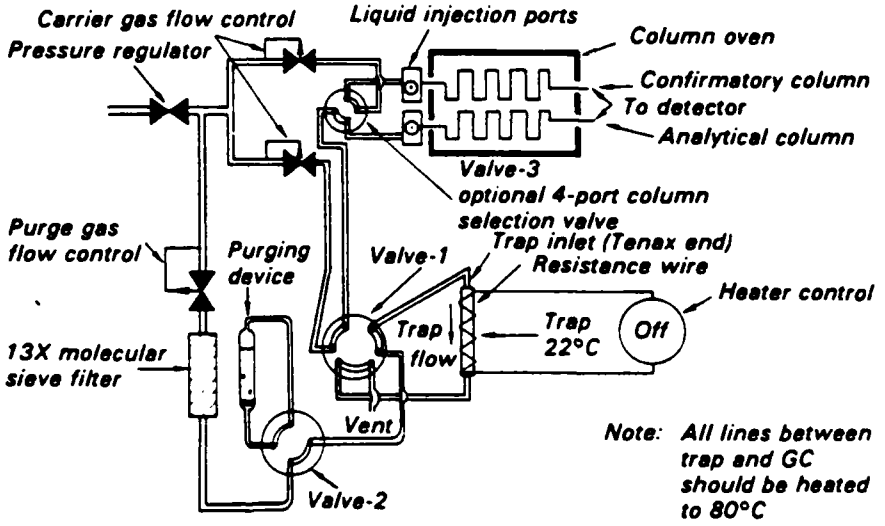


Figure 4. *Purge-trap system (Trap-dry Mode).*

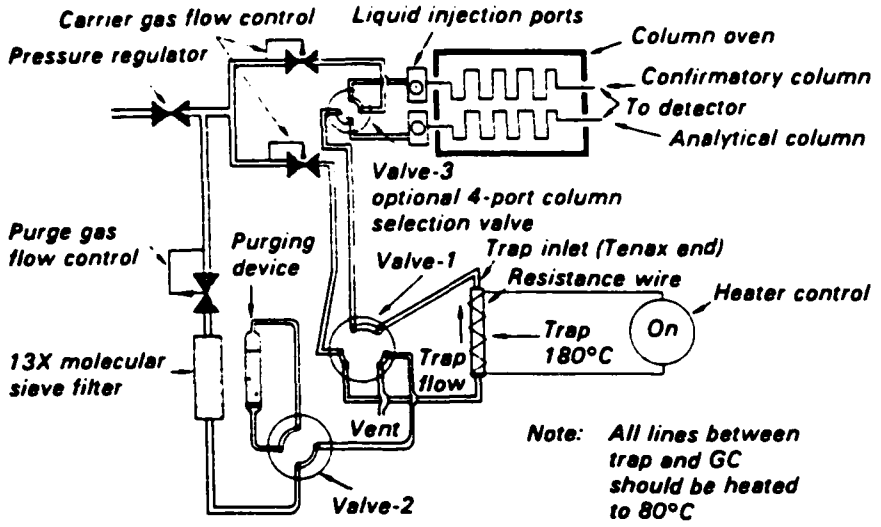


Figure 5. *Purge-trap system (Desorb Mode).*

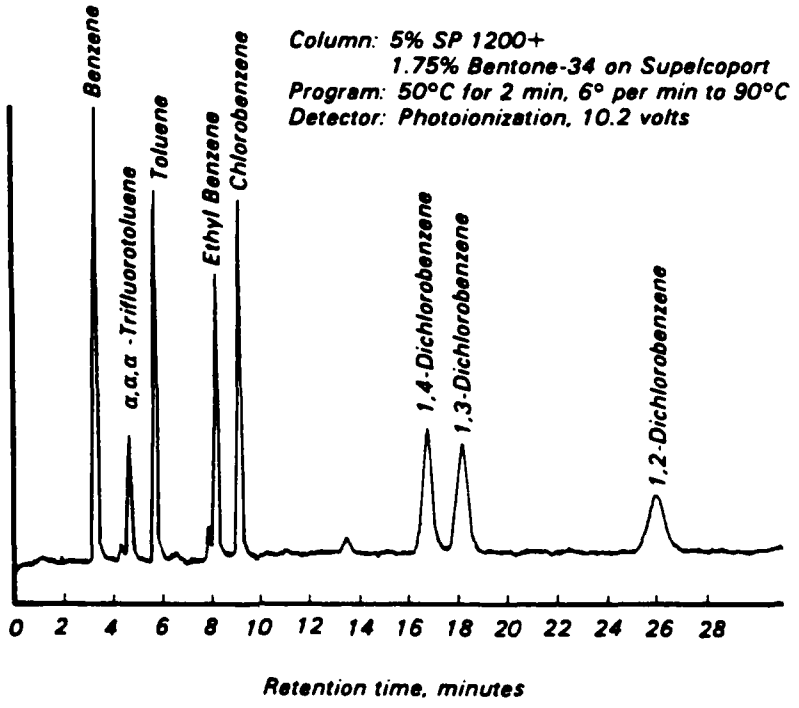


Figure 6. *Gas chromatogram of purgeable aromatics.*

METHOD 5020

HEADSPACE METHOD

1.0 Scope and Application

1.1 Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. It is a simple method that allows large numbers of samples to be analyzed in a relatively short period of time. Because of the large variability and complicated matrices of waste samples in the solid and paste forms, detection limits for this method may vary widely among samples. The method works best for compounds with boiling points of less than 125° C. The sensitivity of this method will depend on the equilibria of the various compounds between the vapor and dissolved phases.

1.2 This method is recommended for use by, or under the supervision of, analysts experienced in the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Summary of Method

The sample is collected in sealed glass containers and allowed to equilibrate at 90° C. A sample of the headspace gas is withdrawn with a gas-tight syringe for analysis by the appropriate gas chromatographic method (8010, 8015, 8020, or 8030).

3.0 Interferences

Refer to Methods 8010, 8015, 8020, or 8030.

4.0 Apparatus and Materials

4.1 Gas-tight syringe: 5-cc with chromatographic needles.

4.2 Headspace standard solutions: Prepare according to procedures in 8010, 8015, 8020, or 8030 at 50 ng/µl and 250 ng/µl concentrations.

4.3 Vials: 125-ml Hypo-Vials (Pierce Chemical Co., #12995, or equivalent).

4.4 Septa: Tuf-Bond (Pierce #12720, or equivalent).

4.5 Seals: Aluminum (Pierce #13214, or equivalent).

4.6 Crimper: Hand (Pierce #13212, or equivalent).

5.0 Reagents

5.1 Refer to Methods 8010, 8015, 8020, or 8030.

6.0 Sample Collection, Preservation, and Handling

6.1 Refer to Methods 8010, 8015, 8020, or 8030.

7.0 Procedure

7.1 Place 10.0 g each of the well-mixed waste sample into three separate 125-ml septum seal vials.

7.2 Dose one sample vial through the septum with 200 µl of a 50-ng/µl methanolic standard of the compounds of interest. Label this "1-ppm spike."

7.3 Dose a separate (empty) 125-ml septum seal vial with 200 µl of the 50 ng/µl standard methanol solution. Label this "1-ppm standard."

7.4 Place the sample, 1-ppm-spike, and 1-ppm-standard vials into a 90° C water bath for 1 hr. Store the remaining sample vial at 4.0° C for possible future analysis.

7.5 While maintaining the vials at 90° C, withdraw 2 ml of the headspace gas with a gas-tight syringe and analyze by injecting into a GC, operating under the appropriate conditions for the GC measurement method being used (8010, 8015, 8020, or 8030).

7.6 Analyze the 1-ppm standard and adjust instrument sensitivity to give a minimum response of at least 2x the background. Record retention times (RT) and peak areas of compounds of interest.

7.7 Analyze the 1-ppm spiked sample in the same manner. Record RT's and peak areas.

7.8 Analyze the undosed sample as in Section 7.7.

8.0 Quality Control

8.1 Before processing any samples, the analyst should demonstrate through the analysis of a distilled water method blank that all glassware and reagents are interference-free. Each time a set of samples is extracted or there is a change in reagents, a method blank should be processed as a

safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified samples do not indicate sufficient sensitivity to detect less than or equal to 1 µg/g of sample, then the sensitivity of the instrument should be increased. Where doubt exists over the identification of a peak on the chromatograph, confirmatory techniques such as mass spectroscopy should be used.

9.0 References

1. Hachenberg, H. and Schmidt, A. 1979. Gas chromatographic headspace analysis. Philadelphia: Hayden & Sons Inc.

2. Friant, S.L. and Suffet, I.H. 1979. Interactive effects of temperature, salt concentration, and pH on headspace analysis for isolating volatile trace organics in aqueous environmental samples. Anal. Chem. 51:2167-2172.

METHOD 8240

GC/MS METHOD FOR VOLATILE ORGANICS

1.0 Scope and Application

1.1 Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 The detection limit of Method 8240 for an individual compound is approximately 1 µg/g (wet weight) in waste samples. For samples containing more than 1 mg/g of total volatile material, the detection limit is proportionately higher.

1.3 Method 8240 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by or under the supervision of analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 Summary of Method

2.1 The volatile compounds are introduced to the gas chromatograph by direct injection, the Headspace Method (Method 5020), or the Purge-and-Trap Method (Method 5030). Method 5030 should be used for groundwater analysis. The components are separated via the gas chromatograph and detected using a mass spectrometer which is used to provide both qualitative and quantitative information. The chromatographic conditions as well as typical mass spectrometer operating parameters are given.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample can be dispersed in methanol or polyethylene glycol (PEG) to dissolve the volatile organic constituents. A portion of the methanolic or PEG solution is combined with water in a specially designed purging chamber. An inert gas is then bubbled through the solution at ambient temperature and the volatile comnponents are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

2.3 An aliquot of each sample must be spiked with an appropriate standard to determine percent recovery and detection limits for that sample.

2.4 Table 1 lists detection limits that can be obtained in wastewaters in the absence of interferences. Detection limits for a typical waste sample would be significantly higher.

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min) Column 1[a]	Method detection limit (µg/l)
Chloromethane	2.3	ND
Bromomethane	3.1	ND
Vinyl chloride	3.8	ND
Chloroethane	4.6	ND
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	ND
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2-Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloropropane	15.7	6.0
trans-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
cis-1,3-Dichloropropene	17.2	ND
2-Chloroethylvinyl ether	18.6	ND
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1
Toluene	23.5	6.0
Chlorobenzene	24.6	6.0
Ethyl benzene	26.4	7.2
1,3-Dichlorobenzene	33.9	ND
1,2-Dichlorobenzene	35.0	ND
1,4-Dichlorobenzene	35.4	ND

ND = not determined.

[a]Column conditions: Carbopack B (60/80 mesh) coated with 1% SP-1000 packed in a 6-ft by 2-mm I.D. glass column with helium carrier gas at a flow rate of 30 ml/min. Column temperature is isothermal at 45° C for 3 min, then programmed at 8° C per minute to 220° and held for 15 min.

3.0 Interferences

3.1 Interferences coextracted from the samples will vary considerably from source to source, depending upon the particular waste or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences under the conditions of the analysis by running method blanks. Method blanks are run by analyzing organic-free water in the normal manner. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride) through the septum seal into the sample during shipment and storage. A field blank prepared from organic-free water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Cross contamination can occur whenever high-level and low-level samples are sequentially analyzed. To reduce cross contamination, the purging device and sample syringe should be rinsed out twice, between samples, with organic-free water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of organic-free water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high organohalide levels, it may be necessary to wash out the purging device with a soap solution, rinse with distilled water, and then dry in a 105° C oven between analyses.

3.4 Low molecular weight impurities in PEG can be volatilized during the purging procedure. Thus, the PEG employed in this method must be purified before use as described in Section 5.2.

4.0 Apparatus and Materials

4.1 Sampling equipment

4.1.1 Vial: 25-ml capacity or larger, equipped with a screw cap (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry for 1 hr at 105° C before use.

4.1.2 Septum: Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water and dry at 105° C for 1 hr before use.

4.2 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the purging chamber, trap, and the desorber. Several complete devices are now commercially available.

4.2.1 The purging chamber must be designed to accept 5-ml or 25-ml samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging chamber, illustrated in Figure 1, meets these design criteria.

4.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 2.5 mm. The trap must be packed to contain the following minimum lengths-of-adsorbents: 1.0 cm of methyl-silicone-coated packing (Section 5.3.2), 15 cm of 2,6-diphenylene oxide polymer (Section 5.3.1), and 8 cm of silica gel (Section 5.3.3). The minimum specifications for the trap are illustrated in Figure 2.

4.2.3 The desorber must be capable of rapidly heating the trap to 180° C within 30 sec. The polymer section of the trap should not be heated higher than 180° C and the remaining sections should not exceed 220° C. The desorber design, illustrated in Figure 2, meets these criteria.

4.2.4 The purge-and-trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

4.3 Gas chromatograph/mass spectrometer system

4.3.1 Gas chromatograph: An analytical system complete with a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

4.3.2 Column: 2-m x 2-mm I.D. stainless steel or glass, packed with 1% SP-1000 on 60/80 mesh Carbopack B or equivalent.

4.3.3 Mass spectrometer: Capable of scanning from 40 to 250 amu every 3 sec or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria in Table 1 when 50 ng of 4-bromofluoro-benzene (BFB) is injected through the GC inlet or introduced in the purge-and-trap mode.

4.3.4 GC/MS interface: Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria (see Section 9) may be used. GC-to-MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane. The interface must be capable of transporting at least 10 ng of the components of interest from the GC to the MS.

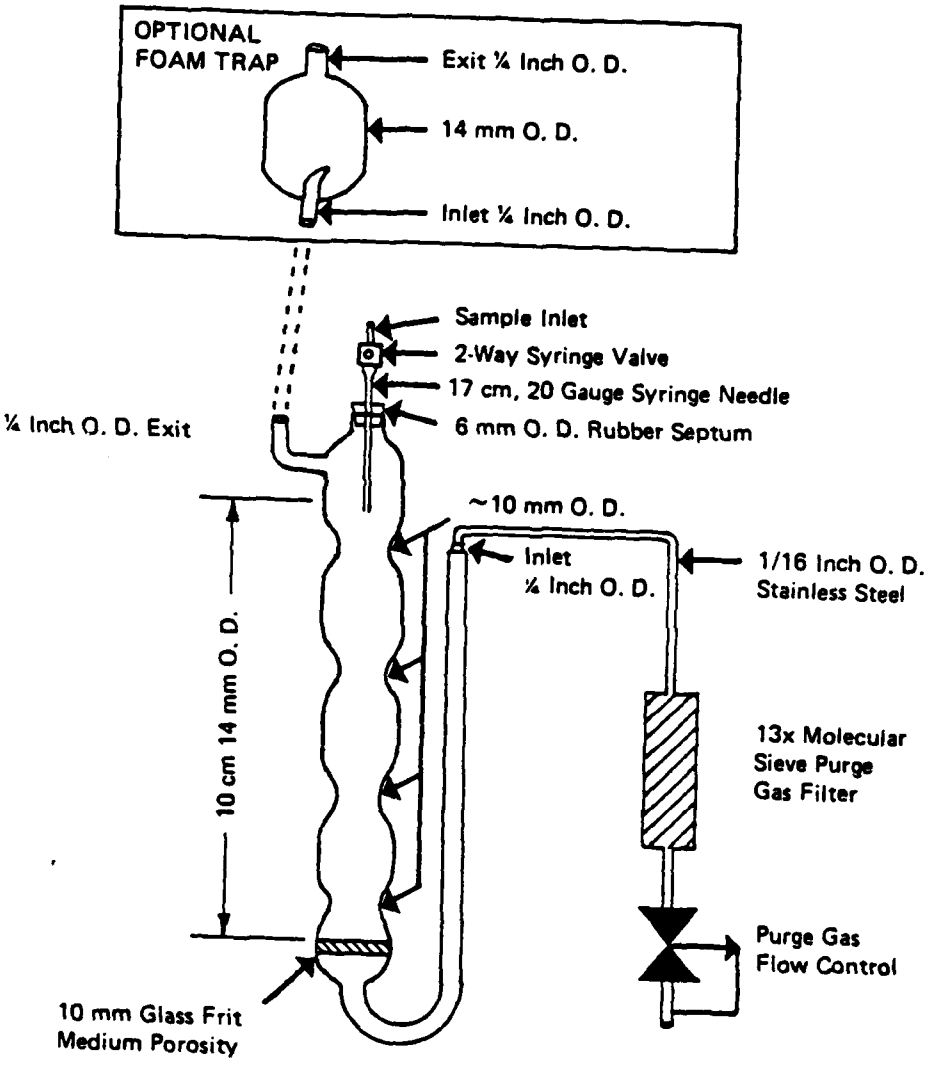


Figure 1. Purging chamber.

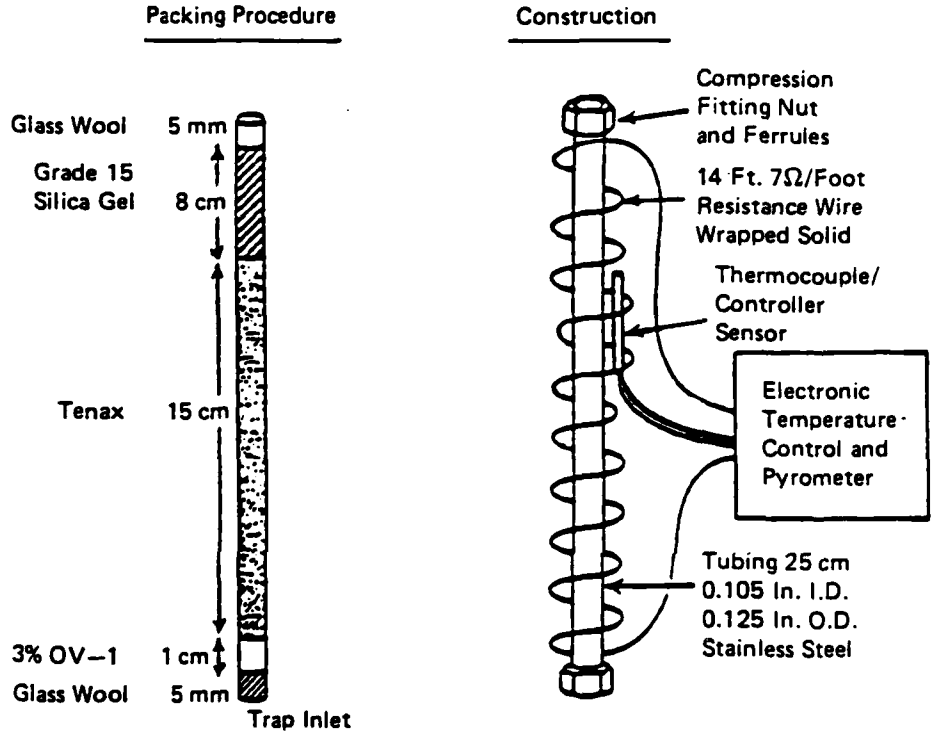


Figure 2. Trap packings and construction to include desorb capability.

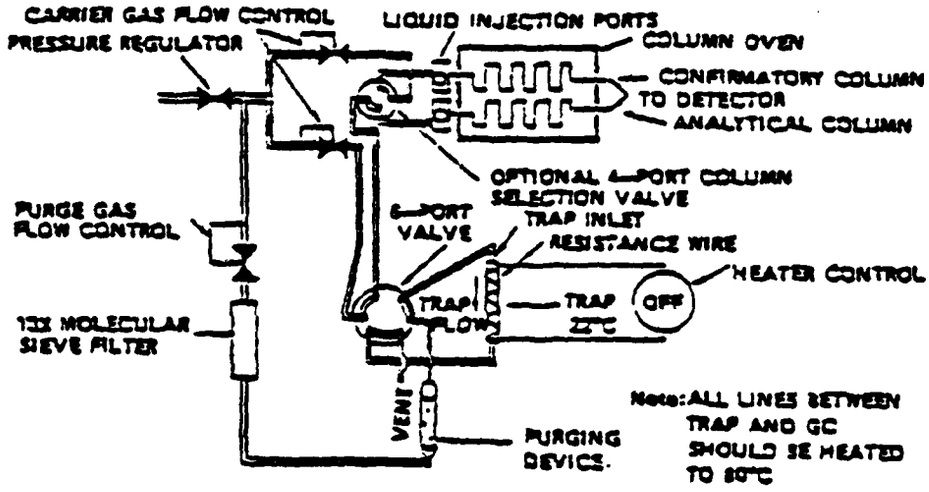


FIGURE 1. Schematic of purge and trap device – purge mode

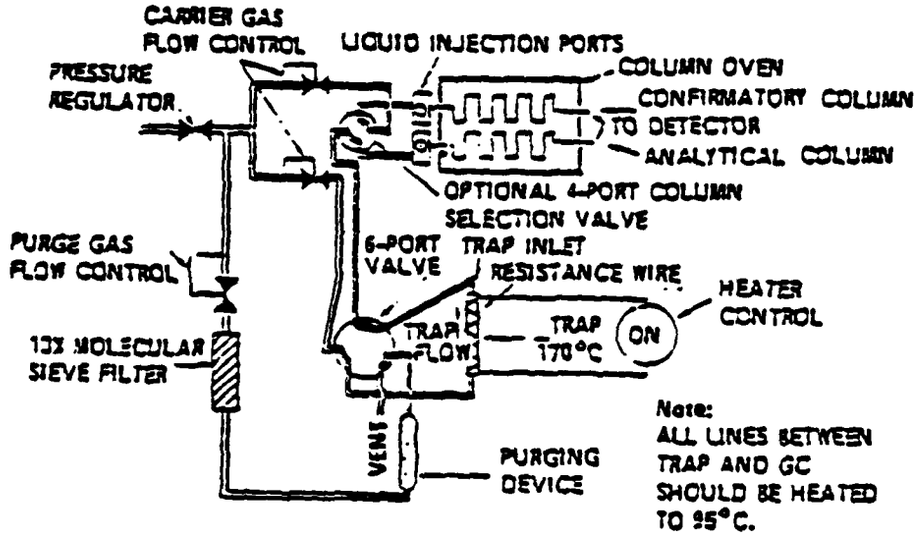


Figure 4. Schematic of purge and trap device - desorb mode

4.3.5 Data system: A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Hardware and software must be available to transform the data into a compatible format. These generally consist of a 9-inch, 800-bpi tape drive and the associated software.

4.4 Sample transfer implements: Implements are required to transfer portions of solid, semisolid, and liquid wastes from sample containers to laboratory glassware. The transfer must be accomplished rapidly to avoid loss of volatile components during the transfer step. Liquids may be transferred using a hypodermic syringe with a wide-bore needle or no needle attached. Samples should be introduced into the syringe by (1) removing the plunger from the syringe, (2) pouring the sample into the barrel, and (3) replacing the barrel and inverting the syringe to remove any air trapped in the syringe. Do not draw the sample up into the syringe. Solids may be transferred using a conventional laboratory spatula, spoon, or coring device. A coring device that is suitable for handling some samples can be made by using a glass tubing saw to cut away the closed end of the barrel of a glass hypodermic syringe.

TABLE 2. BFB KEY ION ABUNDANCE CRITERIA

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 100% of mass 174
177	5 to 9% of mass 176

4.5 Syringes: 5-ml and 25-ml glass hypodermic, equipped with 20-gauge needle, at least 15 cm in length.

4.6 Micro syringes: 10-µl, 25-µl, 100-µl, 250-µl, and 1000-µl. These syringes should be equipped with 20-gauge needles having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device (see Figure 1). The needle length required will depend upon the dimensions of the purging device employed.

4.7 Centrifuge tubes: 50-ml round-bottom glass centrifuge tubes with Teflon-lined screw caps. The tubes must be marked before use to show an approximate 20-ml graduation.

4.8 Centrifuge: Capable of accommodating 50-ml glass tubes.

4.9 Syringe valve: 2-way, with Luer ends (2 each) (Hamilton #86725 valve equipped with one Hamilton #35033 Luer fitting, or equivalent).

4.10 Syringe: 5-ml, gas-tight with shut-off valve.

4.11 Bottle: 15-ml, screw-cap, Teflon cap liner.

4.12 Balance: Analytical, capable of accurately weighing 0.0001 g.

4.13 Rotary evaporator: equipped with Teflon-coated seals (Buchi Rotavapor R-110, or equivalent).

4.14 Vacuum pump: mechanical, two-stage.

5.0 Reagents

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 500 g of activated carbon (Calgon Corp., Filtrasorb-300, or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90° C, bubble a contaminant-free inert gas through the water for 1 hr. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

5.1.4 Reagent water may also be purchased under the name "HPLC water" from several manufacturers (Burdick and Jackson, Baker and Waters, Inc.).

5.2 Reagent PEG: Reagent PEG is defined as PEG having a nominal average molecular weight of 400, and in which interferents are not observed at the method detection limit for compounds of interest.

5.2.1 Reagent PEG is prepared by purification of commercial PEG having a nominal average molecular weight of 400. The PEG is placed in a round-bottom flask equipped with a standard taper joint, and the flask is affixed to a rotary evaporator. The flask is immersed in a water bath at 90-100° C and vacuum is maintained at less than 10 mm Hg for at least 1 hr using a two-stage mechanical pump. The vacuum system is equipped with an all-glass trap, which is maintained in a dry ice/methanol bath.

5.2.2 In order to demonstrate that all interfering volatiles have been removed from the PEG, a reagent water/PEG blank must be analyzed.

5.3 Trap materials

5.3.1 2,6-Diphenylene oxide polymer: 60/80-mesh Tenax, chromatographic grade or equivalent.

5.3.2 Methyl silicone packing: 3 percent OV-1 on 60/80 mesh Chromosorb-W or equivalent.

5.3.3 Silica gel, Davison Chemical (35/60 mesh), grade-15 or equivalent.

5.3.4 Prepared trapping columns may be purchased from several chromatography suppliers.

5.4 Methanol: Distilled-in-glass quality or equivalent.

5.5 Calibration standards; stock solutions (2 mg/ml): Stock solutions of calibration standards may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions of individual compounds in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA-approved toxic gas respirator should be worn by analysts when handling high concentrations of these materials.

5.5.1 Place about 9.8 ml of methanol in 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. Weigh the flask to the nearest 0.1 mg.

5.5.2 Add the assayed reference material as described below.

5.5.2.1 Liquids: Using a 100-µl syringe, immediately add 2 drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.5.2.2 Gases: To prepare standards for any compounds that boil below 30° C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-ml valved gas-tight syringe with a reference standard to the 5.0-ml mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol.

5.5.3 Reweigh, dilute to volume, stopper, then mix by gently inverting the flask several times. Calculate the concentration in µg/µl per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10 to -20° C and protect from light.

5.5.5 Prepare fresh standards weekly for gases or for reactive compounds such as 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

5.6 Calibration standards; secondary dilution solutions: Using stock solutions described in Section 5.5, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the methanol or aqueous PEG calibration solutions prepared as described in Section 6.3.2 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards from them.

5.7 Surrogate standards: Surrogate standards may be added to samples and calibration solutions to assess the effect of the sample matrix on recovery efficiency. The compounds employed for this purpose are 1,2-dibromotetrafluoroethane, bis(perfluoroisopropyl) ketone, fluorobenzene, and m-bromobenzotrifluoride. Prepare methanolic solutions of the surrogate standards using the procedures described in Sections 5.5 and 5.6. The

concentrations prepared and the amount of solution added to each sample should be those required to give an amount of each surrogate in the purging device that is equal to the amount of each internal standard added, assuming a 100% recovery of the surrogate standards.

5.8 Internal standards: In this method, internal standards are employed during analysis of all samples and during all calibration procedures. The analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. However, for general use, D_4-1,2-dichloroethane, D_6-benzene, and D_5-ethylbenzene are recommended as internal standards covering a wide boiling point range.

5.9 4-Bromofluorobenzene (BFB): BFB is added to the internal standard solution or analyzed alone to permit the mass spectrometer tuning for each GC/MS run to be checked.

5.10 Internal standard solution: Using the procedures described in Sections 5.5 and 5.6, prepare a methanolic solution containing each internal standard at a concentration of 12.5 µg/ml.

5.11 Sodium monohydrogen phosphate: 2.0 µ in distilled water.

5.12 n-Nonane and n-dodecane, 98+% purity.

5.13 N-Hexadecane, distilled-in-glass (Burdick and Jackson, or equivalent).

6.0 Sample Collection, Handling, and Preservation

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All samples must be stored in Teflon-lined screw cap vials. Sample containers should be filled as completely as possible so as to minimize headspace or void space. Vials containing liquid sample should be stored in an inverted position.

6.3 All samples must be iced or refrigerated from the time of collection to the time of analysis, and should be protected from light.

7.0 Procedure

7.1 Calibration

7.1.1 Assemble a purge-and-trap device that meets the specifications in Section 4.2 and connect the device to a GC/MS system. Condition the trap overnight at 180° C by backflushing with an inert gas flow of at least 20 ml/min. Prior to use, condition the trap daily for 10 min while backflushing at 180° C.

7.1.2 Operate the gas chromatograph using the conditions described in Section 7.3.5 and operate the mass spectrometer using the conditions described in Section 7.3.2.

7.1.3 Calibration procedure

7.1.3.1 Conduct calibration procedures using a minimum of three concentration levels for each calibration standard. One of the concentration levels should be at a concentration near but above the method detection limit. The remaining two concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

7.1.3.2 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. To the purging device, add 5.0 ml of reagent water or reagent water/PEG solution. This solution is prepared by taking 4.0 ml of reagent water or reagent PEG and diluting to 100 ml with reagent water. The reagent water/PEG solution is added to the purging device using a 5-ml glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of a 20-gauge needle. Next, using a 10-µl or 25-µl micro-syringe equipped with a long needle (see Section 4.6), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards (see Section 5.6). Add the aliquot of calibration solution directly to the reagent water or reagent water/PEG solution in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe be sure that the end of the syringe needle is well beneath the surface of the reagent water or water/PEG solution. Similarly, add 20 µl of the internal standard solution (see Section 5.10). Close the 2-way syringe valve at the sample inlet.

7.1.3.3 Carry out the purge and analysis procedure as described in Section 7.3.4. Tabulate the area response of the primary characteristic ion against concentration for each compound

including the internal standards. Calculate response factors (RF) for each compound as follows:

$$RF = (A_s C_{is})/A_{is} C_s)$$

where:

A_s = Area of the primary characteristic ion for the compound to be measured

A_{is} = Area of the primary characteristic ion of the internal standard

C_{is} = Concentration of the internal standard

C_s = Concentration of the compound to be measured.

The internal standard selected for the calculation of the RF of a compound and subsequent quantification of the compound is generally the internal standard that has a retention time closest to that of the compound. It is assumed that a linear calibration plot will be obtained over the range of concentrations used. If the RF value over the working range is a constant (less than 10% relative standard deviation), the RF can be assumed to be invariant, and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, versus RF.

7.1.3.4 The RF must be verified on each working day. The concentrations selected should be near the midpoint of the working range. The response factors obtained for the calibration standards analyzed immediately before and after a set of samples must be within $\pm$20% of the response factor used for quantification of the sample concentrations.

7.2 Daily GC/MS performance tests

7.2.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for BFB (see Table 2).

7.2.2 The BFB performance test requires the following instrumental parameters:

Electron Energy:	70 volts (nominal)
Mass Range:	40 to 250 amu
Scan Time:	to give approximately 6 scans per peak but not to exceed 3 sec per scan.

7.2.3 Bleed BFB vapor into the mass spectrometer and tune the instrument to achieve all the key ion criteria for the mass spectrum of BFB given in Table 1. A solution containing 20 ng of BFB may be injected onto the gas chromatographic column in order to check the key ion criteria.

7.2.4 The peak intensity of D_6-benzene is used to monitor the mass spectrometer sensitivity. The peak intensity for D_6-benzene observed during each sample analysis must be between 0.7 and 1.4 times the D_6-benzene peak intensity observed during the applicable calibration runs. For example, if the peak intensity of D_6-benzene observed during calibration was 355,000 area counts, then each subsequent sample or blank must give a D_6-benzene peak intensity of between 250,000 and 500,000 area counts. If the D_6-benzene peak intensity is outside the specified range, the sample must be reanalyzed. If the peak intensity is again outside the specified range, the analyst must investigate the cause of the variability in sensitivity and correct the problem.

7.3 Sample extraction and analysis

7.3.1 The analytical procedure involves extracting the non-aqueous sample with methanol or polyethylene glycol (PEG) and analyzing a portion of the extract by a purge-and-trap GC/MS procedure. The amount of the extract to be taken for the GC/MS analysis is based on the estimated total volatile content (TVC) of the sample. The TVC is estimated by extracting the sample with n-hexadecane and analyzing the n-hexadecane extract by gas chromatography.

7.3.2 The estimated TVC is based on the total area response relative to that of n-nonane for all components eluting prior to the retention time of n-dodecane. The response factor for n-nonane and the retention time of n-dodecane are determined by analyzing a 2-µl aliquot of an n-hexadecane solution containing 0.20 mg/ml of n-nonane and n-dodecane.

7.3.2.1 The GC analyses are conducted using a flame ionization detector and a 3-m x 2-mm I.D. glass column packed with 10% OV-101 on 100-200 mesh Chromosorb W-HP. The column temperature is programmed from 80° C to 280° C at 8°/min and held at 280° for 10 min.

7.3.2.2 Determine the area response for n-nonane and divide by 0.2 to obtain the area response factor. Record the retention time of n-dodecane.

7.3.2.3 Add 1.0 g of sample to 20 ml of n-hexadecane and 2 ml of 2.0 M Na_2HPO_4 contained in a 50-ml glass centrifuge tube and cap securely with a Teflon-lined screw cap. Shake the mixture vigorously for one minute. If the sample does not disperse

during the shaking process, sonify the mixture in an ultrasonic bath for 30 min. Allow the mixture to stand until a clear supernatant is obtained. Centrifuge if necessary to facilitate phase separation.

7.3.2.4 Analyze a 2-µl aliquot of the n-hexadecane supernatant using the conditions described in Section 7.3.2.1. Determine the total area response of all components eluting prior to the retention time of n-dodecane and subtract the corresponding area of an n-hexadecane blank. Using the area response factor determined for n-nonane in Section 7.3.2.2, calculate the TVC as follows:

$$TVC = \frac{TAR_{sample} - TAR_{blank}}{\text{n-Nonane Area Response Factor}} \times 20$$

where:

TVC = total volatile content of the sample in mg/g

TAR_{sample} = total area response obtained for the sample

TAR_{blank} = total area response obtained for a blank.

7.3.3 The transfer of an aliquot of the sample for extraction with methanol or PEG should be made as quickly as possible to minimize loss of volatiles from the sample.

7.3.3.1 To a 50-ml glass centrifuge tube with Teflon-lined cap, add 40 ml of reagent methanol or PEG. Weigh the capped centrifuge tube and methanol or PEG on an analytical balance.

7.3.3.2 Using an appropriate implement (see Section 4.4), transfer approximately 2 g of sample to the methanol or PEG in the centrifuge tube in such a fashion that the sample is dissolved in or submerged in the methanol or PEG as quickly as possible. Take care not to touch the sample-transfer implement to the methanol or PEG. Recap the centrifuge tube immediately and weigh on an analytical balance to determine an accurate sample weight.

7.3.3.3 Disperse the sample by vigorous agitation for 1 min. The mixture may be agitated manually or with the aid of a vortex-mixer. If the sample does not disperse during this process, sonify the mixture in an ultrasonic bath for 30 min. Allow the mixture to stand until a clear supernatant is obtained as the sample extract. Centrifuge if necessary to facilitate phase separation.

7.3.3.4 The sample extract may be stored for future analytical needs. If this is desired, transfer the solution to a 10-ml screw cap vial with Teflon cap liner. Store at -10 to -20° C, and protect from light.

7.3.4 Reagent water, internal standard solution, and the sample extract are added to a purging chamber that is connected to the purge-and-trap device and that has been flushed with helium during a 7-min trap reconditioning step (see Section 7.3.4.4). The additions are made using an appropriately sized syringe equipped with a 15-cm 20-gauge needle. Open the syringe valve of the sample inlet (shown in Figure 1) and insert the needle through the valve.

7.3.4.1 Add 5.0 ml of reagent water or aqueous sample to which 20.0 µl of the internal standard solution has been added (see Section 5.10) to the purging chamber. Insert the needle of the syringe well below the surface of the water for the addition of the internal standard solution. If the sample is aqueous go to Section 7.3.5.

7.3.4.2 Add an aliquot of the sample extract from Section 7.3.3.4. The total quantity of volatile components injected should not exceed approximately 10 µg. If the total volatile content (TVC) of the sample as determined in Section 7.3.1.4 is 1.0 mg/g or less, use a 200-µl aliquot of the sample extract. If the TVC is greater than 1.0 mg/g, use an aliquot of the sample extract that contains approximately 10 µg of total volatile components; the volume (in µl) of the aliquot to be taken can be calculated by dividing 200 by the TVC. If the TVC is greater than 20 mg/g, take a 500-µl aliquot of the sample extract and dilute to 10 ml with PEG. In this case calculate the aliquot volume (in µl) of the undiluted extract to be taken by dividing 4,000 by the TVC. If the TVC is less than 1.0 mg/g and greater sensitivity is desired, use a large purging chamber containing 25 ml of reagent water and use a 1.0-ml aliquot of the sample extract.

7.3.4.3 Close the 2-way syringe valve at the sample inlet.

7.3.5 The sample in the purging chamber is purged with helium to transfer the volatile components to the trap. The trap is then heated to desorb the volatile components which are swept by the helium carrier gas onto the GC column for analysis.

7.3.5.1 Adjust the gas (helium) flow rate to 40 $\pm$ 3 ml/min. Set the purging device to purge, and purge the sample for 11.0 $\pm$ 0.1 min at ambient temperature.

7.3.5.2 At the conclusion of the purge time, adjust the device to the desorb mode, and begin the GC/MS analysis and data acquisition using the following GC operating conditions:

Column: 6-ft x 2-mm I.D. glass column of 1% SP-1000 on Carbo-pack B (60-80 mesh).

Temperature: Isothermal at 45° C for 3 min, then increased at 8° C/min to 220° C, and maintained at 220° C for 15 min.

Concurrently, introduce the trapped materials to the GC column by rapidly heating the trap to 180° C while backflushing the trap with helium at a flow rate of 30 ml/min for 4 min. If this rapid heating requirement cannot be met, the GC column must be used as a secondary trap by cooling it to 30° C or lower during the 4-min desorb step and starting the GC program after the desorb step.

7.3.5.3 Return the purge-and-trap device to the purge mode and continue acquiring GC/MS data.

7.3.5.4 Allow the trap to cool for 8 min. Replace the purging chamber with a clean purging chamber. The purging chamber is cleaned after each use by sequential washing with acetone, methanol, detergent solution and distilled water, and then dried at 105° C.

7.3.5.5 Close the syringe valve on the purging chamber after 15 sec to begin gas flow through the trap. Purge the trap at ambient temperature for 4 min. Recondition the trap by heating it to 180° C. Do not allow the trap temperature to exceed 180° C, since the sorption/desorption is adversely affected when the trap is heated to higher temperatures. After heating the trap for approximately 7 min, turn off the trap heater. When cool, the trap is ready for the next sample.

7.3.6 If the response for any ion exceeds the working range of the system, repeat the analysis using a correspondingly smaller aliquot of the sample extract described in Section 7.3.2.3.

7.4 Qualitative identification

7.4.1 Obtain an EICP for the primary characteristic ion and at least two other characteristic ions for each compound when practical. The following criteria must be met to make a qualitative identification.

7.4.1.1 The characteristic ions of each compound of interest must maximize in the same or within one scan of each other.

7.4.1.2 The retention time must fall within ±30 sec of the retention time of the authentic compound.

7.4.1.3 The relative peak heights of the characteristic ions in the EICP's must fall within ±20% of the relative intensities of these ions in a reference mass spectrum. Reference spectra may be generated from the standards analyzed by the analyst or from a reference library. All reference spectra generated from standards must be obtained from an appropriately tuned mass spectrometer.

7.5 Quantitative determination

7.5.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. In general, the primary characteristic ion selected should be a relatively intense ion, as interference-free as possible, and as close as possible in mass to the characteristic ion of the internal standard used. Generally, the base peak of the mass spectrum is used.

8.0 Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of the data that are generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within the accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The laboratory must spike all samples including check samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.1.3 Before processing any samples, the analyst should daily demonstrate, through the analysis of an organic-free water method blank, that the entire analytical system is interference-free. The blank samples should be carried through all stages of the sample preparation and measurement steps.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations using a representative sample as a check sample.

8.2.1 Analyze four aliquots of the unspiked check sample according to the method in Section 7.3.

8.2.2 For each compound to be measured, select a spike concentration representative of twice the level found in the unspiked check sample or a level equal to 10 times the expected detection limit, whichever is greater. Prepare a spiking solution by dissolving the compounds in methanol at the appropriate levels.

8.2.3 Spike a minimum of four aliquots of the check sample with the spiking solution to achieve the selected spike concentrations. Spike the samples by adding the spiking solution to the PEG used for the extraction. Analyze the spiked aliquots according to the method in Section 7.3.

8.2.4 Calculate the average percent recovery, R, and the standard deviation of the percent recovery, s, for all compounds and surrogate standards. Background corrections must be made before R and s calculations are performed. The average percent recovery must be greater than 20 for all compounds to be measured and greater than 60 for all surrogate compounds. The percent relative standard deviation of the percent recovery, s/R x 100, must be less than 20 for all compounds to be measured and all surrogate compounds.

8.3 The analyst must calculate method performance criteria for each of the surrogate standards.

8.3.1 Calculate upper and lower control limits for method performance for each surrogate standard, using the values for R and s calculated in Section 8.2.4:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R - 3s

The UCL and LCL can be used to construct control charts that are useful in observing trends in performance.

8.3.2 For each surrogate standard, the laboratory must maintain a record of the R and s values obtained for each surrogate standard in each waste sample analyzed. An accuracy statement should be prepared from these data and updated regularly.

8.4 The laboratory is required to spike all samples with the surrogate standards to monitor spike recoveries. The spiking level used should be that which will give an amount in the purge apparatus that is equal to the amount of the internal standard assuming a 100% recovery of the surrogate standards. If the recovery for any surrogate standard does not fall within the control limits for method performance, the results reported for that sample must be

qualified as being outside of control limits. The laboratory must monitor the frequency of data so qualified to ensure that it remains at or below 5%. Four surrogate standards, namely 1,2-dibromodifluoroethane, bis(perfluoro-isopropyl) ether, fluorobenzene, and m-bromobenzotrifluoride, are recommended for general use to monitor recovery of volatile compounds varying in volatility and polarity.

8.5 Each day, the analyst must demonstrate through the analysis of a process blank that all glassware and reagent interferences are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field replicates may be analyzed to monitor the precision of the sampling technique. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified waste samples do not indicate sufficient sensitivity to detect less than or equal to 1 µg/g of sample, then the sensitivity of the instrument should be increased or the extract subjected to additional cleanup. Detection limits to be used for groundwater samples are indicated in Table 1. Where doubt exists over the identification of a peak on the chromatograph, confirmatory techniques such as mass spectroscopy should be used.

8.8 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1 were obtained using reagent water. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

8.9 In a single laboratory, using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 3 were obtained. The standard deviation of the measurement in percent recovery is also included in Table 3.

TABLE 3. ACCURACY AND PRECISION FOR PURGEABLE ORGANICS

Parameter	Reagent Water		Wastewater	
	Average percent recovery	Standard deviation (%)	Average percent recovery	Standard deviation (%)
Benzene	99	9	98	10
Bromodichloromethane	102	12	103	10
Bromoform	104	14	105	16
Bromomethane	100	20	88	23
Carbon tetrachloride	102	16	104	15
Chlorobenzene	100	7	102	9
Chloroethane	97	22	103	31
2-Chloroethyl vinyl ether	101	13	95	17
Chloroform	101	10	101	12
Chloromethane	99	19	99	24
Dibromochloromethane	103	11	104	14
1,1-Dichloroethane	101	10	104	15
1,2-Dichloroethane	100	8	102	10
1,1-Dichloroethene	102	17	99	15
trans-1,2-Dichloroethene	99	12	101	10
1,2-Dichloropropane	102	8	103	12
cis-1,3-Dichloropropene	105	15	102	19
trans-1,3-Dichloropropene	104	11	100	18
Ethyl benzene	100	8	103	10
Methylene chloride	96	16	89	28
1,1,2,2-Tetrachloroethane	102	9	104	14
Tetrachloroethene	101	9	100	11
Toluene	101	9	98	14
1,1,1-Trichloroethane	101	11	102	16
1,1,2-Trichloroethane	101	10	104	15
Trichloroethene	101	9	100	12
Trichlorofluoromethane	103	11	107	19
Vinyl chloride	100	13	98	25

Samples were spiked between 10 and 1000 µg/l.

Volatile Organics in Soil by Headspace:
Sampling Protocol

1. Sample containers: 30-mL serum vials with teflon^{R}lined crimp-top caps.

   Prepared containers are labeled & weighed with caps in place Weight is recorded directly on the label, caps are taped in place to keep caps with the respective vial and keep the vial clean inside.

2. Approximately 5-10 grams of the soil are placed into the prepared and preweighed 30-mL serum vial. Care is to be taken during sampling not to place pebbles into the vials. A minimum amount of handling of the soil insures a more accurate analysis.

   Note: To obtain approximately 5-10 grams of soil, the vial should only be filled 1/3 to 1/2 full. (NO MORE THAN 1/2 FULL!)

3. After placing the soil into the vial, clean any particles off the lip of the vial with a chem-wipe and crimp the teflon^{R}-lined septum cap firmly in place, with the teflon^{R} liner of the septum towards the soil.

4. Obtain a total of three separate samples from each location to allow for dilutions, spikes, duplicates, or confirmations when needed.

ORGANIC CARBON, TOTAL

Method 415.1 (Combustion or Oxidation)

STORET NO. Total 00680
Dissolved 00681

1. Scope and Application
   1.1 This method includes the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes. Exclusions are noted under Definitions and Interferences.
   1.2 The method is most applicable to measurement of organic carbon above 1 mg/1.
2. Summary of Method
   2.1 Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation. The CO_2 formed can be measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 is directly proportional to the concentration of carbonaceous material in the sample.
3. Definitions
   3.1 The carbonaceous analyzer measures all of the carbon in a sample. Because of various properties of carbon-containing compounds in liquid samples, preliminary treatment of the sample prior to analysis dictates the definition of the carbon as it is measured. Forms of carbon that are measured by the method are:
      A) soluble, nonvolatile organic carbon; for instance, natural sugars.
      B) soluble, volatile organic carbon; for instance, mercaptans.
      C) insoluble, partially volatile carbon; for instance, oils.
      D) insoluble, particulate carbonaceous materials, for instance; cellulose fibers.
      E) soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter; for instance, oily matter adsorbed on silt particles.
   3.2 The final usefulness of the carbon measurement is in assessing the potential oxygen-demanding load of organic material on a receiving stream. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, or on water taken directly from the stream. In this light, carbonate and bicarbonate carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon which are measured. Instrument manufacturer's instructions should be followed.

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4. Sample Handling and Preservation

4.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. **NOTE 1:** A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

4.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

4.3 In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, the sample is acidified ($pH \leq 2$) with HCl or H_2SO_4.

5. Interferences

5.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

5.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particles which may be included in the sample.

6. Apparatus

6.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

6.2 Apparatus for total and dissolved organic carbon:

6.2.1 A number of companies manufacture systems for measuring carbonaceous material in liquid samples. Considerations should be made as to the types of samples to be analyzed, the expected concentration range, and forms of carbon to be measured.

6.2.2 No specific analyzer is recommended as superior.

7. Reagents

7.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank. Carbon dioxide-free, double distilled water is recommended. Ion exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.

7.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.

NOTE 2: Sodium oxalate and acetic acid are not recommended as stock solutions.

7.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

7.4 Carbonate-bicarbonate, stock solution, 1000 mg carbon/liter: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 ml volumetric flask. Dissolve with distilled water.

7.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to step 7.3.

NOTE 3: This standard is not required by some instruments.

7.6 Blank solution: Use the same distilled water (or similar quality water) used for the preparation of the standard solutions.

8. Procedure

8.1 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

8.2 For calibration of the instrument, it is recommended that a series of standards encompassing the expected concentration range of the samples be used.

9. Precision and Accuracy

9.1 Twenty-eight analysts in twenty-one laboratories analyzed distilled water solutions containing exact increments of oxidizable organic compounds, with the following results:

Increment as TOC mg/liter	Precision as Standard Deviation TOC, mg/liter	Accuracy as Bias, %	Accuracy as Bias, mg/liter
4.9	3.93	+15.27	+0.75
107	8.32	+ 1.01	+1.08

(FWPCA Method Study 3, Demand Analyses)

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METHOD 9020

TOTAL ORGANIC HALIDES (TOX)

1.0 Scope and Application

1.1 Method 9020 determines Total Organic Halides (TOX) as Cl^- in drinking and ground waters. The method uses carbon adsorption with a microcoulometric-titration detector. It requires that all samples be run in duplicate. Under conditions of duplicate analysis, the reliable limit of sensitivity is 5 µg/l.

1.2 Method 9020 detects all organic halides containing chlorine, bromine and iodine that are adsorbed by granular activated carbon under the conditions of the method. Fluorine-containing species are not determined by this method.

1.3 Method 9020 is applicable to samples whose inorganic-halide concentration does not exceed the organic-halide concentration by more than 20,000 times.

1.4 Method 9020 is restricted to use by, or under the supervision of, analysts experienced in the operation of a pyrolysis/microcoulometer and in the interpretation of the results.

1.5 This method is provided as a recommended procedure. It may be used as a reference for comparing the suitability of other methods thought to be appropriate for measurement of TOX (i.e., by comparison of sensitivity, accuracy, and precision data).

2.0 Summary of Method

2.1 A sample of water that has been protected against the loss of volatiles by the elimination of headspace in the sampling container, and that is free of undissolved solids, is passed through a column containing 40 mg of activated carbon. The column is washed to remove any trapped inorganic halides, and is then analyzed to convert the adsorbed organohalides to a titratable species that can be measured by a microcoulometric detector.

3.0 Interferences

3.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be

routinely demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

3.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by treating with chromate cleaning solution. This should be followed by detergent washing in hot water. Rinse with tap water and distilled water, drain dry, and heat in a muffle furnace at 400° C for 15 to 30 min. Volumetric ware should not be heated in a muffle furnace. Glassware should be sealed and stored in a clean environment after drying and cooling to prevent any accumulation of dust or other contaminants.

3.1.2 The use of high purity reagents and gases helps to minimize interference problems.

3.2 Purity of the activated carbon must be verified before use. Only carbon samples that register less than 1000 ng/40 mg should be used. The stock of activated carbon should be stored in its granular form in a glass container with a Teflon seal. Exposure to the air must be minimized, especially during and after milling and sieving the activated carbon. No more than a two-week supply should be prepared in advance. Protect carbon at all times from all sources of halogenated organic vapors. Store prepared carbon and packed columns in glass containers with Teflon seals.

4.0 Apparatus and Materials

4.1 Adsorption system

4.1.1 Dohrmann adsorption module (AD-2), or equivalent, pressurized, sample and nitrate-wash reservoirs.

4.1.2 Adsorption columns: Pyrex, 5-cm-long x 6-mm-O.D. x 2-mm-I.D.

4.2.3 Granular activated carbon (GAC): Filtrasorb-400, Calgon-APC or equivalent, ground or milled, and screened to a 100/200 mesh range. Upon combustion of 40 mg of GAC, the apparent-halide background should be 1000 mg Cl^- equivalent or less.

4.1.4 Cerafelt (available from Johns-Manville), or equivalent: Form this material into plugs using a 2-mm-I.D. stainless-steel borer with ejection rod (available from Dohrmann) to hold 40 mg of GAC in the adsorption columns. CAUTION: Do not touch this material with your fingers.

4.1.5 Column holders (available from Dohrmann).

4.1.6 Volumetric flasks: 100-ml, 50-ml. A general schematic of the adsorption system is shown in Figure 1.

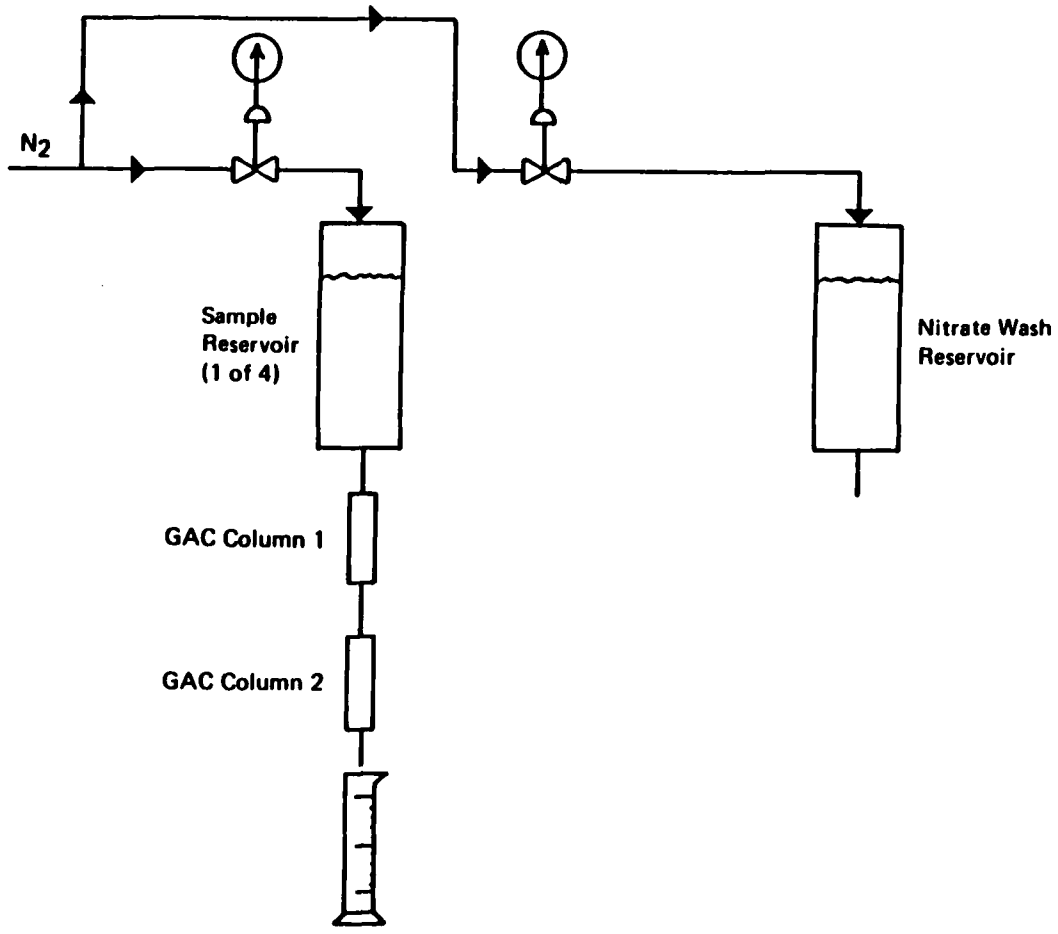


Figure 1. Schematic of Adsorption System.

4.2 Dohrmann microcoulometric-titration system (MCTS-20 or DX-20), or equivalent, containing the following components:

4.2.1 Boat sampler.

4.2.2 Pyrolysis furnace.

4.2.3 Microcoulometer with integrator.

4.2.4 Titration cell: A general description of the analytical system is shown in Figure 2.

4.3 Strip chart recorder.

5.0 Reagents

5.1 Sodium sulfite: 0.1 M, ACS reagent grade (12.6 g/liter).

5.2 Nitric acid: Concentrated.

5.3 Nitrate-wash solution (5000 mg NO_3^-/l): Prepare a nitrate-wash solution by transferring approximately 8.2 g of potassium nitrate into a 1-liter volumetric flask and diluting to volume with reagent water.

5.4 Carbon dioxide: Gas, 99.9% purity.

5.5 Oxygen: 99.9% purity.

5.6 Nitrogen: Prepurified.

5.7 70% acetic acid in water: Dilute 7 volumes of acetic acid with 3 volumes of water.

5.8 Trichlorophenol solution, stock (1 µl = 10 µg Cl^-): Prepare a stock solution by weighing accurately 1.856 g of trichlorophenol into a 100-ml volumetric flask. Dilute to volume with methanol. 10,000 ug/ml

5.9 Trichlorophenol solution, calibration (1 µl = 500 ng Cl^-): = 500 ug/ml
Dilute 5 ml of the trichlorophenol stock solution to 100 ml with methanol.

5.10 Trichlorophenol standard, instrument-calibration: First, nitrate-wash a single column packed with 40 mg of activated carbon as instructed for sample analysis, and then inject the column with 10 µl of the calibration solution.

5.11 Trichlorophenol standard, adsorption-efficiency (100 µg Cl^-/liter): Prepare an adsorption-efficiency standard by injecting 10 µl of stock solution into 1 liter of reagent water.

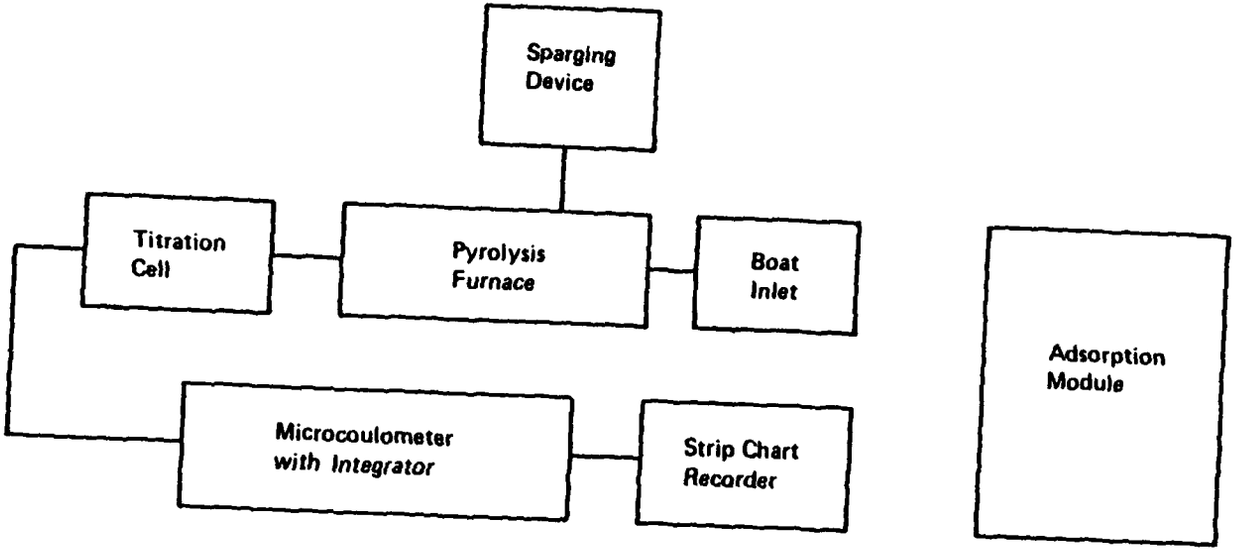


Figure 2. Schematic diagram of CAOX analysis system.

5.12 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

5.13 Blank standard: The reagent water used to prepare the calibration standard should be used as the blank standard.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All samples should be collected in bottles with teflon septa (e.g., Pierce #12722 or equivalent) and be protected from light. If this is not possible, use amber glass, 250-ml, fitted with teflon-lined caps. Foil may be substituted for teflon if the sample is not corrosive. Samples must be protected against loss of volatiles by eliminating headspace in the container. If amber bottles are not available, protect samples from light. The container must be washed and muffled at 400° C before use, to minimize contamination.

6.3 All glassware must be dried prior to use according to the method discussed in 3.1.1.

7.0 Procedure

7.1 Sample preparation

7.1.1 Special care should be taken in handling the sample in order to minimize the loss of volatile organohalides. The adsorption procedure should be performed simultaneously on duplicates.

7.1.2 Reduce residual chlorine by adding sulfite (1 ml of 0.1 M per liter of sample). Sulfite should be added at the time of sampling if the analysis is meant to determine the TOX concentration at the time of sampling. It should be recognized that TOX may increase on storage of the sample. Samples should be stored at 4° C without headspace.

7.1.3 Adjust the pH of the sample to approximately 2 with concentrated HNO_3 just prior to adding the sample to the reservoir.

7.2 Calibration

7.2.1 Check the adsorption efficiency of each newly-prepared batch of carbon by analyzing 100 ml of the adsorption-efficiency standard, in duplicate, along with duplicates of the blank standard. The net recovery should be within 5% of the standard value.

7.2.2 Nitrate-wash blanks (method blanks): Establish the repeatability of the method background each day by first analyzing several nitrate-wash blanks. Monitor this background by spacing nitrate-wash blanks between each group of eight pyrolysis determinations. The nitrate-wash blank values are obtained on single columns packed with 40 mg of activated carbon. Wash with the nitrate solution as instructed for sample analysis, and then pyrolyze the carbon.

7.2.3 Pyrolyze duplicate instrument-calibration standards and the blank standard each day before beginning sample analysis. The net response to the calibration-standard should be within 3% of the calibration-standard value. Repeat analysis of the instrument-calibration standard after each group of eight pyrolysis determinations, and before resuming sample analysis after cleaning or reconditioning the titration cell or pyrolysis system.

7.3 Adsorption procedure

7.3.1 Connect two columns in series, each containing 40 mg of 100/200-mesh activated carbon.

7.3.2 Fill the sample reservoir, and pass a metered amount of sample through the activated-carbon columns at a rate of approximately 3 ml/min. NOTE: 100 ml of sample is the preferred volume for concentrations of TOX between 5 and 500 µg/l; 50 ml for 501 to 1000 µg/l, and 25 ml for 1001 to 2000 µg/l.

7.3.3 Wash the columns-in-series with 2 ml of the 5000-mg/l nitrate solution at a rate of approximately 2 ml/min to displace inorganic chloride ions.

7.4 Pyrolysis procedure

7.4.1 The contents of each column are pyrolyzed separately. After rinsing with the nitrate solution, the columns should be protected from the atmosphere and other sources of contamination until ready for further analysis.

7.4.2 Pyrolysis of the sample is accomplished in two stages. The volatile components are pyrolyzed in a CO_2-rich atmosphere at a low temperature to ensure the conversion of brominated trihalomethanes to a titratable species. The less volatile components are then pyrolyzed at a high temperature in an O_2-rich atmosphere. NOTE: The quartz sampling boat should have been previously muffled at 800° C for at least 2 to 4 min as in a previous analysis, and should be cleaned of any residue by vacuuming.

7.4.3 Transfer the contents of each column to the quartz boat for individual analysis.

7.4.4 If the Dohrmann MC-1 is used for pyrolysis, manual instructions are followed for gas flow regulation. If the MCTS-20 is used, the information on the diagram in Figure 3 is used for gas flow regulation.

7.4.5 Position the sample for 2 min in the 200° C zone of the pyrolysis tube. For the MCTS-20, the boat is positioned just outside the furnace entrance.

7.4.6 After 2 min, advance the boat into the 800° C zone (center) of the pyrolysis furnace. This second and final stage of pyrolysis may require from 6 to 10 min to complete.

7.5 Detection: The effluent gases are directly analyzed in the micro-coulometric-titration cell. Carefully follow manual instructions for optimizing cell performance.

7.6 Breakthrough. The unpredictable nature of the background bias makes it especially difficult to recognize the extent of breakthrough of organohalides from one column to another. All second-column measurements for a properly operating system should not exceed 10% of the two-column total measurement. If the 10% figure is exceeded, one of three events can be happening. Either (1) the first column was overloaded and a legitimate measure of breakthrough was obtained, in which case taking a smaller sample may be necessary; or (2) channeling or some other failure occurred, in which case the sample may need to be rerun; or (3) a high random bias occurred and the result should be rejected and the sample rerun. Because it may not be possible to determine which event occurred, a sample analysis should be repeated often enough to gain confidence in results. As a general rule, any analysis that is rejected should be repeated whenever sample is available. If the second-column measurement is equal to or less than the nitrate-wash blank value, the second-column value should be disregarded.

7.7 Calculations: TOX as Cl^- is calculated using the following formula:

$$\frac{(C_1-C_3) + (C_2 - C_3)}{V} = \mu g/l \text{ Total Organic Halide}$$

where:

C_1 = µg Cl^- on the first column in series

C_2 = µg Cl^- on the second column in series

C_3 = predetermined, daily, average, method-blank value (nitrate-wash blank for a 40-mg carbon column)

V = the sample volume in liters.

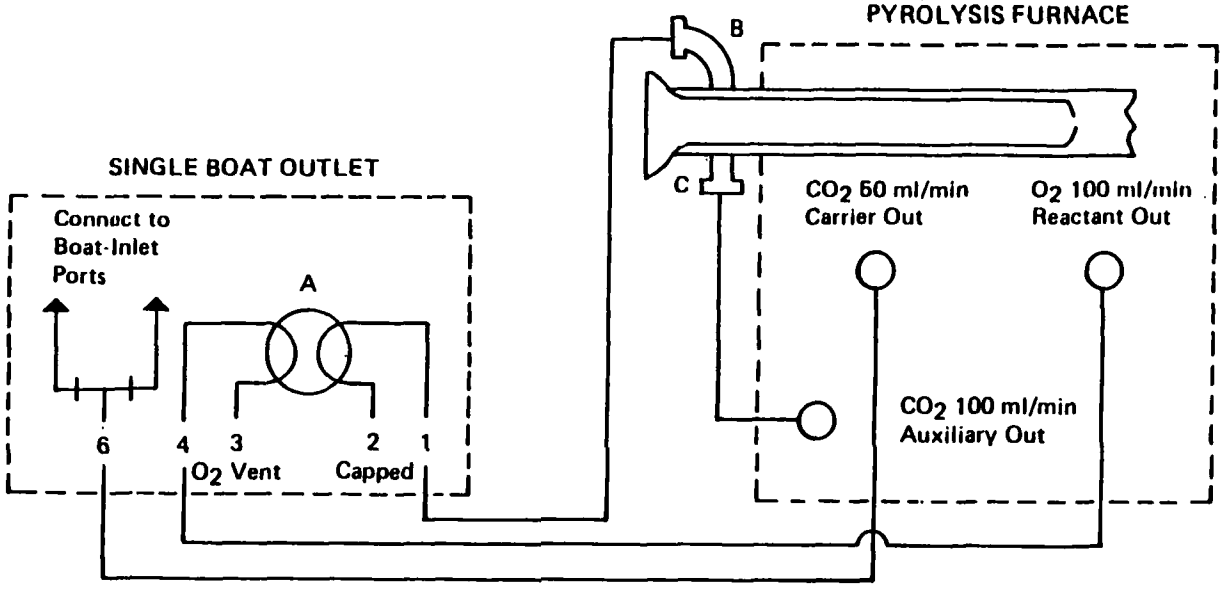


Figure 3. Rear-view plumbing schematic for MCTS–20 System. Valve A is set for first-stage combustion, O_2 venting (push/pull valve out). Port B enters inner combustion tube; Port C enters outer combustion tube.

8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this procedure by analyzing appropriate quality-control check samples.

8.3 The laboratory must develop and maintain a statement of method accuracy for their laboratory. The laboratory should update the accuracy statement regularly as new recovery measurements are made.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Run check standard after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparations process.

8.7 It is recommended that the laboratory adopt additional quality-assurance practices for use with this method. The specific practices that would be most productive will depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance-evaluation studies.

OIL AND GREASE, TOTAL RECOVERABLE

Method 413.2 (Spectrophotometric, Infrared)

STORET NO. 00560

1. Scope and Application
   - 1.1 This method includes the measurement of fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.
   - 1.2 The method is applicable to measurement of most light petroleum fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.
   - 1.3 The method covers the range from 0.2 to 1000 mg/1 of extractable material.
   - 1.4 While this method can be used to obtain an estimate of the oil and grease that would be measured gravimetrically, in many cases the estimate more accurately describes the parameter, as it will measure volatiles more effectively and is not susceptible to interferences such as extractable sulfur. It can be used with the Petroleum Hydrocarbon procedure to obtain an oil and grease value and a petroleum hydrocarbon value on the same sample.
2. Summary of Method
   - 2.1 The sample is acidified to a low pH (< 2) and extracted with fluorocarbon-113. The oil and grease is determined by comparison of the infrared absorbance of the sample extract with standards.
3. Definitions
   - 3.1 The definition of oil and grease is based on the procedure used. The source of the oil and/or grease, and the presence of extractable non-oily matter will influence the material measured and interpretation of results.
4. Sampling and Storage
   - 4.1 A representative sample of 1 liter volume should be collected in a glass bottle. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 5 ml HCl (6.1) at the time of collection and refrigerated at 4°C.
   - 4.2 Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentration over an extended period.
5. Apparatus
   - 5.1 Separatory funnel, 2000 ml, with Teflon stopcock.
   - 5.2 Infrared spectrophotometer, scanning. Non-scanning instruments may also be used but can be subject to positive interferences in complex chemical wastewaters.
   - 5.3 Cells, 10 mm, 50 mm, and 100 mm path length, sodium chloride or infrared grade glass.
   - 5.4 Filter paper, Whatman No. 40, 11 cm.

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6. Reagents

6.1 Hydrochloric acid, 1:1. Mix equal volumes of conc. HCl and distilled water.

6.2 Fluorocarbon-113, (1,1,2-trichloro-1,2,2-trifluoroethane), b. p. 48°C.

6.3 Sodium sulfate, anhydrous crystal.

6.4 Calibration mixtures:

6.4.1 Reference oil: Pipet 15.0 ml n-hexadecane, 15.0 ml isooctane, and 10.0 ml chlorobenzene into a 50 ml glass stoppered bottle. Maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

6.4.2 Stock standard: Pipet 1.0 ml reference oil (6.4.1) into a tared 200 ml volumetric flask and immediately stopper. Weigh and dilute to volume with fluorocarbon-113.

6.4.3 Working standards: Pipet appropriate volumes of stock standard (6.4.2) into 100 ml volumetric flasks according to the cell pathlength to be used. Dilute to volume with fluorocarbon-113. Calculate concentration of standards from the stock standard.

7. Procedure

7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 ml hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.

7.2 Pour the sample into a separatory funnel.

7.3 Add 30 ml fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate.

7.4 Filter the solvent layer into a 100 ml volumetric flask through a funnel containing solvent-moistened filter paper.

NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

7.5 Repeat (7.3 and 7.4) twice more with 30 ml portions of fresh solvent, combining all solvent in the volumetric flask.

7.6 Rinse the tip of the separatory funnel, filter paper, and the funnel with a total of 5–10 ml fluorocarbon-113 and collect the rinsings in the flask. Dilute the extract to 100 ml, and stopper the flask.

7.7 Select appropriate working standards and cell pathlength according to the following table of approximate working ranges:

Pathlength	Range
10 mm	2–40 mg
50 mm	0.4–8 mg
100 mm	0.1–4 mg

7.8 Scan standards and samples from 3200 cm^{-1} to 2700 cm^{-1} with fluorocarbon-113 in the reference beam and record the results on absorbance paper. The absorbances of samples

and standards are measured by constructing a straight baseline over the range of the scan and measuring the absorbance of the peak maximum at 2930 cm^{-1} and subtracting the baseline absorbance at that point. For an example of a typical oil spectrum and baseline construction, see Gruenfeld[3]. Non-scanning instruments should be operated according to manufacturer's instructions, although calibration must be performed using the standards described above (6.4). If the absorbance exceeds 0.8 for a sample, select a shorter pathlength or dilute as required.

7.9 Use a calibration plot of absorbance vs. mg oil prepared from the standards to determine the mg oil in the sample solution.

8. Calculation

8.1 $\text{mg/l total oil and grease} = \frac{R \times D}{V}$

where:

R = oil in solution, determined from calibration plot, in milligrams.
D = extract dilution factor, if used.
V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

9. Precision and Accuracy

9.1 The two oil and grease methods in this manual were tested by a single laboratory (EMSL) on sewage. This method determined the oil and grease level in the sewage to be 17.5 mg/l. When 1 liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 99% with a standard deviation of ±1.4 mg/l.

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PHENOLICS, TOTAL RECOVERABLE

Method 420.1 (Spectrophotometric, Manual 4-AAP with Distillation)

STORET NO. 32730

1. Scope and Application
   - 1.1 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The method is capable of measuring phenolic materials at the 5 *ug/1* level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.
   - 1.3 The method is capable of measuring phenolic materials that contain more than 50 *ug/1* in the aqueous phase (without solvent extraction) using phenol as a standard.
   - 1.4 It is not possible to use this method to differentiate between different kinds of phenols.
2. Summary of Method
   - 2.1 Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.
3. Comments
   - 3.1 For most samples a preliminary distillation is required to remove interfering materials.
   - 3.2 Color response of phenolic materials with 4-amino antipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.
4. Sample Handling and Preservation
   - 4.1 Biological degradation is inhibited by the addition of *1 g/1 of copper sulfate to the* sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.
5. Interference
   - 5.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4 with H_3PO_4 and aerating briefly by stirring and adding $CuSO_4$.
   - 5.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate (~~6.5~~ 7.5). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

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6. Apparatus
   - 6.1 Distillation apparatus, all glass consisting of a 1 liter pyrex distilling apparatus with Graham condenser.
   - 6.2 pH meter.
   - 6.3 Spectrophotometer, for use at 460 or 510 nm.
   - 6.4 Funnels.
   - 6.5 Filter paper.
   - 6.6 Membrane filters.
   - 6.7 Separatory funnels, 500 or 1,000 ml.
   - 6.8 Nessler tubes, short or long form.
7. Reagents
   - 7.1 Phosphoric acid solution, 1 + 9: Dilute 10 ml of 85% H_3PO_4 to 100 ml with distilled water.
   - 7.2 Copper sulfate solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in distilled water and dilute to 1 liter.
   - 7.3 Buffer solution: Dissolve 16.9 g NH_4Cl in 143 ml conc. NH_4OH and dilute to 250 ml with distilled water. Two ml should adjust 100 ml of distillate to pH 10.
   - 7.4 Aminoantipyrine solution: Dissolve 2 g of 4AAP in distilled water and dilute to 100 ml.
   - 7.5 Potassium ferricyanide solution: Dissolve 8 g of $K_3Fe(CN)_6$ in distilled water and dilute to 100 ml.
   - 7.6 Stock phenol solution: Dissolve ~~1.0 g~~ 1.00g phenol in freshly boiled and cooled distilled water and dilute to 1 liter. 1 ml = 1 mg phenol.
   - 7.7 Working solution A: Dilute 10 ml stock phenol solution to 1 liter with distilled water. 1 ml = 10 *ug* phenol.
   - 7.8 Working solution B: Dilute 100 ml of working solution A to 1000 ml with distilled water. 1 ml = 1 *ug* phenol.
   - 7.9 Chloroform
8. Procedure
   - 8.1 Distillation
     - 8.1.1 Measure 500 ml sample into a beaker. Lower the pH to approximately 4 with 1 + 9 H_3PO_4 (7.1), add 5 ml $CuSO_4$ solution (7.2) and transfer to the distillation apparatus. Omit adding H_3PO_4 and $CuSO_4$ if sample was preserved as described in 4.1.
     - 8.1.2 Distill 450 ml of sample, stop the distillation, and when boiling ceases add 50 ml of warm distilled water to the flask and resume distillation until 500 ml have been collected.
     - 8.1.3 If the distillate is turbid, filter through a prewashed membrane filter.
   - 8.2 Direct photometric method
     - 8.2.1 Using working solution A (7.7), prepare the following standards in 100 ml volumetric flasks.

ml of working solution A	Conc. ug/l
0	0.0
0.5	50.0
1.0	100.0
2.0	200.0
5.0	500.0
8.0	800.0
10.0	1000.0

8.2.2 To 100 ml of distillate or an aliquot diluted to 100 ml and/or standards, add 2 ml of buffer solution (7.3) and mix. The pH of the sample and standards should be 10 ±0.2.

8.2.3 Add 2.0 ml aminoantipyrine solution (7.4) and mix.

8.2.4 Add 2.0 ml potassium ferricyanide solution (7.5) and mix.

8.2.5 After 15 minutes read absorbance at 510 nm.

8.3 Chloroform extraction method

8.3.1 Using working solution B (7.8), prepare the following standards. Standards may be prepared by pipetting the required volumes into the separatory funnels and diluting to 500 ml with distilled water.

ml of working solution B	Conc. ug/l
0.0	0.0
3.0	6.0
5.0	10.0
10.0	20.0
20.0	40.0
25.0	50.0

8.3.2 Place 500 ml of distillate or an aliquot diluted to 500 ml in a separatory funnel. The sample should not contain more than 25 ug phenol.

8.3.3 To sample and standards add 10 ml of buffer solution (7.3) and mix. The pH should be 10 ±0.2.

8.3.4 Add 3.0 ml aminoantipyrine solution (7.4) and mix.

8.3.5 Add 3.0 ml potassium ferricyanide solution (7.5) and mix.

8.3.6 After three minutes, extract with 25 ml of chloroform (7.9). Shake the separatory funnel at least 10 times, let $CHCl_3$ settle, shake again 10 times and let chloroform settle again.

8.3.7 Filter chloroform extracts through filter paper. Do not add more chloroform.

8.3.8 Read the absorbance of the samples and standards against the blank at 460 nm.

9. Calculation

9.1 Prepare a standard curve by plotting the absorbance value of standards versus the corresponding phenol concentrations.

9.2 Obtain concentration value of sample directly from standard curve.

10. Precision and Accuracy

10.1 Using the extraction procedure for concentration of color, six laboratories analyzed samples at concentrations of 9.6, 48.3, and 93.5 *ug*/1. Standard deviations were ±0.99, ±3.1 and ±4.2 *ug*/1, respectively.

10.2 Using the direct photometric procedure, six laboratories analyzed samples at concentrations of 4.7, 48.2 and 97.0 mg/1. Standard deviations were ±0.18, ±0.48 and ±1.58 mg/1, respectively.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D1783–70, p553 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p574–581, Method 510 through 510C, (1975).

CYANIDE, TOTAL

Method 335.2 (Titrimetric; Spectrophotometric)

STORET NO. 00720

1. Scope and Application
   - 1.1 This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The titration procedure using silver nitrate with p-dimethylamino-benzal-rhodanine indicator is used for measuring concentrations of cyanide exceeding 1 mg/1 (0.25 mg/250 ml of absorbing liquid).
   - 1.3 The colorimetric procedure is used for concentrations below 1 mg/1 of cyanide and is sensitive to about 0.02 mg/1.
2. Summary of Method
   - 2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.
   - 2.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.
   - 2.3 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.
3. Definitions
   - 3.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.
4. Sample Handling and Preservation
   - 4.1 The sample should be collected in plastic or glass bottles of 1 liter or larger size. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.
   - 4.2 Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

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4.3 Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample ($pH \geq 12$) at the time of collection.

4.4 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C.

5. Interferences

5.1 Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1 through 8.5.

5.2 Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 ml more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide as described in 4.3.

5.3 Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect.

5.3.1 Acidify the sample with acetic acid (1+9) to pH 6.0 to 7.0.

Caution: This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed.

5.3.2 Extract with iso-octane, hexane, or chloroform (preference in order named) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.

6. Apparatus

6.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber.

6.2 Microburet, 5.0 ml (for titration).

6.3 Spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.

7. Reagents

7.1 Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.

7.2 Cadmium carbonate: powdered.

7.3 Ascorbic acid: crystals.

7.4 Dilute sodium hydroxide solution, 0.25N: Dilute 200 ml of sodium hydroxide solution (7.1) to 1000 ml with distilled water.

7.5 Sulfuric acid: concentrated.

7.6 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in 1 liter of distilled water. Refrigerate this solution.

7.7 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 liter of distilled water. Standardize with 0.0192 N $AgNO_3$. Dilute to appropriate concentration so that 1 ml = 1 mg CN.

7.8 Standard cyanide solution, intermediate: Dilute 50.0 ml of stock (1 ml = 1 mg CN) to 1000 ml with distilled water (1 ml = 50.0ug).

7.9 Standard cyanide solution: Prepare fresh daily by diluting 100.0 ml of intermediate cyanide solution to 1000 ml with distilled water and store in a glass stoppered bottle. 1 ml = 5.0ug CN (5.0 mg/1).

7.10 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ crystals and drying to constant weight at 40°C. Weigh out 3.2647 g of dried $AgNO_3$, dissolve in distilled water, and dilute to 1000 ml (1 ml = 1mg CN).

7.11 Rhodanine indicator: Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100 ml of acetone.

7.12 Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly.

7.13 Color Reagent — One of the following may be used:

7.13.1 Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCl (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

7.13.2 Pyridine-pyrazolone solution:

7.13.2.1 3-Methyl-1-phenyl-2-pyrazolin-5-one reagent, saturated solution: Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolin-5-one to 50 ml of distilled water, heat to 60°C with stirring. Cool to room temperature.

7.13.2.2 3,3'Dimethyl-1, 1'-diphenyl-[4,4'-bi-2 pyrazoline]-5,5'dione (bispyrazolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine.

7.13.2.3 Pour solution (7.13.2.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (7.13.2.2) collecting the filtrate in the same container as filtrate from (7.13.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.

7.14 Magnesium chloride solution: Weight 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 ml flask, dissolve and dilute to 1 liter with distilled water.

8. Procedure

8.1 Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Add 50 ml of sodium hydroxide (7.1) to the absorbing tube and dilute if necessary with distilled

water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

8.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

Caution: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

8.3 Slowly add 25 ml conc. sulfuric acid (7.5) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride (7.4) into the air inlet and wash down with a stream of water.

8.4 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

8.5 Drain the solution from the absorber into a 250 ml volumetric flask and bring up to volume with distilled water washings from the absorber tube.

8.6 Withdraw 50 ml or less of the solution from the flask and transfer to a 100 ml volumetric flask. If less than 50 ml is taken, dilute to 50 ml with 0.25 N sodium hydroxide solution (7.4). Add 15.0 ml of Sodium phosphate solution (7.6) and mix.

8.6.1 Pyridine – Barbituric Acid Method: Add 2 ml of chloramine T (7.12) and mix. After 1 to 2 minutes, add 5 ml of pyridine – barbituric acid solution (7.13.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

8.6.2 Pyridine – pyrazolone method: Add 0.5 ml of chloramine T (7.12) and mix. After 1 to 2 minutes add 5 ml of pyridine – pyrazolone solution (7.13.1) and mix. Dilute to mark with distilled water and mix again. After 40 minutes read absorbance at 620 nm in a 1 cm cell.

NOTE: More than 0.5 ml of Chloramine T will prevent the color from developing with pyridine-pyrazolone.

8.7 Prepare a series of standards by pipeting suitable volumes of standard solution into 250 ml volumetric flasks. To each standard add 50 ml of 1.25 N sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ML of Standard Solution (1.0 = 5 ug CN)	Conc. mgCN per 250 ml
0	BLANK
1.0	5
2.0	10
5.0	25
10.0	50
15.0	60
20.0	100

8.7.1 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree within ±10% of the undistilled standards the operator should find the cause of the apparent error before proceeding.

8.7.2 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

8.7.3 To check the efficiency of the sample distillation, add an increment of cyanide from either the intermediate standard (7.8) or the working standard (7.9) to insure a level of 20 *ug*/1 or a significant increase in absorbance value. Proceed with the analysis as in Procedure (8.8.1) using the same flask and system from which the previous sample was just distilled.

8.8 Alternatively, if the sample contains more than 1 mg of CN transfer the distillate, or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10–12 drops of the benzalrhodanine indicator.

8.9 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

8.10 The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 ml microburet may be conveniently used to obtain a more precise titration.

9. Calculation

9.1 If the colorimetric procedure is used, calculate the cyanide, in *ug*/1, in the original sample as follows:

$$\text{CN, } ug/l = \frac{A \times 1{,}000}{B} \times \frac{50}{C}$$

where:

A = *ug* CN read from standard curve
B = ml of original sample for distillation
C = ml taken for colorimetric analysis

METALS

(Atomic Absorption Methods)

1. Scope and Application

1.1 Metals in solution may be readily determined by atomic absorption spectroscopy. The method is simple, rapid, and applicable to a large number of metals in drinking, surface, and saline waters, and domestic and industrial wastes. While drinking waters free of particulate matter may be analyzed directly, domestic and industrial wastes require processing to solubilize suspended material. Sludges, sediments and other solid type samples may also be analyzed after proper pretreatment.

1.2 Detection limits, sensitivity and optimum ranges of the metals will vary with the various makes and models of satisfactory atomic absorption spectrophotometers. The data shown in Table 1, however, provide some indication of the actual concentration ranges measurable by direct aspiration and using furnace techniques. In the majority of instances the concentration range shown in the table by direct aspiration may be extended much lower with scale expansion and conversely extended upwards by using a less sensitive wavelength or by rotating the burner head. Detection limits by direct aspiration may also be extended through concentration of the sample and/or through solvent extraction techniques. Lower concentrations may also be determined using the furnace techniques. The concentration ranges given in Table 1 are somewhat dependent on equipment such as the type of spectrophotometer and furnace accessory, the energy source and the degree of electrical expansion of the output signal. When using furnace techniques, however, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To insure valid data with furnace techniques, the analyst must examine each matrix for interference effects (see 5.2.1) and if detected, treat accordingly using either successive dilution, matrix modification or method of standard additions (see 8.5).

1.3 Where direct aspiration atomic absorption techniques do not provide adequate sensitivity, in addition to the furnace procedure, reference is made to specialized procedures such as the gaseous hydride method for arsenic and selenium, the cold vapor technique for mercury, and the chelation–extraction procedure for selected metals. Reference to approved colorimetric methods is also made.

1.4 Atomic absorption procedures are provided as the methods of choice; however, other instrumental methods have also been shown to be capable of producing precise and accurate analytical data. These instrumental techniques include emission spectroscopy, X-ray fluorescence, spark source mass spectroscopy, and anodic stripping to name but a few. The analyst should be cautioned that these methods are highly specialized techniques requiring a high degree of skill to interpret results and obtain valid data.

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These above mentioned techniques are presently considered as alternate test procedures and approval must be obtained prior to their use.

2. Summary of Method

2.1 In direct aspiration atomic absorption spectroscopy a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp whose cathode is made of the element to be determined is directed through the flame into a monochromator, and onto a detector that measures the amount of light absorbed. Absorption depends upon the presence of free unexcited ground state atoms in the flame. Since the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectroscopy.

2.2 Although methods have been reported for the analysis of solids by atomic absorption spectroscopy (Spectrochim Acta, *24B* 53, 1969) the technique generally is limited to metals in solution or solubilized through some form of sample processing.

2.2.1 Preliminary treatment of wastewater and/or industrial effluents is usually necessary because of the complexity and variability of the sample matrix. Suspended material must be subjected to a solubilization process before analysis. This process may vary because of the metals to be determined and the nature of the sample being analyzed. When the breakdown of organic material is necessitated, the process should include a wet digestion with nitric acid.

2.2.2 In those instances where complete characterization of a sample is desired, the suspended material must be analyzed separately. This may be accomplished by filtration and acid digestion of the suspended material. Metallic constituents in this acid digest are subsequently determined and the sum of the dissolved plus suspended concentrations will then provide the total concentrations present. The sample should be filtered as soon as possible after collection and the filtrate acidified immediately.

2.2.3 The total sample may also be treated with acid without prior filtration to measure what may be termed "total recoverable" concentrations.

2.3 When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atoms are vaporized and dissociated for absorption in the tube than the flame, the use of small sample volumes or detection of low concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption except a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor.

TABLE 1

Atomic Absorption Concentration Ranges[1]

	Direct Aspiration			Furnace Procedure[4,5]	
Metal	Detection Limit mg/l	Sensitivity mg/l	Optimum Concentration Range mg/l	Detection Limit ug/l	Optimum Concentration Range ug/l
Aluminum	0.1	1	5 - 50	3	20 - 200
Antimony	0.2	0.5	1 - 40	3	20 - 300
Arsenic[2]	0.002	-	0.002 - 0.02	1	5 - 100
Barium(p)	0.1	0.4	1 - 20	2	10 - 200
Beryllium	0.005	0.025	0.05 - 2	0.2	1 - 30
Cadmium	0.005	0.025	0.05 - 2	0.1	0.5 - 10
Calcium	0.01	0.08	0.2 - 7	-	-
Chromium	0.05	0.25	0.5 - 10	1	5 - 100
Cobalt	0.05	0.2	0.5 - 5	1	5 - 100
Copper	0.02	0.1	0.2 - 5	1	5 - 100
Gold	0.1	0.25	0.5 - 20	1	5 - 100
Iridium(p)	3	8	20 - 500	30	100 - 1500
Iron	0.03	0.12	0.3 - 5	1	5 - 100
Lead	0.1	0.5	1 - 20	1	5 - 100
Magnesium	0.001	0.007	0.02 - 0.5	-	-
Manganese	0.01	0.05	0.1 - 3	0.2	1 - 30
Mercury[3]	0.0002	-	0.0002 - 0.01	-	-
Molybdenum(p)	0.1	0.4	1 - 40	1	3 - 60
Nickel(p)	0.04	0.15	0.3 - 5	1	5 - 100
Osmium	0.3	1	2 - 100	20	50 - 500
Palladium(p)	0.1	0.25	0.5 - 15	5	20 - 400
Platinum(p)	0.2	2	5 - 75	20	100 - 2000
Potassium	0.01	0.04	0.1 - 2	-	-
Rhenium(p)	5	15	50 - 1000	200	500 - 5000
Rhodium(p)	0.05	0.3	1 - 30	5	20 - 400
Ruthenium	0.2	0.5	1 - 50	20	100 - 2000
Selenium[2]	0.002	-	0.002 - 0.02	2	5 - 100
Silver	0.01	0.06	0.1 - 4	0.2	1 - 25
Sodium	0.002	0.015	0.03 - 1	-	-
Thallium	0.1	0.5	1 - 20	1	5 - 100
Tin	0.8	4	10 - 300	5	20 - 300
Titanium (p)	0.4	2	5 - 100	10	50 - 500
Vanadium (p)	0.2	0.8	2 - 100	4	10 - 200
Zinc	0.005	0.02	0.05 - 1	0.05	0.2 - 4

(1) The concentrations shown are not contrived values and should be obtainable with any satisfactory atomic absorption spectrophotometer.

(2) Gaseous hydride method.

(3) *Cold vapor technique.*

(4) For furnace sensitivity values consult instrument operating manual.

(5) The listed furnace values are those expected when using a 20 *ul* injection and normal gas flow except in the case of arsenic and selenium where gas interrupt is used. The symbol (p) indicates the use of pyrolytic graphite with the furnace procedure.

The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation.

3. Definition of Terms

3.1 Optimum Concentration Range: A range, defined by limits expressed in concentration, below which scale expansion must be used and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating condition employed.

3.2 Sensitivity: The concentration in milligrams of metal per liter that produces an absorption of 1%.

3.3 Detection Limit: Detection limits can be expressed as either an instrumental or method parameter. The limiting factor of the former using acid water standards would be the signal to noise ratio and degree of scale expansion used; while the latter would be more affected by the sample matrix and preparation procedure used. The Scientific Apparatus Makers Association (SAMA) has approved the following definition for detection limit: that concentration of an element which would yield an absorbance equal to twice the standard deviation of a series of measurements of a solution, the concentraton of which is distinctly detectable above, but close to blank absorbance measurement. The detection limit values listed in Table I and on the individual analysis sheets are to be considered minimum working limits achievable with the procedures given in this manual. These values may differ from the optimum detection limit reported by the various instrument manufacturers.

3.4 Dissolved Metals: Those constituents (metals) which will pass through a 0.45 u membrane filter.

3.5 Suspended Metals: Those constituents (metals) which are retained by a 0.45 u membrane filter.

3.6 Total Metals: The concentration of metals determined on an unfiltered sample following vigorous digestion (Section 4.1.3), or the sum of the concentrations of metals in both the dissolved and suspended fractions.

3.7 Total Recoverable Metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid (Section 4.1.4).

4. Sample Handling and Preservation

4.1 For the determination of trace metals, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. For liquid samples, containers can introduce either positive or negative errors in the measurement of trace metals by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. The sample bottle whether borosilicate glass, linear polyethylene, polyproplyene or Teflon should be thoroughly washed with detergent and tap water; rinsed with 1:1 nitric acid,

tap water, 1:1 hydrochloric acid, tap water and finally deionized distilled water in that order.

NOTE 1: Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product—NOCHROMIX—available from Godax Laboratories, 6 Varick St. New York, N.Y. 10013, may be used in place of chromic acid. [Chromic acid should not be used with plastic bottles.]

NOTE 2: If it can be documented through an active analytical quality control program using spiked samples, reagent and sample blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

Before collection of the sample a decision must be made as to the type of data desired, i.e., dissolved, suspended, total or total recoverable. For container preference, maximum holding time and sample preservation at time of collection see Table 1 in the front part of this manual. Drinking water samples containing suspended and setteable material should be prepared using the total recoverable metal procedure (section 4.1.4).

4.1.1 For the determination of dissolved constituents the sample must be filtered through a 0.45 u membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus using plain, non-grid marked, membrane filters are recommended to avoid possible contamination.) Use the first 50–100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of < 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the sample (See Note 3). If hexavalent chromium is to be included in the analytical scheme, a portion of the filtrate should be transferred before acidification to a separate container and analyzed as soon as possible using Method 218.3. Analyses performed on a sample so treated shall be reported as "dissolved" concentrations.

NOTE 3: If a precipitate is formed upon acidification, the filtrate should be digested using 4.1.3. Also, it has been suggested (International Biological Program, Symposium on Analytical Methods, Amsterdam, Oct. 1966) that additional acid, as much as 25 ml of conc. HCl/liter, may be required to stabilize certain types of highly buffered samples if they are to be stored for any length of time. Therefore, special precautions should be observed for preservation and storage of unusual samples intended for metal analysis.

4.1.2 For the determination of suspended metals a representative volume of unpreserved sample must be filtered through a 0.45 *u* membrane filter. When considerable suspended material is present, as little as 100 ml of a well mixed sample is filtered. Record the volume filtered and transfer the membrane filter containing the insoluble material to a 250 ml Griffin beaker and add 3 ml conc. redistilled HNO_3. Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane. Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 ml of conc. redistilled HNO_3. Cover and continue heating until

the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (DO NOT BAKE), add 5 ml distilled HCl (1:1) and warm the beaker gently to dissolve any soluble material. (If the sample is to be analyzed by the furnace procedure, 1 ml of 1:1 distilled HNO_3 per 100 ml dilution should be substituted for the distilled 1:1 HCl.) Wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected concentrations of metals present. This volume will vary depending on the metal to be determined. The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended" (See Note 4.)

NOTE 4: Certain metals such as antimony arsenic, gold, iridium, mercury, osmium, palladium, platinium, rhenium, rhodium, ruthenium, selenium, silver, thallium, tin and titanium require modification of the digestion procedure and the individual sheets for these metals should be consulted.

4.1.3 For the determination of total metals the sample is acidified with 1:1 redistilled HNO_3 to a pH of less than 2 at the time of collection. The sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. If much suspended material is present, as little as 50–100 ml of well mixed sample will most probably be sufficient. (The sample volume required may also vary proportionally with the number of metals to be determined.)

Transfer a representative aliquot of the well mixed sample to a Griffin beaker and add 3 ml of conc. redistilled HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil. (DO NOT BAKE.) Cool the beaker and add another 3 ml portion of conc. redistilled HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, evaporate to near dryness and cool the beaker. Add a small quantity of redistilled 1:1 HCl (5 ml/100 ml of final solution) and warm the beaker to dissolve any precipitate or residue resulting from evaporation. (If the sample is to be analyzed by the furnace procedure, substitute distilled HNO_3 for 1:1 HCl so that the final dilution contains 0.5% (v/v) HNO_3.) Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis. Concentrations so determined shall be reported as "total" (see Note 4).

4.1.4 To determine total recoverable metals, acidify the entire sample at the time of collection with conc. redistilled HNO_3, 5 ml/1. At the time of analysis a 100 ml aliquot of well mixed sample is transferred to a beaker or flask. Five ml of distilled HCl (1:1) is added and the sample heated on a steam bath or hot plate until the

volume has been reduced to 15–20 ml making certain the samples do not boil. (If the sample is being prepared for furnace analysis, the same process should be followed except HCl should be omitted.) After this treatment the sample is filtered to remove silicates and other insoluble material that could clog the atomizer and the volume adjusted to 100 ml. The sample is then ready for analysis. Concentrations so determined shall be reported as "total". (See Notes 4, 5, and 6.)

NOTE 5: The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy certain metal complexes if a colorimetric procedure is to be employed for the final determination. When this is suspect, the more vigorous digestion given in 4.1.3 should be followed.

NOTE 6: For drinking water analyses by direct aspiration, the final volume may be reduced to effect up to a 10X concentration of the sample, provided the total dissolved solids in the original sample do not exceed 500 mg/1, the determination is corrected for any non-specific absorbance and there is no loss by precipitation.

5. Interferences

5.1 Direct Aspiration

5.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or because the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome the phosphate interference in the magnesium, calcium and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium.

5.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. While complexing agents are primarily employed to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

5.1.3 The presence of high dissolved solids in the sample may result in an interference from non-atomic absorbance such as light scattering. If background correction is not available, a non-absorbing wavelength should be checked. Preferably, high solids type samples should be extracted (see 5.1.1 and 9.2).

5.1.4 Ionization interferences occur where the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positive charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess of an easily ionized element.

5.1.5 Although quite rare, spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Also, interference can occur

when resonant energy from another element in a multi-element lamp or a metal impurity in the lamp cathode falls within the bandpass of the slit setting and that metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.

5.2 Flameless Atomization

5.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical and matrix interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference use the following procedure. Withdraw from the sample two equal aliquots. To one of the aliquots add a known amount of analyte and dilute both aliquots to the same predetermined volume. [The dilution volume should be based on the analysis of the undiluted sample. Preferably, the dilution should be 1:4 while keeping in mind the optimum concentration range of the analysis. Under no circumstances should the dilution be less than 1:1]. The diluted aliquots should then be analyzed and the unspiked results multiplied by the dilution factor should be compared to the original determination. Agreement of the results (within ±10%) indicates the absence of interference. Comparison of the actual signal from the spike to the expected response from the analyte in an aqueous standard should help confirm the finding from the dilution analysis. Those samples which indicate the presence of interference, should be treated in one or more of the following ways.

a. The samples should be successively diluted and reanalyzed to determine if the interference can be eliminated.

b. The matrix of the sample should be modified in the furnace. Examples are the addition of ammonium nitrate to remove alkali chlorides, ammonium phosphate to retain cadmium, and nickel nitrate for arsenic and selenium analyses [ATOMIC ABSORPTION NEWSLETTER Vol. 14, No. 5, p 127, Sept-Oct 1975]. The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.

c. Analyze the sample by method of standard additions while noting the precautions and limitations of its use (See 8.5).

5.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, either the use of background correction or choosing an alternate wavelength outside the absorption band should eliminate this interference. Non-specific broad band absorption interference can also be compensated for with background correction.

5.2.3 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in

the presence of air. Care must be taken, however, to prevent loss of the analysis element.

5.2.4 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion prior to being placed in the furnace. In this way broad band absorption will be minimized.

5.2.5 From anion interference studies in the graphite furnace it is generally accepted that nitrate is the preferred anion. Therefore nitric acid is preferable for any digestion or solubilization step. If another acid in addition to HNO_3 is required a minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

5.2.6 Carbide formation resulting from the chemical environment of the furnace has been observed with certain elements that form carbides at high temperatures. Molybdenum may be cited as an example. When this takes place, the metal will be released very slowly from the carbide as atomization continues. For molybdenum, one may be required to atomize for 30 seconds or more before the signal returns to baseline levels. This problem is greatly reduced and the sensitivity increased with the use of pyrolytically-coated graphite.

5.2.7 Ionization interferences have to date not been reported with furnace techniques.

5.2.8 For comments on spectral interference see section 5.1.5.

5.2.9 Contamination of the sample can be a major source of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed in part 6.9 of the Atomic Absorption Methods section of this manual. Pipet tips have been known to be a source of contamination. If suspected, they should be acid soaked with 1:5 HNO_3 and rinsed thoroughly with tap and deionized water. The use of a better grade pipet tip can greatly reduce this problem. It is very important that special attention be given to reagent blanks in both analysis and the correction of analytical results. Lastly, pyrolytic graphite because of the production process and handling can become contaminated. As many as five to possibly ten high temperature burns may be required to clean the tube before use.

6. Apparatus

6.1 Atomic absorption spectrophotometer: Single or dual channel, single-or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip chart recorder.

6.2 Burner: The burner recommended by the particular instrument manufacturer should be used. For certain elements the nitrous oxide burner is required.

6.3 Hollow cathode lamps: Single element lamps are to be preferred but multi-element lamps may be used. Electrodeless discharge lamps may also be used when available.

6.4 Graphite furnace: Any furnace device capable of reaching the specified temperatures is satisfactory.

6.5 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can be easily recognized.

6.6 Pipets: Microliter with disposable tips. Sizes can range from 5 to 100 microliters as required. **NOTE 7**: Pipet tips which are white in color, do not contain CdS, and have been found suitable for research work are available from Ulster Scientific, Inc. 53 Main St. Highland, NY 12528 (914) 691-7500.

6.7 Pressure-reducing valves: The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

6.8 Separatory flasks: 250 ml, or larger, for extraction with organic solvents.

6.9 Glassware: All glassware, linear polyethylene, polyproplyene or Teflon containers, including sample bottles, should be washed with detergent, rinsed with tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water and deionized distilled water in that order. [See Notes 1 and 2 under (4.1) concerning the use of chromic acid and the cleaning procedure.]

6.10 Borosilicate glass distillation apparatus.

7. Reagents

7.1 Deionized distilled water: Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized distilled water for the preparation of all reagents, calibration standards, and as dilution water.

7.2 Nitric acid (conc.): If metal impurities are found to be present, distill reagent grade nitric acid in a borosilicate glass distillation apparatus or use a spectrograde acid.

Caution: Distillation should be performed in hood with protective sash in place.

7.2.1 Nitric Acid (1:1): Prepare a 1:1 dilution with deionized, distilled water by adding the conc. acid to an equal volume of water.

7.3 Hydrochloric acid (1:1): Prepare a 1:1 solution of reagent grade hydrochloric acid and deionized distilled water. If metal impurities are found to be present, distill this mixture from a borosilicate glass distillation apparatus or use a spectrograde acid.

7.4 Stock standard metal solutions: Prepare as directed in (8.1) and under the individual metal procedures. Commercially available stock standard solutions may also be used.

7.5 Calibration standards: Prepare a series of standards of the metal by dilution of the appropriate stock metal solution to cover the concentration range desired.

7.6 Fuel and oxidant: Commercial grade acetylene is generally acceptable. Air may be supplied from a compressed air line, a laboratory compressor, or from a cylinder of compressed air. Reagent grade nitrous oxide is also required for certain determinations. Standard, commercially available argon and nitrogen are required for furnace work.

7.7 Special reagents for the extraction procedure.

7.7.1 Pyrrolidine dithiocarbamic acid (PDCA) "see footnote": Prepare by adding 18 ml of analytical reagent grade pyrrolidine to 500 ml of chloroform in a liter flask.

The name pyrrolidine dithiocarbamic acid (PDCA), although commonly referenced in the scientific literature is ambiguous. From the chemical reaction of pyrrolidine and carbon disulfide a more proper name would be 1-pyrrolidine carbodithioic acid, PCDA (CAS Registry No. 25769-03-3).

(See Note 8) Cool and add 15 ml of carbon disulfide in small portions and with swirling. Dilute to 1 liter with chloroform. The solution can be used for several months if stored in a brown bottle in a refrigerator.

NOTE 8: An acceptable grade of pyrrolidine may be obtained from the Aldrich Chemical Co., 940 West St. Paul Ave., Milwaukee, WI. 53233 (414, 273–3850).

7.7.2 Ammonium hydroxide, 2N: Dilute 3 ml conc. NH_4OH to 100 ml with deionized distilled water.

7.7.3 Bromphenol blue indicator (1g/liter): Dissolve 0.1g bromphenol blue in 100 ml of 50 percent ethanol or isopropanol.

7.7.4 HCl, 2.5% v/v: Dilute 2 ml redistilled HCl to 40 ml with deionized distilled water.

8. Preparation of Standards and Calibration

8.1 Stock standard solutions are prepared from high purity metals, oxides or nonhygroscopic reagent grade salts using deionized distilled water and redistilled nitric or hydrochloric acids. (See individual analysis sheets for specific instruction.) Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements. The stock solutions are prepared at concentrations of 1000 mg of the metal per liter. Commercially available standard solutions may also be used.

8.2 Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. For best results, calibration standards should be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least four calibration standards in graduated amounts in the appropriate range. The calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. As filtered water samples are preserved with 1:1 redistilled HNO_3 (3 ml per liter), calibration standards for these analyses should be similarly prepared with HNO_3. Beginning with the blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure a reliable average reading for each solution. Calibration standards for furnace procedures should be prepared as described on the individual sheets for that metal.

8.3 Where the sample matrix is so complex that viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition must be used. This technique relies on the addition of small, known amounts of the analysis element to portions of the sample—the absorbance difference between those and the original solution giving the slope of the calibration curve. The method of standard addition is described in greater detail in (8.5).

8.4 For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of standards which produce an absorption of 0 to 80 percent. The correct method is to convert the percent absorption readings to absorbance and plot that value against concentration. The following relationship is used to convert absorption values to absorbance:

$$\text{absorbance} = \log(100/\%T) = 2 - \log \%T$$
$$\text{where } \%T = 100 - \% \text{ absorption}$$

As the curves are frequently nonlinear, especially at high absorption values, the number of standards should be increased in that portion of the curve.

8.5 Method of Standard Additions: In this method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The volume of the blank and the standards must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Fig. 1.

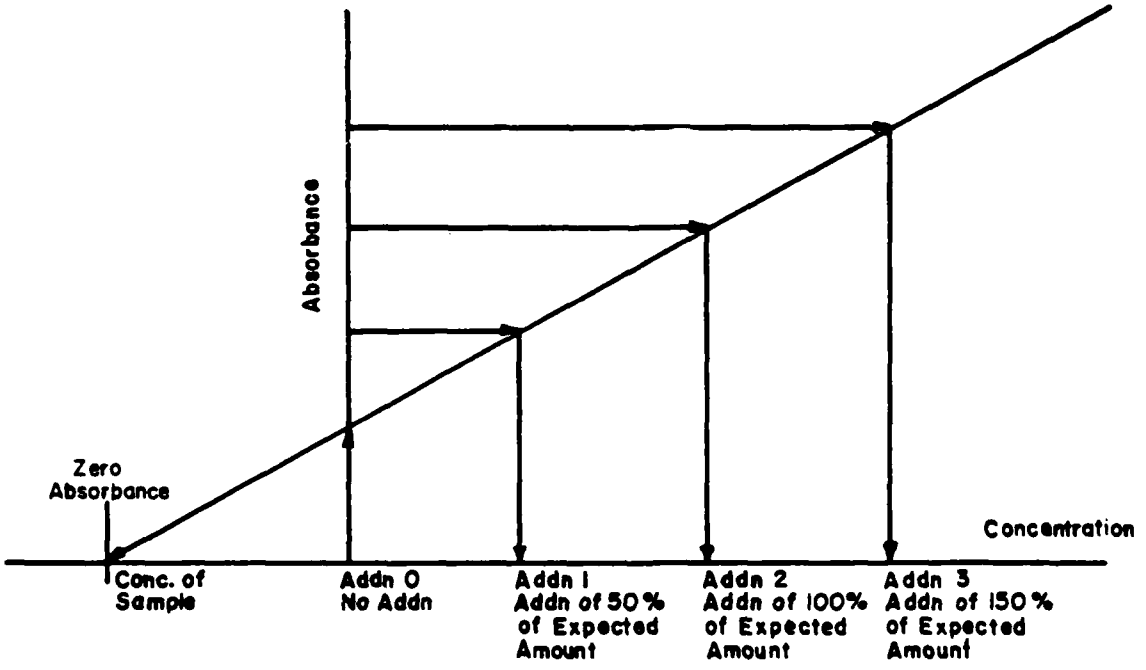


FIGURE 1. STANDARD ADDITION PLOT

The method of standard additions can be very useful, however, for the results to be valid the following limitations must be taken into consideration:

a) the absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%) caution should be exercised.

b) the effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes and the standard addition should respond in a similar manner as the analyte.

c) the determination must be free of spectral interference and corrected for non-specific background interference.

9. General Procedure for Analysis by Atomic Absorption

9.1 Direct Aspiration: Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for his particular instrument. In general, after choosing the proper hollow cathode lamp for the analysis, the lamp should be allowed to warm up for a minimum of 15 minutes unless operated in a double beam mode. During this period, align the instrument, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the hollow cathode current according to the manufactuerer's recommendation. Subsequently, light the flame and regulate the flow of fuel and oxidant, adjust the burner and nebulizer flow rate for maximum percent absorption and stability, and balance the photometer. Run a series of standards of the element under analysis and construct a calibration curve by plotting the concentrations of the standards against the absorbance. For those instruments which read directly in concentration set the curve corrector to read out the proper concentration. Aspirate the samples and determine the concentrations either directly or from the calibration curve. Standards must be run each time a sample or series of samples are run.

9.1.1 Calculation - Direct determination of liquid samples: Read the metal value in mg/1 from the calibration curve or directly from the readout system of the instrument.

9.1.1.1 If dilution of sample was required:

$$\text{mg/l metal in sample} = A \left(\frac{C + B}{C}\right)$$

where:

A = mg/1 of metal indiluted aliquot from calibration curve
B = ml of deionized distilled water used for dilution
C = ml of sample aliquot

9.1.2 For samples containing particulates:

$$\text{mg/l metal in sample} = A\left(\frac{V}{C}\right)$$

where:

A = mg/l of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
C = ml of sample aliquot processed

9.1.3 For solid samples: report all concentrations as mg/kg dry weight

9.1.3.1 Dry sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{D}$$

where:

A = mg/l of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
D = weight of dry sample in grams

9.1.3.2 Wet sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{W \times P}$$

where:

A = mg/l of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
W = weight of wet sample in grams
P = % solids

9.2 Special Extraction Procedure: When the concentration of the metal is not sufficiently high to determine directly, or when considerable dissolved solids are present in the sample, certain metals may be chelated and extracted with organic solvents. Ammonium pyrrolidine dithiocarbamate (APDC) (see footnote) in methyl isobutyl ketone (MIBK) is widely used for this purpose and is particularly useful for zinc, cadmium, iron, manganese, copper, silver, lead and chromium[4-6]. Tri-valent chromium does not react with APDC unless it has first been converted to the hexavalent form [Atomic Absorption Newsletter *6*, p 128 (1967)]. This procedure is described under method 218.3.

The name ammonium pyrrolidine dithiocarbamate (APDC) is somewhat ambiguous and should more properly be called *ammonium, 1-pyrollidine carbodithioate* (APCD), CAS Registry No. 5108-96-3.

Aluminum, beryllium, barium and strontium also do not react with APDC. While the **APDC-MIBK** chelating-solvent system can be used satisfactorily, it is possible to experience difficulties. (See Note 9.)

NOTE 9: Certain metal chelates, manganese-APDC in particular, are not stable in MIBK and will redissolve into the aqueous phase on standing. The extraction of other metals is sensitive to both shaking rate and time. As with cadmium, prolonged extraction beyond 1 minute, will reduce the extraction efficiency, whereas 3 minutes of vigorous shaking is required for chromium.

Also, when multiple metals are to be determined either larger sample volumes must be extracted or individual extractions made for each metal being determined. The acid form of APDC-pyrrolidine dithiocarbamic acid prepared directly in chloroform as described by Lakanen, [Atomic Absorption Newsletter *5*, p 17 (1966)], (see 7.7.1) has been found to be most advantageous. In this procedure the more dense chloroform layer allows for easy combination of multiple extractions which are carried out over a broader pH range favorable to multielement extractions. Pyrrolidine dithiocarbamic acid in chloroform is very stable and may be stored in a brown bottle in the refrigerator for months. Because chloroform is used as the solvent, it may not be aspirated into the flame. The following procedure is suggested.

9.2.1 Extraction procedure with pyrrolidine dithiocarbamic acid (PDCA) in chloroform.

9.2.1.1 Transfer 200 ml of sample into a 250 ml separatory funnel, add 2 drops bromphenol blue indicator solution (7.7.3) and mix.

9.2.1.2 Prepare a blank and sufficient standards in the same manner and adjust the volume of each to approximately 200 ml with deionized distilled water. All of the metals to be determined may be combined into single solutions at the appropriate concentration levels.

9.2.1.3 Adjust the pH by addition of 2N NH_4OH solution (7.7.2) until a blue color persists. Add HCl (7.7.4) dropwise until the blue color just disappears; then add 2.0 ml HCl (7.7.4) in excess. The pH at this point should be 2.3. (The pH adjustment may be made with a pH meter instead of using indicator.)

9.2.1.4 Add 5 ml of PDCA-chloroform reagent (7.7.1) and shake vigorously for 2 minutes. Allow the phases to separate and drain the chloroform layer into a 100 ml beaker. (See NOTE 10.)

NOTE 10: If hexavalent chromium is to be extracted, the aqueous phase must be readjusted back to a pH of 2.3 after the addition of PDCA–chloroform and maintained at that pH throughout the extraction. For multielement extraction, the pH may adjusted upward after the chromium has been extracted.

9.2.1.5 Add a second portion of 5 ml PDCA-chloroform reagent (7.7.1) and shake vigorously for 2 minutes. Allow the phases to separate and combine the chloroform phase with that obtained in step (9.2.1.4).

9.2.1.6 Determine the pH of the aqueous phase and adjust to 4.5.

9.2.1.7 Repeat step (9.2.1.4) again combining the solvent extracts.

9.2.1.8 Readjust the pH to 5.5, and extract a fourth time. Combine all extracts and evaporate to dryness on a steam bath.

9.2.1.9 Hold the beaker at a 45 degree angle, and slowly add 2 ml of conc. distilled nitric acid, rotating the beaker to effect thorough contact of the acid with the residue.

9.2.1.10 Place the beaker on a low temperature hotplate or steam bath and evaporate just to dryness.

9.2.1.11 Add 2 ml of nitric acid (1:1) to the beaker and heat for 1 minute. Cool, quantitatively transfer the solution to a 10 ml volumetric flask and bring to volume with distilled water. The sample is now ready for analysis.

9.2.2 Prepare a calibration curve by plotting absorbance versus the concentration of the metal standard (*ug*/l) in the 200 ml extracted standard solution. To calculate sample concentration read the metal value in *ug*/l from the calibration curve or directly from the readout system of the instrument. If dilution of the sample was required use the following equation:

$$\text{mg/l metal in sample} = Z\left(\frac{C + B}{C}\right)$$

where:

Z = *ug*/l of metal in diluted aliquot from calibration curve
B = ml of deionized distilled water used for dilution
C = ml of sample aliquot

9.3 Furnace Procedure: Furnace devices (flameless atomization) are a most useful means of extending detection limits. Because of differences between various makes and models of satisfactory instruments, no detailed operating instuctions can be given for each instrument. Instead, the analyst should follow the instructions provided by the manufacturer of his particular instrument and use as a guide the temperature settings and other instrument conditions listed on the individual analysis sheets which are recommended for the Perkin-Elmer HGA-2100. In addition, the following points may be helpful.

9.3.1 With flameless atomization, background correction becomes of high importance especially below 350 nm. This is because certain samples, when atomized, may absorb or scatter light from the hollow cathode lamp. It can be caused by the presence of gaseous molecular species, salt particules, or smoke in the sample

beam. If no correction is made, sample absorbance will be greater than it should be, and the analytical result will be erroneously high.

9.3.2 If during atomization all the analyte is not volatilized and removed from the furnace, memory effects will occur. This condition is dependent on several factors such as the volatility of the element and its chemical form, whether pyrolytic graphite is used, the rate of atomization and furnace design. If this situation is detected through blank burns, the tube should be cleaned by operating the furnace at full power for the required time period as needed at regular intervals in the analytical scheme.

9.3.3 Some of the smaller size furnace devices, or newer furnaces equipped with feedback temperature control (Instrumentation Laboratories MODEL 555, Perkin-Elmer MODELS HGA 2200 and HGA 76B, and Varian MODEL CRA-90) employing faster rates of atomization, can be operated using lower atomization temperatures for shorter time periods than those listed in this manual.

9.3.4 Although prior digestion of the sample in many cases is not required providing a representative aliquot of sample can be pipeted into the furnace, it will provide for a more uniform matrix and possibly lessen matrix effects.

9.3.5 Inject a measured microliter aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

9.3.6 To verify the absence of interference, follow the procedure as given in part 5.2.1.

9.3.7 A check standard should be run approximately after every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced. Even though tube life depends on sample matrix and atomization temperature, a conservative estimate would be that a tube will last at least 50 firings. A pyrolytic-coating would extend that estimate by a factor of 3.

9.3.8 Calculation–For determination of metal concentration by the furnace: Read the metal value in *ug*/l from the calibration curve or directly from the readout system of the instrument.

9.3.8.1 If different size furnace injection volumes are used for samples than for standards:

$$ug/l \text{ of metal in sample} = Z \left(\frac{S}{U}\right)$$

where:

Z = *ug*/l of metal read from calibration curve or readout system
S = ul volume standard injected into furnace for calibration curve
U = ul volume of sample injected for analysis

9.3.8.2 If dilution of sample was required but sample injection volume same as for standard:

$$\mu g/l \text{ of metal in sample} = Z \left(\frac{C + B}{C}\right)$$

where:

Z = $\mu g/l$ metal in diluted aliquot from calibration curve
B = ml of deionized distilled water used for dilution
C = ml of sample aliquot

9.3.9 For sample containing particulates:

$$\mu g/l \text{ of metal in sample} = Z \left(\frac{V}{C}\right)$$

where:

Z = $\mu g/l$ of metal in processed sample from calibration curve (See 9.3.8.1)
V = final volume of processed sample in ml
C = ml of sample aliquot processed

9.3.10 For solid samples: Report all concentrations as mg/kg dry weight

9.3.10.1 Dry sample:

$$\text{mg metal/kg sample} = \frac{\left(\frac{Z}{1{,}000}\right)V}{D}$$

where:

Z = $\mu g/l$ of metal in processed sample from calibration curve (See 9.3.8.1)
V = final volume of processed sample in ml
D = weight of dry sample in grams

9.3.10.2 Wet sample:

$$\text{mg metal/kg sample} = \frac{\left(\frac{Z}{1{,}000}\right)V}{W \times P}$$

where:

Z = $\mu g/l$ of metal in processed sample from calibration curve (See 9.3.8.1)
V = final volume of processed sample in ml
W = weight of wet sample in grams
P = % solids

10 Quality Control For Drinking Water Analysis

10.1 Minimum requirements

10.1.1 All quality control data should be maintained and available for easy reference or inspection.

10.1.2 An unknown performance sample (when available) must be analyzed once per year for the metals measured. Results must be within the control limit established by EPA. If problems arise, they should be corrected, and a follow-up performance sample should be analyzed.

10.2 Minimum Daily control

10.2.1 After a calibration curve composed of a minimum of a reagent blank and three standards has been prepared, subsequent calibration curves must be verified by use of at least a reagent blank and one standard at or near the MCL. Daily checks must be within ±10 percent of original curve.

10.2.2 If 20 or more samples per day are analyzed, the working standard curve must be verified by running an additional standard at or near the MCL every 20 samples. Checks must be within ±10 percent of original curve.

10.3 Optional Requirements

10.3.1 A current service contract should be in effect on balances and the atomic absorption spectrophotometer.

10.3.2 Class S weights should be available to make periodic checks on balances.

10.3.3 Chemicals should be dated upon receipt of shipment and replaced as needed or before shelf life has been exceeded.

10.3.4 A known reference sample (when available) should be analyzed once per quarter for the metals measured. The measured value should be within the control limits established by EPA.

10.3.5 At least one duplicate sample should be run every 10 samples, or with each set of samples to verify precision of the method. Checks should be within the control limit established by EPA.

10.3.6 Standard deviation should be obtained and documented for all measurements being conducted.

10.3.7 Quality Control charts or a tabulation of mean and standard deviation should be used to document validity of data on a daily basis.

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303 A. Determination of Antimony, Bismuth, Cadmium*, Calcium, Cesium, Chromium*, Cobalt*, Copper, Gold, Iridium, Iron*, Lead*, Lithium, Magnesium, Manganese*, Nickel*, Platinum, Potassium, Rhodium, Ruthenium, Silver*, Sodium, Strontium, Thallium, Tin, and Zinc* by Direct Aspiration into an Air-Acetylene Flame

1. Apparatus

Atomic absorption spectrophotometer and associated equipment: See Section 303.2. Use burner head recommended by the manufacturer.

*For low concentrations of Cd, Cr, and Pb (<50, 200, and 500 µg/L respectively) and Co, Fe, Mn, Ni, Ag, and Zn, see Section 303B.

2. Reagents

a. Air, cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or commercially bottled gas.

b. Acetylene, standard commercial grade. Acetone, which always is present in acetylene cylinders, can be prevented

from entering and damaging the burner head by replacing a cylinder when its pressure has fallen to 689 kPa acetylene.

c. Metal-free water: Use metal-free water for preparing all reagents and calibration standards and as dilution water. Prepare metal-free water by deionizing tap water and/or by using one of the following processes, depending on the metal concentration in the sample: single distillation, redistillation, or sub-boiling. Always check deionized or distilled water to determine whether the element of interest is present in trace amounts. (CAUTION: If the source water contains Hg or other volatile metals, deionized and single- or redistilled water may not be suitable for trace analysis because these metals distill over with the distilled water. In such cases, use sub-boiling to prepare metal-free water).

d. Calcium solution: Dissolve 630 mg calcium carbonate, $CaCO_3$, in 50 mL of 1 + 5 HCl. If necessary, heat and boil gently to obtain complete solution. Cool and dilute to 1,000 mL with water.

e. Hydrochloric acid, HCl, conc.

f. Lanthanum solution: Dissolve 58.65 g lanthanum oxide, La_2O_3, in 250 mL conc HCl. Add acid slowly until the material is dissolved and dilute to 1,000 mL with water.

g. Hydrogen peroxide, 30%.

h. Nitric acid, HNO_3, conc.

i. Aqua regia: Add 3 volumes conc HCl to 1 volume conc HNO_3.

j. Iodine solution, 1*N*: Dissolve 20 g potassium iodide, KI, in 50 mL water, add 12.7 g iodine, and dilute to 100 mL.

k. Cyanogen iodide (CNI) solution: To 50 mL water add 6.5 g potassium cyanide, KCN, 5.0 mL 1*N* iodine solution, and 4.0 mL conc NH_4OH. Mix and dilute to 100 mL with water. Prepare fresh solution every 2 wk.

l. Standard metal solutions: Prepare a series of standard metal solutions in the optimum concentration range by appropriate dilution of the following stock metal solutions with water containing 1.5 mL conc HNO_3/L. Thoroughly dry reagents before use. In general, use reagents of the highest purity. For hydrates, use fresh reagents.

1) *Antimony:* Dissolve 2.7426 g antimony potassium tartrate hemihydrate (analytical reagent grade), $K(SbO)C_4H_4O_6 \cdot {}^1/_2H_2O$, in 1,000 mL water; 1.00 mL = 1.00 mg Sb.

2) *Bismuth:* Dissolve 1.000 g bismuth metal in a minimum volume of 1 + 1 HNO_3. Dilute to 1,000 mL with 2% (v/v) HNO_3; 1.00 mL = 1.00 mg Bi.

3) *Cadmium:* Dissolve 1.000 g cadmium metal in a minimum volume of 1 + 1 HCl. Dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Cd.

4) *Calcium:* To 2.4972 g $CaCO_3$ add 50 mL water and add dropwise a minimum volume of conc HCl (about 10 mL) to complete solution. Dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Ca.

5) *Cesium:* Dissolve 1.267 g cesium chloride, CsCl, in 1,000 mL water; 1.00 mL = 1.00 mg Cs.

6) *Chromium:* Dissolve 2.828 g anhydrous potassium dichromate, $K_2Cr_2O_7$, in about 200 mL water, add 1.5 mL conc HNO_3, and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Cr.

7) *Cobalt:* Dissolve 1.407 g cobaltic oxide, Co_2O_3, in 20 mL hot conc HCl. Cool and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Co.

8) *Copper:* Dissolve 1.000 g copper metal in 15 mL of 1 + 1 HNO_3 and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Cu.

9) *Gold:* Dissolve 0.1000 g gold metal in a minimum volume of aqua regia. Evaporate to dryness, dissolve residue in 5 mL conc HCl, cool, and dilute to 100 mL with water; 1.00 mL = 1.00 mg Au.

10) *Iridium:* Dissolve 1.147 g ammonium chloroiridate, $(NH_4)_2IrCl_6$, in a minimum volume of 1% (v/v) HCl and dilute to 100

mL with 1% (v/v) HCl; 1.00 mL = 5.00 mg Ir.

11) *Iron:* Dissolve 1.000 g iron wire in 50 mL of 1 + 1 HNO_3 and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Fe.

12) *Lead:* Dissolve 1.598 g lead nitrate, $Pb(NO_3)_2$, in about 200 mL water, add 1.5 mL conc HNO_3, and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Pb.

13) *Lithium:* Dissolve 5.324 g lithium carbonate, Li_2CO_3, in a minimum volume of 1 +1 HCl and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Li.

14) *Magnesium:* Dissolve 4.952 g magnesium sulfate, $MgSO_4$, in 200 mL water, add 1.5 mL conc HNO_3, and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Mg.

15) *Manganese:* Dissolve 3.076 g manganous sulfate, $MnSO_4 \cdot H_2O$, in about 200 mL water, add 1.5 mL conc HNO_3, and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Mn.

16) *Nickel:* Dissolve 1.273 g nickel oxide, NiO, in a minimum volume of 10% (v/v) HCl and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Ni.

17) *Platinum:* Dissolve 0.1000 g platinum metal in a minimum volume of aqua regia and evaporate just to dryness. Add 5 mL conc HCl and 0.1 g NaCl and again evaporate just to dryness. Dissolve residue in 20 mL of 1 + 1 HCl and dilute to 100 mL with water; 1.00 mL = 1.00 mg Pt.

18) *Potassium:* Dissolve 1.907 g potassium chloride, KCl, in water and make up to 1,000 mL; 1.00 mL = 1.00 mg K.

19) *Rhodium:* Dissolve 0.412 g ammonium hexachlororhodate, $(NH_4)_3Rh\ Cl_6 \cdot 1.5\ H_2O$, in a minimum volume of 10% (v/v) HCl and dilute to 100 mL with 10% (v/v) HCl; 1.00 mL = 1.00 mg Rh.

20) *Ruthenium:* Dissolve 0.2052 g ruthenium chloride, $RuCl_3$, in a minimum volume of 20% (v/v) HCl and dilute to 100 mL with 20% (v/v) HCl; 1.00 mL = 1.00 mg Ru.

21) *Silver:* Dissolve 1.575 g silver nitrate, $AgNO_3$, in water, add 1.5 mL conc HNO_3, and make up to 1,000 mL; 1.00 mL = 1.00 mg Ag.

22) *Sodium:* Dissolve 2.542 g sodium chloride, NaCl, dried at 140 C, in water and make up to 1,000 mL; 1.00 mL = 1.00 mg Na.

23) *Strontium:* Dissolve 2.415 g strontium nitrate, $Sr(NO_3)_2$, in 1,000 mL of 1% (v/v) HNO_3; 1.00 mL = 1.00 mg Sr.

24) *Thallium:* Dissolve 1.303 g thallium nitrate, $TlNO_3$, in water. Add 10 mL conc HNO_3 and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Tl.

25) *Tin:* Dissolve 1.000 g tin metal in 100 mL conc HCl and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Sn.

26) *Zinc:* Dissolve 1.000 g zinc metal in 20 mL 1 + 1 HCl and dilute to 1,000 mL with water; 1.00 mL = 1.00 mg Zn.

3. Procedure

a. Instrument operation: Because of differences between makes and models of atomic absorption spectrophotometers, it is not possible to formulate instructions applicable to every instrument. See manufacturer's operating manual. In general, proceed according to the following: Install a hollow cathode lamp for the desired metal in the instrument and roughly set the wavelength dial according to Table 303:1. Set slit width according to manufacturer's suggested setting for the element being measured. Turn on instrument, apply to the hollow cathode lamp the current suggested by the manufacturer, and let instrument warm up until energy source stabilizes, generally about 10 to 20 min. Readjust current as necessary after warm up. Optimize wavelength by adjusting wavelength dial until optimum energy gain is obtained. Align lamp in accordance with manufacturer's instructions.

Install suitable burner head and adjust burner head position. Turn on air and adjust flow rate to that specified by manufacturer to give maximum sensitivity for the

... tion curves for calcium and magnesium based on original concentration of standards before dilution with lanthanum solution. Plot calibration curves for iron and manganese based on original concentration of standards before dilution with calcium solution. Plot calibration curve for chromium based on original concentration of standard before addition of H_2O_2.

Check standards periodically during a run. Recheck calibration curve by aspirating at least one standard after completing analysis of a group of samples. For instruments with built-in memory, enter one to three standards to register a calibration curve for use in subsequent sample analysis.

... Rinse ... water containing 5 ml conc HNO_3 l⁻¹. Aspirate blank and zero instrument. Aspirate sample and determine absorbance.

When determining calcium or magnesium, dilute and mix 100 ml sample with 20 ml lanthanum solution. Add 2 ml H_2O_2 before atomization. When determining iron or manganese, mix 100 ml sample with 5 ml calcium solution. Add 2 ml H_2O_2 before ... When ... of H_2O_2 ...

LEAD

Method 239.2 (Atomic Absorption, furnace technique)

STORET NO. Total 01051
Dissolved 01049
Suspended 01050

Optimum Concentration Range: 5–100 *ug*/l
Detection Limit: 1 *ug*/l

Preparation of Standard Solution

1. Stock solution: Prepare as described under "direct aspiration method".
2. Lanthanum Nitrate Solution: Dissolve 58.64 g of ACS reagent grade La_2O_3 in 100 ml conc. HNO_3 and dilute to 1000 ml with deionized distilled water. 1 ml = 50 mg La.
3. Working Lead Solution: Prepare dilutions of the stock lead solution to be used as calibration standards at the time of analysis. Each calibration standard should contain 0.5% (v/v) HNO_3. To each 100 ml of diluted standard add 10 ml of the lanthanum nitrate solution.

Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% (v/v) HNO_3.
2. To each 100 ml of prepared sample solution add 10 ml of the lanthanum nitrate solution.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–500°C.
3. Atomizing Time and Temp: 10 sec–2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 283.3 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Analysis Procedure

1. For the analysis procedure in the calculation see "Furnace Procedure", part 9.3 of the Atomic Absorption Methods section of this manual.

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Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 *ul* injection, continuous flow purge gas and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.
2. The use of background correction is recommended.
3. Greater sensitivity can be achieved using the 217.0 nm line, but the optimum concentration range is reduced. The use of a lead electrodeless discharge lamp at this lower wavelength has been found to be advantageous. Also a lower atomization temperature (2400°C) may be preferred.
4. To suppress sulfate interference (up to 1500 ppm) lanthanum is added as the nitrate to both samples and calibration standards. (Atomic Absorption Newsletter Vol. 15, No. 3, p 71, May-June 1976.)
5. Since glassware contamination is a severe problem in lead analysis, all glassware should be cleaned immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.
6. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
7. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
8. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
9. Data to be entered into STORET must be reported as *ug*/1.

Precision and Accuracy

1. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 25, 50, and 100 *ug* Pb/1, the standard deviations were ±1.3, ±1.6, and ±3.7, respectively. Recoveries at these levels were 88%, 92%, and 95% respectively.

CADMIUM

Method 213.2 (Atomic Absorption, furnace technique)

STORET NO. 01027
Dissolved 01025
Suspended 01026

Optimum Concentration Range: 0.5–10 ug/l
Detection Limit: 0.1 ug/l

Preparation of Standard Solution

1. Stock solution: Prepare as described under "direct aspiration method".
2. Ammonium Phosphate solution (40%): Dissolve 40 grams of ammonium phosphate, $(NH_4)2HPO_4$ (analytical reagent grade) in deionized distilled water and dilute to 100 ml.
3. Prepare dilutions of the stock cadmium solution to be used as calibration standards at the time of analysis. To each 100 ml of standard and sample alike add 2.0 ml of the ammonium phosphate solution. The calibration standards should be prepared to contain 0.5% (v/v) HNO_3.

Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% (v/v) HNO_3.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–500°C.
3. Atomizing Time and Temp: 10 sec–1900°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 228.8 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Analysis Procedure

1. For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

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Issued 1978

Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injection, continuous flow purge gas and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.
2. The use of background correction is recommended.
3. Contamination from the work area is critical in cadmium analysis. Use of pipet tips which are free of cadmium is of particular importance. (See part 5.5.7 of the Atomic Absorption Methods section of this manual.)
4. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
5. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
6. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
7. Data to be entered into STORET must be reported as ug/1.

Precision and Accuracy

1. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 2.5, 5.0 and 10.0 *ug* Cd/1, the standard deviations were ±0.10, ±0.16 and ±0.33, respectively. Recoveries at these levels were 96%, 99% and 98%, respectively.

MERCURY
Method 245.1 (Manual Cold Vapor Technique)

STORET NO. Total 71900
Dissolved 71890
Suspended 71895

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 In addition to inorganic forms of mercury, organic mercurials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potasssium persulfate has been found to give approximately 100% recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.
   1.3 The range of the method may be varied through instrument and/or recorder expansion. Using a 100 ml sample, a detection limit of 0.2 *ug* Hg/1 can be achieved; concentrations below this level should be reported as < 0.2 (see Appendix 11.2).
2. Summary of Method
   2.1 The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.
3. Sample Handling and Preservation
   3.1 Until more conclusive data are obtained, samples should be preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection. If only dissolved mercury is to be determined, the sample should be filtered through an all glass apparatus before the acid is added. For total mercury the filtration is omitted.
4. Interference
   4.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/1 of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

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4.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/l had no effect on recovery of mercury from spiked samples.

4.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 ml). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 ml). In addition, the dead air space in the BOD bottle must be purged before the addition of stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from sea water using this technique.

4.4 Interference from certain volatile organic materials which will absorb at this wavelength is also possible. A preliminary run without reagents should determine if this type of interference is present (see Appendix 11.1).

5. Apparatus

5.1 Atomic Absorption Spectrophotometer: (See Note 1) Any atomic absorption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

Note 1: Instruments designed specifically for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

5.2 Mercury Hollow Cathode Lamp: Westinghouse WL-22847, argon filled, or equivalent.

5.3 Recorder: Any multi-range variable speed recorder that is compatible with the UV detection system is suitable.

5.4 Absorption Cell: Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1" O.D. X 4-1/2". The ends are ground perpendicular to the longitudinal axis and quartz windows (1" diameter X 1/16" thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2" by 2" cards. One inch diameter holes are cut in the middle of each card; the cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

5.5 Air Pump: Any peristaltic pump capable of delivering 1 liter of air per minute may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

5.6 Flowmeter: Capable of measuring an air flow of 1 liter per minute.

5.7 Aeration Tubing: A straight glass frit having a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

5.8 Drying Tube: 6" X 3/4" diameter tube containing 20 g of magnesium perchlorate (see Note 2). The apparatus is assembled as shown in Figure 1.

NOTE 2: In place of the magnesium perchlorate drying tube, a small reading lamp with 60W bulb may be used to prevent condensation of moisture inside the cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10°C above ambient.

6. Reagents

6.1 Sulfuric Acid, Conc.: Reagent grade.

6.1.1 Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1.0 liter.

6.2 Nitric Acid, Conc: Reagent grade of low mercury content (See Note 3).

NOTE 3: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

6.3 Stannous Sulfate: Add 25 g stannous sulfate to 250 ml of 0.5 N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

6.4 Sodium Chloride-Hydroxylamine Sulfate Solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in distilled water and dilute to 100 ml. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

6.5 Potassium Permanganate: 5% solution, w/v. Dissolve 5 g of potassium permanganate in 100 ml of distilled water.

6.6 Potassium Persulfate: 5% solution, w/v. Dissolve 5 g of potassium persulfate in 100 ml of distilled water.

6.7 Stock Mercury Solution: Dissolve 0.1354 g of mercuric chloride in 75 ml of distilled water. Add 10 ml of conc. nitric acid and adjust the volume to 100.0 ml. 1 ml = 1 mg Hg.

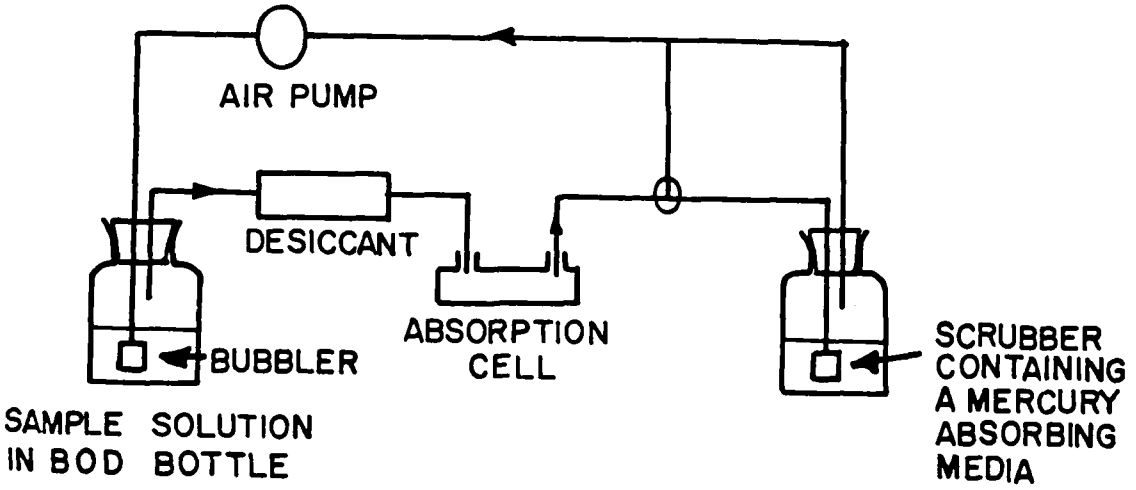


FIGURE 1. APPARATUS FOR FLAMELESS MERCURY DETERMINATION

6.8 Working Mercury Solution: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 *u*g per ml. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before the addition of the aliquot.

7. Calibration

7.1 Transfer 0, 0.5, 1.0, 2.0, 5.0 and 10.0 ml aliquots of the working mercury solution containing 0 to 1.0 *u*g of mercury to a series of 300 ml **BOD** bottles. Add enough distilled water to each bottle to make a total volume of 100 ml. Mix thoroughly and add 5 ml of conc. sulfuric acid (6.1) and 2.5 ml of conc. nitric acid (6.2) to each bottle. Add 15 ml of $KMnO_4$ (6.5) solution to each bottle and allow to stand at least 15 minutes. Add 8 ml of potassium persulfate (6.6) to each bottle and heat for 2 hours in a water bath maintined at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution (6.4) to reduce the excess permanganate. When the solution has been decolorized wait 30 seconds, add 5 ml of the stannous sulfate solution (6.3) and immediately attach the bottle to the aeration apparatus forming a closed system. At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter per minute, is allowed to run continuously (See Note 4). The absorbance will increase and reach maximum within 30 seconds. As soon as the recorder pen levels off, approximately 1 minute, open the bypass valve and continue the aeration until the absorbance returns to its minimum value (see Note 5). Close the bypass valve, remove the stopper and frit from the **BOD** bottle and continue the aeration. Proceed with the standards and construct a standard curve by plotting peak height versus micrograms of mercury.

NOTE 4: An open system where the mercury vapor is passed through the absorption cell only once may be used instead of the closed system.

NOTE 5: Because of the toxic nature of mercury vapor precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as:

a) equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4
b) 0.25% iodine in a 3% KI solution

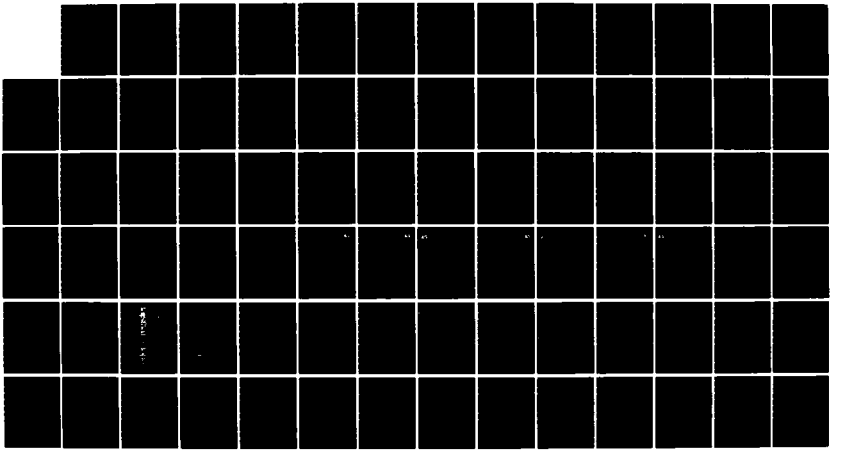
A specially treated charcoal that will adsorb mercury vapor is also available from Barnebey and Cheney, E. 8th Ave. and N. Cassidy St., Columbus, Ohio 43219, Cat. #580–13 or #580–22.

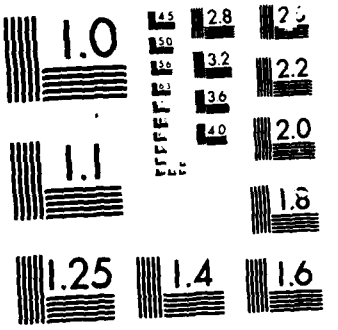
8. Procedure

8.1 Transfer 100 ml, or an aliquot diluted to 100 ml, containing not more than 1.0 *u*g of mercury, to a 300 ml **BOD** bottle. Add 5 ml of sulfuric acid (6.1) and 2.5 ml of conc. nitric acid (6.2) mixing after each addition. Add 15 ml of potassium permanganate solution (6.5) to each sample bottle. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Add 8 ml of potassium persulfate (6.6) to each bottle and heat for 2 hours in a water bath at 95°C. Cool and add 6

AD-A165 514 INSTALLATION RESTORATION PROGRAM FINAL REPORT PHASE II 5/5
STAGE 1 - PROBLEM..(U) WESTON (ROY F) INC WEST CHESTER
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MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

ml of sodium chloride-hydroxylamine sulfate (6.4) to reduce the excess permanganate. After a delay of at least 30 seconds add 5 ml of stannous sulfate (6.3) and immediately attach the bottle to the aeration apparatus. Continue as described under Calibration.

9. Calculation

9.1 Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

9.2 Calculate the mercury concentration in the sample by the formula:

$$\mu g\ Hg/l = \left(\begin{matrix}\mu g\ Hg\ in \\ aliquot\end{matrix}\right)\left(\frac{1{,}000}{\text{volume of aliquot in ml}}\right)$$

9.3 Report mercury concentrations as follows: Below 0.2 *ug*/1, <0.2; between 1 and 10 *ug*/1, one decimal; above 10 *ug*/1, whole numbers.

10. Precision and Accuracy

10.1 In a single laboratory (EMSL), using an Ohio River composite sample with a background mercury concentration of 0.35 *ug*/1, spiked with concentrations of 1.0, 3.0 and 4.0 *ug*/1, the standard deviations were ±0.14, ±0.10 and ±0.08, respectively. Standard deviation at the 0.35 level was ±0.16. Percent recoveries at the three levels were 89, 87, and 87%, respectively.

10.2 In a joint EPA/ASTM interlaboratory study of the cold vapor technique for total mercury in water, increments of organic and inorganic mercury were added to natural waters. Recoveries were determined by difference. A statistical summary of this study follows:

Number of Labs	True Values *ug*/liter	Mean Value *ug*/liter	Standard Deviation *ug*/liter	Accuracy as % Bias
76	0.21	0.349	0.276	66
80	0.27	0.414	0.279	53
82	0.51	0.674	0.541	32
77	0.60	0.709	0.390	18
82	3.4	3.41	1.49	0.34
79	4.1	3.81	1.12	-7.1
79	8.8	8.77	3.69	-0.4
78	9.6	9.10	3.57	-5.2

11. Appendix

11.1 While the possibility of absorption from certain organic substances actually being present in the sample does exist, EMSL has not encountered such samples. This is mentioned only to caution the analyst of the possibility. A simple correction that may be used is as follows: If an interference has been found to be present (4.4), the sample should be analyzed both by using the regular procedure and again under oxidizing conditions only,

that is without the reducing reagents. The true mercury value can then be obtained by subtracting the two values.

11.2 If additional sensitivity is required, a 200 ml sample with recorder expansion may be used provided the instrument does not produce undue noise. Using a Coleman MAS-50 with a drying tube of magnesium perchlorate and a variable recorder, 2 mv was set to read full scale. With these conditions, and distilled water solutions of mercuric chloride at concentrations of 0.15, 0.10, 0.05 and 0.025 *ug/l* the standard deviations were ±0.027, ±0.006, ±0.01 and ±0.004. Percent recoveries at these levels were 107, 83, 84 and 96%, respectively.

11.3 Directions for the disposal of mercury-containing wastes are given in ASTM Standards, Part 31, "Water", p 349, Method D3223 (1976).

Bibliography

1. Kopp, J. F., Longbottom, M. C. and Lobring, L. B., "Cold Vapor Method for Determining Mercury", AWWA, vol 64, p. 20, Jan., 1972.
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D3223–73, p 343 (1976).
3. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 156 (1975).

FISH TISSUE PREPARATION AND ANALYTICAL METHODS

BY_____

CHKD BY ___

PROJECT ___

SUBJECT ___

Ashing Procedure Ni Cd Pb Cu Zn Cr

Weigh 25-35 gm of fish tissue into a clean acid-washed 125-ml beaker. Store remainder in freezer.

Dry in drying oven at 103°C. for approximately 48 hrs.

Weigh to constant weight. Calculate wet to dry ratio.

Add 20-25 ml concentrated nitric acid to each sample or standard, cover and digest on a hot plate. Do not allow to go to dryness or to char. Add additional nitric acid as necessary to maintain oxidizing conditions. Ash to a clear solution.

Transfer the ashed solution to a 50-ml centrifuge tube using 2% nitric acid.

Centrifuge, and pour supernatant into an acid-washed 50-ml volumetric flask. Rinse precipitate three times with 2% nitric acid and add rinsings to flask. Dilute to volume with distilled water. Discard precipitate.

Samples were run on Inductively Coupled Plasma (ICP).

Ashing Procedure

As

Weigh 10-gm of blended wet tissue into a clean, sulfuric acid-washed 125-ml Phillips beaker.

Add 10 ml sulfuric acid and 25 ml nitric acid to each sample or standard. Cover and digest on a hotplate at moderate heat. If the sample starts to darken or char, additional nitric acid in (5-ml) must be added to prevent loss of metals as oxides or chlorides.

As the solution evaporates, add nitric acid in 5-ml portions until all organic matter is destroyed. This point is reached when no darkening of the solution occurs on continued heating after the production of a clear, colorless solution and copious fumes of sulfur trioxide. Cool.

Cautiously rinse sides of beaker with several small portions of about 20 ml of distilled water.

Add 10 ml of a saturated solution of ammonium oxalate to aid in the expulsion of nitrogen oxide fumes.

Evaporate to the appearance of sulfur trioxide fumes. Cool.

Using distilled water, cautiously rinse sides of flask with about 25-ml of distilled water and again evaporate to the appearance of sulfur trioxide fumes. Cool.

Dilute to about 50ml with distilled water.

Samples were run on Atomic Absorption Furnace (A.A.) (E.P.A. Method 206.2)

BY
CH
PF
SU

Ashing Procedure Hg

Weigh approximately 1.0 gram of homogenized fish tissue into a 300 ml BOD bottle.

Add 5.0 ml of concentrated H_2SO_4 and 3.0 ml of concentrated HNO_3 to the BOD bottles and allow to digest overnight (or incubate at 50°C, until a clear solution is obtained.

Add 5.0 ml of potassium permanganite (20% solution) and 3.0 ml of potassium persulfate (3% solution) and incubate at 55°C. for two hours. Check samples for depletion of permanganate. Add more if needed.

Allow solution to cool then proceed to Figure 10, analytical procedures for Hg.

Analytical Procedure (Mercury)

- Add 5.0 ml of Hydroxylamine (HCl) (1.5%) to the cooled clear liquid obtained in the ashing procedure to reduce any remaining permanganate.
- After five minutes, add 5.0 ml of stannous chloride (10%) to the sample and place the BOD bottle onto the mercury vapor apparatus (see reference 5).
- Measure the absorbance of mercury vapor by flameless atomic absorption spectrophotometry.

FISH TISSUE PREPARATION AND ANALYTICAL METHODS

BY_____

CHKD BY ____

PROJECT ____

SUBJECT ____

Ashing Procedure Ni Cd Pb Cu Zn Cr

Weigh 25-35 gm of fish tissue into a clean acid-washed 125-ml beaker. Store remainder in freezer.

Dry in drying oven at 103°C. for approximately 48 hrs.

Weigh to constant weight. Calculate wet to dry ratio.

Add 20-25 ml concentrated nitric acid to each sample or standard, cover and digest on a hot plate. Do not allow to go to dryness or to char. Add additional nitric acid as necessary to maintain oxidizing conditions. Ash to a clear solution.

Transfer the ashed solution to a 50-ml centrifuge tube using 2% nitric acid.

Centrifuge, and pour supernatant into an acid-washed 50-ml volumetric flask. Rinse precipitate three times with 2% nitric acid and add rinsings to flask. Dilute to volume with distilled water. Discard precipitate.

Samples were run on Inductively Coupled Plasma (ICP).

B
C
P
S

Ashing Procedure

As

Weigh 10-gm of blended wet tissue into a clean, sulfuric acid-washed 125-ml Phillips beaker.

Add 10 ml sulfuric acid and 25 ml nitric acid to each sample or standard. Cover and digest on a hotplate at moderate heat. If the sample starts to darken or char, additional nitric acid in (5-ml) must be added to prevent loss of metals as oxides or chlorides.

As the solution evaporates, add nitric acid in 5-ml portions until all organic matter is destroyed. This point is reached when no darkening of the solution occurs on continued heating after the production of a clear, colorless solution and copious fumes of sulfur trioxide. Cool.

Cautiously rinse sides of beaker with several small portions of about 20 ml of distilled water.

Add 10 ml of a saturated solution of ammonium oxalate to aid in the expulsion of nitrogen oxide fumes.

Evaporate to the appearance of sulfur trioxide fumes. Cool.

Using distilled water, cautiously rinse sides of flask with about 25-ml of distilled water and again evaporate to the appearance of sulfur trioxide fumes. Cool.

Dilute to about 50ml with distilled water.

Samples were run on Atomic Absorption Furnace (A.A)
(E.P.A. Method 206.2)

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Allow solution to cool then proceed to Figure 10, analytical procedures for Hg.

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- Measure the absorbance of mercury vapor by flameless atomic absorption spectrophotometry.

APPENDIX J

LABORATORY QA/QC PLAN

WESTON

APPENDIX J

LABORATORY QA/QC PLAN

J-1 QUALITY ASSURANCE PLAN

WESTON Analytical Serives enforces a rigid QA/QC program toward maintenace of validity and reliability of all analytical data. The Laboratory QA/QC Manual (Table of Contents thereof is Attachment No. 1 to this appendix) outlines the specifics of the QA/QC plan. This plan is patterned after the EPA Handbook for Analytical Quality Control in Waste and Wastewater Laboratories (EPA-600/4-79-019, March 1979), augmented by general applicable experience and interaction with the QZ/AC plan of the U. S. Army Toxic and Hazardous Materials Agency (USATHAMA). All methods and procedures followed by WESTON are either USEPA or ASTM-approved. Any variations from such procedures, regarless of cause, are documented by the responsible analyst(s) and are documentable, and, literature-traceable. A general review of this QA/QC plan is in the following paragraphs.

Although specific QA/QC measures for each method are designated in WESTON's Laboratory Quality Assurance Manual, the general QA/QC program normally includes:

- EPA-acceptable sample preparation and analytical methods.
- Instrument calibration via use of Standard Analytical Reference Materials (SARMS).
- Regular equipment maintenance and servicing.
- Use of SARMS and QA/QC samples (spikes, laboratory blanks, replicates, and splits) to ascertain overall precision.
- Statistical evaluation of data to delineate acceptable limits.
- Documentation of system/operator performance.
- Suitable chain-of-custody procedures.
- Maintenance and archiving of all records, charts, and logs generated in the above.
- Proper reporting.

Acceptable analyses at WESTON's Analytical Laboratory Services include, but are not limited to, the above.

In general, WESTON's QA/QC sequence follows the following diagram (Figure J-1). Documentation (as available from instrument recordings and technicians' notebooks) is sufficient to validate each step in the sequence.

J.2 CONTAINER PREPARATION

Another consideration in this, or any, analytical project is that of sample container preparation. Accordingly, all appropriate sample bottles shall be cleaned in a manner mandated by the U.S. EPA to insure maximal cleanliness (and minimal contamination) before the containers go to the field. Sufficient bottles to accommodate both laboratory and field blank requirements will be preferred in a single batch mode for each monthly sampling requirement.

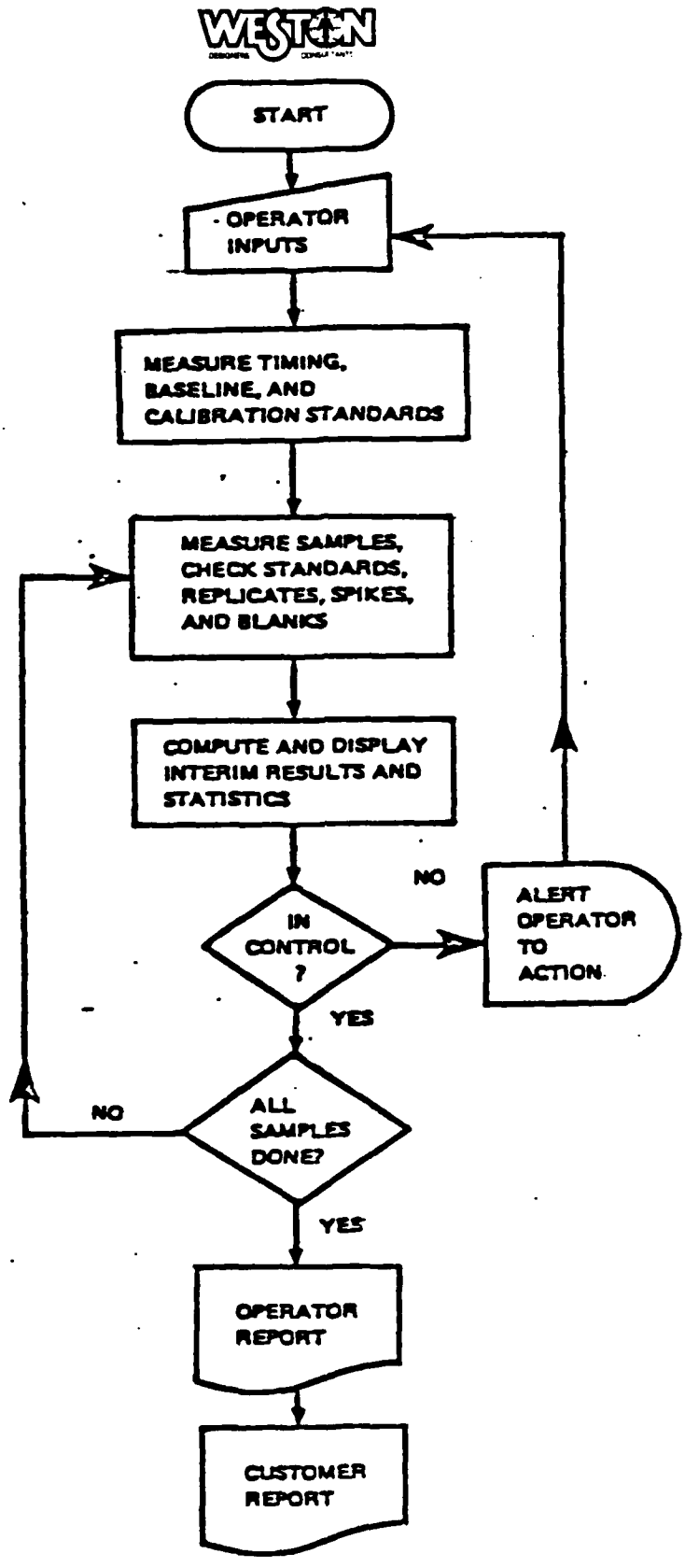


Figure J-1: Flow Chart of the Sequence of Events during a Controlled Series of Laboratory Measurements.

J.3 VERIFICATION/VALIDATION

In the laboratory, the analytical scheme begins with initial verification, which is comprised of:

- Lab Blanks - To insure that no background level of specific analytes is introduced by laboratory procedures.
- Standard Analytical Reference Materials (SARMS) - To determine the accuracy and precision of procedures.
- Spikes - To determine the percent recovery of analyte(s).

If the laboratory QA/QC program is extended to the field, it includes a fifth item:

- Field Blanks - To provide a check on contamination of containers and/or preservatives and to establish "practical" detection limits.

WESTON has used all of the above in this project. All data resulting from these verification media have been archived for future reference, retrieval, or processing. (QA/QC data from WESTON's above-described, internal QA/QC plan normally are not available to clientele without associated reimbursement to WESTON).

J.4 DATA HANDLING - LABORATORY

Use of any analytical data should be preceded by an assessment of its quality. The assessment should be based on accuracy, precision, completeness, representativeness, and comparability. These criteria are, in turn, assessed as follows:

- Accuracy - Is it acceptable for the planned use? QA/QC shall measure the accuracy of all data.

- Precision - Is it acceptable for the planned use? QA/QC shall reflect the reproducibility of the measurements.

- Completeness - Are the data sufficient for the planned use? QA/QC shall identify the quantity of data needed to match the goals.

- Representativeness -Do the data accurately reflect actual site conditions, sampling procedures, and analytical method? QA/QC shall ensure this.

- Comparability - Is the report self-consistent in format, units, and standardization of methods used to generate it? QA/QC shall ensure this.

Additionally, statistical methods outlined in the QA/QC program have been applicable to data evaluation.

The Laboratory Supervisor and the Laboratory QA/QC Officer have been responsible for the evaluation of the above criteria and for enforcement of analytical protocols that will necessarily lead to acceptable data quality. The signature of the Supervisor and QA/QC Officer accompany each laboratory analytical report and serve to ensure the overall validity of the reported data.

J.5 SAMPLE PLAN/LOG

Normal protocol demands client-and /or site-specific logging of all sample batches delivered to WESTON. Basic information -- such as client name, address, etc.; client phone number; reporting/invoicing instructions; site descriptions; and parameter-specifications and total requirements -- is initiated here. Additionally, sample storage/disposal instructions as well as turnaround requirements and sample collection requirements are addressed at this point.

The appropriate number of method blanks is also logged at this point, and in-house chain-of-custody documentation is initiated here.

J.6 SAMPLE RESULTS

WESTON's analytical protocols generally require five-point calibration curve plus a reagent blank s the basis for

quantification analytes from a linear calibration curve. (A three-point plus blank curve vs. the original five point one is acceptable if it falls within the QA/QC requirements of ± 3 standard deviation of the original curve.) Linear regression analysis is then performed. Method- and detection limit-specific data are accessed for quantitation and report-writing from each such data set. For reporting accuracy, the algorithm

$$\frac{\text{Linear-Regressed Raw Concentration from Calibration Curve} \times \text{Solid Sample Extract Volume If Solid}}{\text{Solid Sample Mass If Solid} \times \text{Fraction Solids If Solid}} \times \text{Concentration or Dilution Factor} = \text{Final Concentration}$$

is used for all quantitations. (All such algorithm input data are archived for long-term storage.) Detection limits for solids are generated on a per-sample basis and calculated by replacing "LINEAR-REGRESSED RAW CONCENTRATION FROM CALIBRATION CURVE" with "DETECTION LIMIT OF ANALYTE IN LIQUID MATRIX" in the above equation.

J.7 CHAIN-OF-CUSTODY

Since they document the history of samples, chain-of-custody procedures are a crucial part of a sampling/analysis program. Chain-of-custody documentation enables identification and tracking of a sample from collection to analysis to reporting.

WESTON's chain-of-custody program necessitates the use of EPA-approved sample labels, secure custody, and attendant recordkeeping. Depending on the client's requirements, WESTON also offers container sealing during unattended transportation of samples.

In essence, WESTON considers a sample in custody if it: is in a WESTON employee's physical possession; it is in view of that WESTON employee; is secured by that WESTON employee to prevent tampering; or is secured by that WESTON employee in an area that is restricted to authorized personnel.

Each time a sample is relinquished from one analyst to another or from one major location to another, WESTON's analytical personnel are required to make appropriate entries. Personnel-specific initials are used as identifiers of analysts, as are location codes for various locations (refrigerators, extraction areas, analytical areas, etc.)

within the laboratory. Each transaction for each sample is accompanied by a specific reason for transfer. Chain-of-custody documentation is given in Appendix F.

J.8 QA/QC OFFICER

Toward maintenance of a rigid, credible QA/QC regimen, WESTON Analytical Services maintains a full-time, in-house QA/QC officer who retains independent authority to declare out-of-control situations, thereby precluding reporting of unacceptable data. The QA/QC officer has been available, as needed, on the project.

ATTACHMENT 1

LABORATORY QUALITY ASSURANCE MANUAL

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APPENDIX K

LABORATORY ANALYTICAL REPORTS -

RAW CHEMICAL DATA

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
	B1-1 0094	0	NO PEAKS FOUND	
	B1-1 1037	0	NO PEAKS FOUND	
	B1-2 0095	0	BROMODICHLOROMETHANE	1.0
	B1-2 0095	0	NO PEAKS FOUND	
	B1-2 1038	0	TRICHLOROETHENE	1.7
			TETRACHLOROETHENE	1.2
			BENZENE	1100
			TOLUENE	1300
			ETHYLBENZENE	10000
	B1-2 1038	0	BENZENE	550
			TOLUENE	1300
			ETHYLBENZENE	10000
	B1-3 0096	0	NO PEAKS FOUND	
	B1-3 1039	0	NO PEAKS FOUND	
	B1-A4 1000	0	1,1-DICHLOROETHANE	3.3
			TRANS-1,2-DICHLOROETHENE	11
			CHLOROFORM	1.7
			1,2-DICHLOROETHANE	5.3
	B1-A5 1001	0	1,1-DICHLOROETHANE	7.0
			TRANS-1,2-DICHLOROETHENE	3.0
			CHLOROFORM	4.5
			1,2-DICHLOROETHANE	8.2
	B2-1 1040	0	1,4-DICHLOROBENZENE	4.0
			TOLUENE	410
	B2-2 1041	0	1,2-DICHLOROETHANE	2.5
	B2-2 1041	0	TOLUENE	83
	B2-3 1004	0	NO PEAKS FOUND	
	B2-4 1005	0	NO PEAKS FOUND	
	B2-4 1005	0	NO PEAKS FOUND	
	B2-5 1006	0	NO PEAKS FOUND	
	B2-6 1007	0	NO PEAKS FOUND	
	B2-7 1008	0	CARBON TETRACHLORIDE	1.4
	B2-B1 1042	0	BENZENE	1000

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
	B2-B1 1042	0	TOLUENE	1200
			ETHYLBENZENE	8000
	B3-2 1044	0	TOLUENE	410
	B3-3 1011	0	NO PEAKS FOUND	
	B3-3 1011	0	NO PEAKS FOUND	
	B3-3 1045	0	NO PEAKS FOUND	
	B3-3 1045	0	NO PEAKS FOUND	
	B3-4 1012	0	NO PEAKS FOUND	
	B3-5 1013	0	NO PEAKS FOUND	
	B3-6 1014	0	NO PEAKS FOUND	
	B3-6 1014	0	NO PEAKS FOUND	
	B3-B1 1015	0	NO PEAKS FOUND	
	B3-B1 1046	0	METHYLENE CHLORIDE	3.0
	B4-1 1047	0	1,1-DICHLOROETHANE	17
			TOLUENE	340
	B4-2 1048	0	METHYLENE CHLORIDE	80
			1,1-DICHLOROETHENE	11
			TRANS-1,2-DICHLOROETHENE	3.3
			CHLOROFORM	1.7
			1,2-DICHLOROETHANE	5.3
	B4-2 1048	0	CHLOROFORM	150
			1,2-DICHLOROETHANE	90
			TETRACHLOROETHENE	6.0
	B4-3 1018	0	METHYLENE CHLORIDE	2.0
	B4-3 1049	0	TOLUENE	190
	B4-4 1019	0	METHYLENE CHLORIDE	4.0
	B4-4 1019	0	METHYLENE CHLORIDE	2.0
	B4-4 1050	0	TETRACHLOROETHENE	9.3
			1,3-DICHLOROBENZENE	3.9
	B4-4 1050	0	TETRACHLOROETHENE	4.3
			CHLOROBENZENE	51
			1,2-DICHLOROBENZENE	23

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
	B4-5 1020	0	METHYLENE CHLORIDE	12
	B4-6 1021	0	NO PEAKS FOUND	
	B4-7 1022	0	METHYLENE CHLORIDE	6.7
	B5-1 1051	0	1,1-DICHLOROETHENE	19
			1,1-DICHLOROETHANE	2.8
			1,2-DICHLOROETHANE	11
			TRICHLOROETHENE	2.8
			TETRACHLOROETHENE	2.0
	B5-1 1051	0	TETRACHLOROETHENE	2.0
	B5-3 1025	0	NO PEAKS FOUND	
	B5-3 1025	0	NO PEAKS FOUND	
	B5-3 1053	0	TETRACHLOROETHENE	2.7
	B5-3 1053		TETRACHLOROETHENE	2.7
	B5-4 1026	6	METHYLENE CHLORIDE	16
			CHLOROFORM	0.63
			CHLOROBENZENE	970
			1,2-DICHLOROBENZENE	1800
			1,4-DICHLOROBENZENE	1800
			TOLUENE	600
			ETHYLBENZENE	250
	B5-5 1027	0	1,2-DICHLOROBENZENE	1900
			1,3-DICHLOROBENZENE	61000
			1,4-DICHLOROBENZENE	15000
	B5-5 1027	0	1,2-DICHLOROBENZENE	970
			1,3-DICHLOROBENZENE	41000
			1,4-DICHLOROBENZENE	10000
	B5-6 1028	2	METHYLENE CHLORIDE	24000
			CHLOROFORM	142
			1,2-DICHLOROETHANE	9700
			TRICHLOROETHENE	98
			TETRACHLOROETHENE	70
			CHLOROBENZENE	3500000
			1,2-DICHLOROBENZENE	15000
			1,3-DICHLOROBENZENE	24000
			1,4-DICHLOROBENZENE	17000
	B5-6 1028	3	METHYLENE CHLORIDE	12000
			CHLOROFORM	277

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
	B5-6 1028	3	1,2-DICHLOROETHANE	11600
			TRICHLOROETHENE	110
			TETRACHLOROETHENE	69
			CHLOROBENZENE	3500000
			1,2-DICHLOROBENZENE	15000
			1,3-DICHLOROBENZENE	24000
			1,4-DICHLOROBENZENE	17000
	B5-7 1029	0	METHYLENE CHLORIDE	27000
			1,2-DICHLOROBENZENE	35000
			1,3-DICHLOROBENZENE	60000
			1,4-DICHLOROBENZENE	45000
	B5-7 1029	0	METHYLENE CHLORIDE	27000
			1,2-DICHLOROBENZENE	37000
			1,3-DICHLOROBENZENE	70000
			1,4-DICHLOROBENZENE	56000
	B6-1 1030	0	1,2-DICHLOROBENZENE	37000
			1,3-DICHLOROBENZENE	76000
			1,4-DICHLOROBENZENE	56000
	B6-1 1055	0	NO PEAKS FOUND	
	B6-2 1056	1	TRANS-1,2-DICHLOROETHENE	50
			CHLOROFORM	6.0
			1,2-DICHLOROETHANE	30
			TRICHLOROETHENE	77
			TETRACHLOROETHENE	20
	B6-2 1056	0	TRICHLOROETHENE	46
			TETRACHLOROETHENE	13
	B6-2 1056	1	TRANS-1,2-DICHLOROETHENE	50
			CHLOROFORM	6.0
			1,2-DICHLOROETHANE	30
			TRICHLOROETHENE	77
			TETRACHLOROETHENE	20
	B6-3 1057	0	TOLUENE	630
	B6-4 1058	0	TOLUENE	83
	B6-B1 1032	0	BENZENE	390
	B6-B2 1033	0	1,2-DICHLOROBENZENE	10%
			1,3-DICHLOROBENZENE	10%
			1,4-DICHLOROBENZENE	10%
	B6-B3 1034	0	1,4-DICHLOROBENZENE	3700
	B6-B4 1035	0	METHYLENE CHLORIDE	3.7

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
	B6-B4 1035	0	TOLUENE	97
	BLANK 1/11	0	NO PEAKS FOUND	
	BLANK 2/6	0	METHYLENE CHLORIDE	8.5
	BLANK 2/7	0	NO PEAKS FOUND	
	BLANK 2/8	0	METHYLENE CHLORIDE	12
	BLANK 2/9	0	METHYLENE CHLORIDE	10
	BLANK 4/10	0	1,2-DICHLOROETHANE	2.0
	BLANK 4/19	0	NO PEAKS FOUND	
	BLANK 6/11	0	NO PEAKS FOUND	

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	COMMENTS
B1-2 1038	UNKNOWN HYDROCARBON MIXTURE PRESENT
B2-1 1040	UNKNOWN HYDROCARBON MIXTURE PRESENT
B2-2 1041	UNKNOWN HYDROCARBON MIXTURE PRESENT
B2-B1 1042	UNKNOWN HYDROCARBON MIXTURE PRESENT
B3-2 1044	UNKNOWN HYDROCARBON MIXTURE PRESENT
B4-1 1047	UNKNOWN HYDROCARBON MIXTURE PRESENT
B4-3 1049	UNKNOWN HYDROCARBON MIXTURE PRESENT
B5-4 1026	UNKNOWN HYDROCARBON MIXTURE PRESENT
B5-4 1040	LOST SAMPLE
B6-1 1055	UNKNOWN HYDROCARBON MIXTURE PRESENT
B6-3 1057	UNKNOWN HYDROCARBON MIXTURE PRESENT
B6-4 1058	UNKNOWN HYDROCARBON MIXTURE PRESENT
B6-B1 1032	UNKNOWN HYDROCARBON MIXTURE PRESENT
B6-B4 1035	UNKNOWN HYDROCARBON MIXTURE PRESENT

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR WATERS
EPA METHOD 601/602

B ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/L)
[illegible]699	MW-1	3	METHYLENE CHLORIDE	3.0
S-0707	MW-2	0	METHYLENE CHLORIDE	0.98
			TRANS-1,2-DICHLOROETHENE	0.53
			1,2-DICHLOROETHANE	0.12
			1,2-DICHLOROPROPANE	0.52
			TRICHLOROETHENE	1.1
[illegible]691	MW-3	0	METHYLENE CHLORIDE	3.0
			1,1-DICHLOROETHENE	0.30
			CHLOROFORM	1.8
S-0716	MW-4	0	METHYLENE CHLORIDE	0.90
			TRICHLOROETHENE	0.70
S-0709	MW-5	0	METHYLENE CHLORIDE	3.7
			CHLOROFORM	26
[illegible]709	MW-5	0	METHYLENE CHLORIDE	0.81
			CHLOROFORM	1.0
[illegible]712	MW-6	1	CHLOROFORM	30
S-0712	MW-6	1	METHYLENE CHLORIDE	0.80
			CHLOROFORM	1.0
S-0746	MW-7	3	METHYLENE CHLORIDE	2.0
			TRICHLOROETHENE	0.59
S-0714	MW-8	0	METHYLENE CHLORIDE	1.4
			CHLOROFORM	0.05
			TETRACHLOROETHENE	0.04
[illegible]718	MW-9	2	METHYLENE CHLORIDE	0.56
S-0720	MW-10	2	METHYLENE CHLORIDE	1.1
			CHLOROFORM	0.07
			1,1,1-TRICHLOROETHANE	1.1
			TRICHLOROETHENE	40
			TETRACHLOROETHENE	0.13
[illegible]722	MW-11	2	METHYLENE CHLORIDE	1.8
			1,1,1-TRICHLOROETHANE	0.10
			TETRACHLOROETHENE	5.0
S-0724	MW-12	2	METHYLENE CHLORIDE	1.5
			TETRACHLOROETHENE	0.93
[illegible]0726	MW-13	1	METHYLENE CHLORIDE	1.0
			TETRACHLOROETHENE	2.0
[illegible]0728	MW-14	2	METHYLENE CHLORIDE	3.4

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR WATERS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/L)
S-0728	MW-14	2	TRANS-1,2-DICHLOROETHENE	3.0
			TRICHLOROETHENE	230
			METHYL ETHYL KETONE	12
S-0730	MW-15	2	CHLOROETHANE	3.0
			METHYLENE CHLORIDE	2.0
			1,1-DICHLOROETHENE	1.5
			1,1-DICHLOROETHANE	0.71
			TRANS-1,2-DICHLOROETHENE	420
			TRICHLOROETHENE	1000
			TETRACHLOROETHENE	0.10
			BENZENE	65
			TOLUENE	640
			ETHYLBENZENE	41
			METHYL ETHYL KETONE	43
S-0732	MW-16	5	VINYL CHLORIDE	96
			METHYLENE CHLORIDE	2.7
			1,1-DICHLOROETHENE	0.60
			1,1-DICHLOROETHANE	0.36
			TRANS-1,2-DICHLOROETHENE	200
			CHLOROBENZENE	3.0
			1,4-DICHLOROBENZENE	3.0
S-0734	MW-17	5	VINYL CHLORIDE	310
			METHYLENE CHLORIDE	3.0
			TRANS-1,2-DICHLOROETHENE	22
			CHLOROBENZENE	1.1
			1,4-DICHLOROBENZENE	2.9
S-0736	MW-18	3	VINYL CHLORIDE	450
			METHYLENE CHLORIDE	1.6
			TRANS-1,2-DICHLOROETHENE	11
			CHLOROBENZENE	0.50
			1,4-DICHLOROBENZENE	1.4
S-0738	MW-19	2	METHYLENE CHLORIDE	2.1
			TRANS-1,2-DICHLOROETHENE	1.2
			CHLOROFORM	27
			TRICHLOROETHENE	0.56
			TETRACHLOROETHENE	0.50
			1,4-DICHLOROBENZENE	0.52
S-0738	MW-19	1	METHYLENE CHLORIDE	1.1
			TRANS-1,2-DICHLOROETHENE	1.3
			CHLOROFORM	1.3
			1,4-DICHLOROBENZENE	0.53
S-0703	MW-20	0	METHYLENE CHLORIDE	2.2
			CHLOROFORM	30
S-0705	MW-21	0	METHYLENE CHLORIDE	1.1

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR WATERS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/L)
-0705	MW-21	0	CHLOROFORM	2.6
			TRICHLOROETHENE	0.16
			TETRACHLOROETHENE	0.04
-0740	MW-22	2	METHYLENE CHLORIDE	2.3
S-0742	MW-23	2	METHYLENE CHLORIDE	2.5
			TRICHLOROETHENE	1.1
			TETRACHLOROETHENE	4.9
-0744	MW-25	6	VINYL CHLORIDE	130
			METHYLENE CHLORIDE	3.9
			TRANS-1,2-DICHLOROETHENE	149
			TRICHLOROETHENE	0.24
			CHLOROBENZENE	2.9
			1,4-DICHLOROBENZENE	5.4
-0687	POND 1-W	3	METHYLENE CHLORIDE	1.8
			CHLOROFORM	0.95
			TRICHLOROETHENE	0.13
S-0695	POND 2-W	1	METHYLENE CHLORIDE	2.5
			CHLOROFORM	1.3
-0697	POND 3-W	1	METHYLENE CHLORIDE	0.74
			CHLOROFORM	0.83
S-0701	FB-1	1	METHYLENE CHLORIDE	1.0
			CHLOROFORM	1.2
			TRICHLOROETHENE	0.69
	BLANK 7/13	0	METHYLENE CHLORIDE	3.0
			CHLOROFORM	0.09
	BLANK 7/16	1	METHYLENE CHLORIDE	1.9
S-0625	BLANK 7/13	1	METHYLENE CHLORIDE	2.2
S-0637	BLANK 7/16	2	METHYLENE CHLORIDE	0.60
S-0626	BLANK 7/16	1	METHYLENE CHLORIDE	2.5
S-0627	BLANK 7/17	3	METHYLENE CHLORIDE	4.8
S-0635	BLANK 7/17	2	NO PEAKS FOUND	
S-0630	BLANK 7/18	0	METHYLENE CHLORIDE	1.2
			CHLOROFORM	22
S-0633	BLANK 7/18	0	METHYLENE CHLORIDE	0.99
			CHLOROFORM	1.3

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR WATERS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/L)
S-0646	BLANK 7/18	1	METHYLENE CHLORIDE	0.75
			CHLOROFORM	1.3

Analyst's Notes:

(1) Subtract 1.8 ppb from methylene chloride results to account for in-house contamination.

(2) In-house contamination with Freon (from oil and grease extractions) is thought to be responsible for reported values of chloroform in the 20-30 ug/l range. Do not report these values.

(3) Second column confirmations were run on the nine samples exhibiting significant levels of VOA and levels reported have been confirmed.

WESTON

EPA METHOD 601 HALL DETECTOR
EPA METHOD 602 FID DETECTOR

COMPOUND	DETECTION LIMIT ug/l
Chloromethane	0.08
Bromomethane	1.18
Dichlorodifluromethane	1.81
Vinyl chloride	0.18
Chloroethane	0.52
Methylene chloride	0.25
Trichlorofluoromethane	1.0
1,1 Dichloroethene	0.13
1,1 Dichloroethane	0.07
Trans 1,2 Dichloroethene	0.10
Chloroform	0.05
1,2 Dichloroethane	0.03
1,1,1-Trichloroethane	0.03
Carbon tetrachloride	0.12
Bromodichloromethane	0.10
1,2 Dichloropropane	0.04
Trans 1,3-Dichloropropene	0.34
Trichloroethene	0.12
Dibromochloromethane	0.09
1,1,2 Trichloroethane	0.02
Cis 1,3-Dichloropropene	0.20
2-Chloroethylvinylether	0.13
Bromoform	0.20
1,1,2,2-Tetrachloroethane	0.03
Tetrachloroethene	0.03
Chlorobenzene	0.25
1,3 Dichlorobenzene	0.32
1,2 Dichlorobenzene	0.15
1,4 Dichlorobenzene	0.24
Benzene	10
Toluene	10
Ethylbenzene	10

Detection Limits for soils are ten (10) times greater than for water.

Notes: FID optional detector for Method 602

Reference: EPA Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater
EPA 600 14-82-057 - July 1982

Phenol Results Norton AFB April 17, 1984

Robert Watson

Lab Number	Site Location	Parameter
1395	B6-B4	No peaks
1394	B6-B3	No peaks
1392	B6-B2	No peaks
1391	B6-B1	No peaks
1388	B6-1	No peaks
1379	B4-7	No peaks
1378	B4-6	No peaks
1377	B4-5	No peaks
1376	B4-4	No peaks
1375	B4-3	No peaks
1370	B3-B1	No peaks
1368	B3-6	No peaks
1369	B3-6 Duplicate	No peaks
1367	B3-5	No peaks
1366	B3-4	No peaks
1365	B3-3	No peaks
1362	B2-7	No peaks
1361	B2-6	No peaks
1360	B2-5	No peaks
1359	B2-4	No peaks
1358	B2-3	No peaks
1387	B5-7	No peaks
1387	B5-7 Duplicate	No peaks
1386	B5-6	No peaks
1386	B5-6 Duplicate	No peaks
1385	B5-5	No peaks
1385	B5-5 Duplicate	No peaks
1384	B5-4	No peaks
1384	B5-4 Duplicate	No peaks
1383	B5-3	No peaks
1383	B5-3 Duplicate	No peaks

Robert Watson

Lab Number	Site Location	Parameter
1355	B1 A5	No peaks
1354	B1 A4	No peaks
1350	B1-3	No peaks
1349	B1-2	No peaks
1348	B1-1	No peaks
	14 Blanks Soils	No peaks

No phenols were identified in the samples at the following detection limits.

2-Chlorophenol 13 ppm
2-Nitrophenol 13 ppm
Phenol 13 ppm
2,4-Dimethylphenol 13 ppm
2,4-Dichlorophenol 13 ppm
2,4,6-Trichlorophenol 40 ppm
4-Chloro-3 Methylphenol 60 ppm
2,4-Dinitrophenol 40 ppm
2-Methyl-4,6 Dinitrophenol 60 ppm
Pentachlorophenol 60 ppm
4-Nitrophenol 60 ppm

Judith A. Porta

WESTON

DATA SUMMARY FOR: NORTON AFB

DATE RECIEVED: 13 July 1984

RFW SAMPLE NO.	SAMPLE DESCRIPTION	PHENOL mg/L	Cyanide mg/L	TOC mg/L
8407-452-0010	Pond 1W	0.041		
8407=452-0020	MW 8	0.036		
8407-452-0030	MW 2	0.044		
8407-452-0040	Pond 3W	0.041		
8407-452-0050	MW 1	0.043		
8407-452-0060	MW 4	0.064		
8407-452-0070	Pond 2W	0.043		
8407-452-0080	MW 3	0.036		
8407-452-0090	FB-1	0.045		
8407-452-0100	MW 19		NF	
8407-452-0110	MW 17		NF	
8407-452-0120	MW 16		NF	
8407-452-0130	MW 25		NF	
8407-452-0140	MW 18		NF	
8407-452-0150	MW 8		NF	
8407-452-0160	MW 1			6.3
8407-452-0170	Pond 2W			5.1
8407-452-0180	FB-1			NF
8407-452-0190	MW 3			2.4
8407-452-0200	Pond 1W			NF
8407-452-0210	Pond 3W			2.1
8407-452-0220	MW 6			9.7
8407-452-0230	MW 5			2.3
8407-452-0240	MW 2			2.8
8407-452-0250	MW 20			3.8
8407-452-0260	MW 8			2.3
8407-452-0270	MW 4			NF
8407-452-0280	MW 21			NF
8407-452-0290	MW 25			13.7
8407-452-0300	MW 18			10.8
8407-452-0310	MW 19			3.5
8407-452-0320	MW 23			NF
8407-452-0330	MW 10			4.0
8407-452-0340	MW 11			NF
8407-452-0350	MW 12			5.5
8407-452-0360	MW 17			18.1
8407-452-0370	MW 15			41.6
8407-452-0380	MW 8			4.1
8407-452-0390	MW 16			14.1
8407-452-0400	MW 13			3.5
8407-452-0410	MW 14			NF

WESTON

DATA SUMMARY FOR: NORTON AFB

DATE RECEIVED: 13 July 1984

RFW SAMPLE NO.	SAMPLE DESCRIPTION	TOX, ug/L
8407-452-0420	MW 3	11.1
8407-452-0430	Pond 3W	19.5
8407-452-0440	MW 1	17.9
8407-452-0450	FB-1	15.1
8407-452-0460	Pond 1W	20.0
8407-452-0470	Pond 2W	22.4
8407-452-0480	MW 6	6.2
8407-452-0490	MW 24	13.2
8407-452-0500	MW 2	22.8
8407-452-0510	MW 5	14.4
8407-452-0520	MW 4	27.1
8407-452-0530	MW 8	48.0
8407-452-0540	MW 20	125
8407-452-0550	MW 21	12.9
8407-452-0560	MW 23	34.0
8407-452-0570	MW 18	51.2
8407-452-0580	MW 22	15.6
8407-452-0590	MW 25	119
8407-452-0600	MW 19	71.4
8407-452-0610	MW 22	33.4
8407-452-0620	MW 15	288
8407-452-0630	MW 14	127
8407-452-0640	MW 10	26.8
8407-452-0650	MW 8	14.1
8407-452-0660	MW 17	93.7
8407-452-0670	MW 12	NF
8407-452-0680	MW 11	13.8
8407-452-0690	MW 13	6.5
8407-452-0700	MW 16	145

RFW SAMPLE NO.	SAMPLE DESCRIPTION	Phenol, ppm
8407-452-0710	Pond 1S	0.038
8407-452-0720	Pond 2S	0.012
8407-452-0730	Pond 3S	NF
8407-452-0740	Pond 1s1	NF

NF = Not Found

Limit of Detection for TOX = 5 ug/L Phenol = 0.01 ppm

WESTON

DATA SUMMARY FOR: NORTON AFB

RFW SAMPLE NO: 8407-452-	0930	0940	0950	0960	0970	0980
SAMPLE DESCRIPTION:	MW 12	MW 15	MW 3	Pond 1W	MW 18	MW 17
ANALYSIS:						
Pb, mg/L	0.05	NF	NF	NF	0.06	NF
Ni, mg/L	NF	NF	NF	NF	NF	NF
Cd, mg/L	NF	NF	NF	NF	NF	NF
As, ug/L	NF	NF	NF	NF	NF	NF
Zn, mg/L	NF	NF	NF	NF	NF	NF
Cr, mg/L	NF	NF	NF	NF	NF	NF
Cu, mg/L	NF	NF	NF	NF	NF	NF
Hg, ug/L	NF	NF	NF	NF	NF	NF
Li, mg/L	NF					

RFW SAMPLE NO:	0990	1000	1010	1020	1070	1060
SAMPLE DESCRIPTION:	MW 22	0091	MW 19	MW 23	MW 16	MW 7
ANALYSIS:						
Pb, mg/L	NF	NF	NF	NF	NF	NF
Ni, mg/L	NF	NF	NF	NF	NF	NF
Cd, mg/L	NF	NF	NF	NF		NF
As, ug/L	NF	NF	NF	NF	NF	NF
Zn, mg/L	NF	NF	NF	NF	NF	NF
Cr, mg/L	NF	NF	NF	NF	NF	NF
Cu, mg/L	NF	NF	NF	NF	NF	NF
Hg, ug/L	NF	NF	NF	NF		NF
Li, mg/L				NF		

RFW SAMPLE NO:	1030	1040	1050
SAMPLE DESCRIPTION:	MW 24	MW 7	MW 7
ANALYSIS:			
TOC, mg/L	1.7		NF
TOX, ug/L		NF	

WESTON

DATA SUMMARY FOR: NORTON AFB

RFW SAMPLE NO: 8407-452-	0750	0760	0770	0780	0790	0800
SAMPLE DESCRIPTION:	Blank	MW 21	MW 2	MW 6	Pond 2W	Pond 3W
ANALYSIS:						
Pb, mg/L	NF	NF	NF	NF	NF	NF
Ni, mg/L	NF	NF	NF	NF	NF	NF
Cd, mg/L	NF	NF	NF	NF	NF	NF
As, ug/L	NF	NF	NF	NF	NF	NF
Zn, mg/L	NF	NF	NF	NF	NF	NF
Cr, mg/L	NF	NF	NF	NF	NF	NF
Cu, mg/L	NF	NF	NF	NF	NF	NF
Hg, ug/L	NF	NF	NF	NF	NF	NF

RFW SAMPLE NO:	0810	0820	0830	0840	0850	0860
SAMPLE DESCRIPTION:	FB-1	MW 1	MW 20	MW 24	MW 5	MW 8
ANALYSIS:						
Pb, mg/L	NF	NF	NF	NF	NF	NF
Ni, mg/L	NF	NF	NF	NF	NF	NF
Cd, mg/L	NF	NF	NF	NF	NF	NF
As, ug/L	NF	NF	NF	NF	NF	NF
Zn, mg/L	NF	NF	NF	NF	NF	NF
Cr, mg/L	NF	NF	NF	NF	NF	NF
Cu, mg/L	NF	NF	NF	NF	NF	NF
Hg, ug/L	NF	NF	NF	NF	NF	NF

RFW SAMPLE NO:	0870	0880	0890	0900	0910	0920
SAMPLE DESCRIPTION:	MW 4	MW 9	MW 14	MW 11	MW 10	MW 13
ANALYSIS:						
Pb, mg/L	0.43	0.10	0.07	0.06	NF	NF
Ni, mg/L	NF	NF	NF	NF	NF	NF
Cd, mg/L	NF	NF	NF	NF	NF	NF
As, ug/L	NF	NF	NF	NF	354	NF
Zn, mg/L	NF	NF	NF	NF	NF	NF
Cr, mg/L	NF	NF	NF	NF	NF	NF
Cu, mg/L	NF	NF	NF	NF	NF	NF
Hg, ug/L	NF	NF	NF	NF	NF	NF
Li, mg/L				NF		NF

WESTON

DATA SUMMARY FOR: NORTON AFB

RFW SAMPLE NO: 8408-525-	0010	0020	0030	0040	0050	
SAMPLE DESCRIPTION:	0103	Pond 2F	Pond 3F	0106	Pond 1F	104Filt
ANALYSIS:						
Pb, ppm	NF	NF	NF	NF	NF	NF
Cr, ppm	0.38	0.79	0.72	0.50	0.50	NF
Ni, ppm	NF	NF	NF	NF	1.2	NF
Cd, ppm	0.50	NF	NF	NF	0.5	NF
As, ppm	NF	NF	NF	NF	NF	NF
Cu, ppm	2.5	2.7	2.5	2.0	4.8	6.0
Zn, ppm	34.4	201	37.9	40.0	37.2	27.0
Hg, ppb	NF	NF	NF	30.0	NF	NF

NF = Not Found
Detection Limits
Pb = 0.5 ppm
Cr = 0.05 ppm
Ni = 0.1 ppm
Cd = 0.05 ppm
As = 0.01 ppm
Cu = 0.03 ppm
Zn = 0.02 ppm
Hg = 1.0 ppb

WESTON

DATA SUMMARY FOR: NORTON AFB

DETECTION LIMIT

Cyanide = 0.03 mg/L
TOC = 1 mg/L
TOX = 5 ug/L
Phenol = 0.01 ppm (soil)
Pb = 0.05 mg/L
Ni = 0.1 mg/L
Cd = 0.05 mg/L
As = 10 ug/L
Zn = 0.02 mg/L
Cr = 0.05 mg/L
Cu = 0.03 mg/L
Hg = 0.5 ug/L
Li = 1 mg/L

Judith A. Porta

WESTON

DATA SUMMARY FOR: NORTON AFB

RFW SAMPLE NO: 8408-525-	0010	0020	0030	0040	0050	
SAMPLE DESCRIPTION:	0103	Pond 2F	Pond 3F	0106	Pond 1F	104Filt
ANALYSIS:						
Pb, ppm	NF	NF	NF	NF	NF	NF
Cr, ppm	0.38	0.79	0.72	0.50	0.50	NF
Ni, ppm	NF	NF	NF	NF	1.2	NF
Cd, ppm	0.50	NF	NF	NF	0.5	NF
As, ppm	NF	NF	NF	NF	NF	NF
Cu, ppm	2.5	2.7	2.5	2.0	4.8	6.5
Zn, ppm	34.4	201	37.9	40.0	37.2	27.0
Hg, ppb	NF	NF	NF	30.0	NF	NF

NF = Not Found
Detection Limits
Pb = 0.5 ppm
Cr = 0.05 ppm
Ni = 0.1 ppm
Cd = 0.05 ppm
As = 0.01 ppm
Cu = 0.03 ppm
Zn = 0.02 ppm
Hg = 1.0 ppb

NORTON AFB
VOLATILE ORGANIC ANALYSIS FOR SOILS
EPA METHOD 601/602

LAB ID	FIELD NO.	UNIDENTIFIED PEAK(S)	PARAMETER	RESULTS (UG/KG)
S-0689	POND 1-S	1	METHYLENE CHLORIDE	0.40
			TRANS-1,2-DICHLOROETHENE	0.14
			CHLOROFORM	0.69
S-0690	POND 1-SI	0	METHYLENE CHLORIDE	0.11
			TRANS-1,2-DICHLOROETHENE	0.11
			CHLOROFORM	0.34
S-0693	POND 2-S	1	METHYLENE CHLORIDE	0.38
			TRANS-1,2-DICHLOROETHENE	1.1
			CHLOROFORM	0.48
			TRICHLOROETHENE	0.40
S-0694	POND 3-S	2	TRANS-1,2-DICHLOROETHENE	1.2
			CHLOROFORM	0.75

OIL AND GREASE RESULTS

NORTON AIR FORCE BASE, November 1984

Field site	Oil & Grease, mg/L	Date extracted	Weston Lab. No.
TW1	1.1	11/8/84	S1698
TW2	0.8	11/8/84	S1699
TW3	2.0	11/8/84	S1700
TW4	0.8	11/8/84	S1701
TW5	3.0	11/8/84	S1702
TW6	1.0	11/8/84	S1703
TW7	4.0	11/8/84	S1704
TW8	4.0	11/8/84	S1705
TW9	7.2	11/8/84	S1706
TW10	3.0	11/8/84	S1707
TW11	20	11/8/84	S1708
TW12	3.0	11/8/84	S1709
TW13	3.6	11/9/84	S1710
TW14	2.0	11/9/84	S1711
TW15	3.6	11/9/84	S1712
TW16	1.4	11/9/84	S1713
TW17	< 0.6	11/9/84	S1714
TW18	0.9	11/9/84	S1715
TW19	0.8	11/12/84	S1716
TW20	1.0	11/12/84	S1717
TW21	1.0	11/12/84	S1718
TW22	1.0	11/12/84	S1719
TW23[1]	2.0	11/12/84	S1720
TW24[2]	2.7	11/12/84	S1721
TW25[3]	12	11/12/84	S1722
TW26[4]	4.6	11/12/84	S1723

1. TW 23 - field duplicate of TW 22
2. TW 24 - field duplicate of TW 6
3. TW 25 - field duplicate of TW 11
4. TW 26 - field duplicate of TW 16

APPENDIX L

FEDERAL AND STATE DRINKING WATER AND HUMAN HEALTH STANDARDS APPLICABLE IN STATE OF CALIFORNIA



GUIDE TO GROUND-WATER STANDARDS OF THE UNITED STATES

API PUBLICATION 4366

JULY 1983

Prepared by
Ecological Analysts, Inc.
15 Loveton Circle
Sparks, Maryland 21152

3. FEDERAL PROTECTION OF GROUND-WATER QUALITY



The federal programs dealing with the protection of ground-water quality are administered largely by the Environmental Protection Agency (EPA). The federal programs which provide the framework for state regulations are summarized in this section.

3.1 GROUND-WATER PROTECTION POLICY

At this writing, February 1983, U.S. EPA's final policy on ground-water protection, scheduled for September 1982 release, has not been published. Based on the proposed strategy published by EPA in November 1980 and recent press releases, it appears that EPA will be implementing a policy that would give the states lead responsibility in the protection of ground-water quality. EPA's efforts apparently will be focused in three major areas:

1. Development of an internally consistent federal approach to ground-water protection
2. Monitoring, research and development efforts directed toward more comprehensive problem definition and new detection, controls, and clean-up technology development
3. Guidance, coordination, and assistance to states in the development of state policies

A significant component of EPA's policy is expected to be a ground-water classification system which could be used to determine the degree of protection needed for various types of ground water. Ground-water classification is discussed in Chapter 4.

3.2 CLEAN WATER ACT

This statute refers to ground-water protection in municipal waste water treatment, planning, and research programs. Its principal regulatory programs, however, focus on surface water. Section 303 empowers EPA to approve states' water quality standards which are based on the states' classification of rivers and streams. Many states have included ground water in their definition of "waters of the state" for purposes of this act (state summaries). On this basis the National (state) Pollutant Discharge Elimination System (NPDES/SPDES) permitting process may be invocable for purposes of ground-water protection. In addition the act empowers EPA to

1. Develop a comprehensive program for ground-water pollution control [Section 102(a)]
2. In cooperation with states, equip and maintain a surveillance system for monitoring ground-water quality [Section 104(a)(5)]
3. Provide grants to states and area-wide agencies to develop ground-water quality management plans to identify salt water intrusion and control disposal of pollutants in subsurface excavations, and control disposition of wastes. (May include authority for comprehensive ground-water management plans, including conjunctive use with surface water) [Section 102(c), 208(b)]
4. Require development of Best Management Practices (BMP) to control nonpoint source pollution problems to ground-water quality [Section 208(b)]
5. Develop criteria for ground-water quality considering kind and extent of effects on health and welfare from the presence of pollutants [Section 304(a)]
6. Determine information necessary to restore and maintain chemical, physical, and biological integrity of ground water [Section 304(a)]
7. Issue information on the factors necessary to restore and maintain chemical, physical, and biological integrity of ground water [Sections 304(a)(2)]

3.3 SAFE DRINKING WATER ACT

This statute authorizes EPA to set maximum contaminant levels (MCLs) and monitoring requirements for public water systems and provides for the protection of underground sources of drinking water. The MCLs regulate the quality of "finished" water, i.e., water as delivered, not the quality of the source water. As discussed below, the MCLs have been utilized by EPA and the states as the basis for other regulations dealing with ground-water quality and protection.



3.3.1 National Interim Primary Drinking Water Regulations

EPA initiated a detailed study of the health effects of various contaminants in water soon after the Safe Drinking Act (SDWA) was signed into law. So that the regulations could include the findings of this and other studies, the primary drinking water regulations were to be developed in two stages: an interim version and a final version. The interim version of the regulation became effective 24 June 1977. SDWA provides for delegation of authority to the states. State Primary Drinking Water Regulations must be at least as stringent as the federal regulations.

The National Interim Primary Drinking Water Regulations define Maximum Contaminant Level as the maximum permissible level of a contaminant in water which is delivered to the free-flowing outlet of the ultimate user of a public water system, except in the case of turbidity (applicable to surface water only) where the maximum permissible level is measured at the point of entry to the distribution system. The MCLs are provided with the state summaries.

3.3.2 National Secondary Drinking Water Regulations

These regulations control contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water. At considerably higher concentrations of these contaminants, health implications may also exist as well as aesthetic degradation. The National Secondary Drinking Water Regulations are not federally enforceable but are intended as guidelines for the states.

Secondary Maximum Contaminant Levels (SMCLs) are defined as the maximum permissible level of a contaminant in water which is delivered to the free-flowing outlet of the ultimate user of a public water system. Federal and state SMCLs are provided in the state summaries. The states may establish higher or lower levels which may be appropriate depending upon local conditions such as unavailability of alternate sources of water or other compelling factors, provided the public health and welfare are not adversely affected.

3.3.3 Sole Source Aquifer

The Sole Source Aquifer provisions of SDWA allow EPA to designate an aquifer as the sole source of drinking water for an area thereby guaranteeing protection from contamination by federally assisted activities. Local, regional, or state agencies can petition EPA for sole source designation. The EPA Administrator may designate an aquifer which is a sole or principal drinking water source if its contamination would create a significant hazard to public health. If the designation is made, no federal money or financial commitment may be made for any project which the Administrator determines may contaminate the designated aquifer through its recharge zone.

At this writing, February 1983, EPA has designated the following ten sole source aquifers:

Biscayne Aquifer - Florida
Buried Valley Aquifer - New Jersey
Edwards Aquifer - Texas
Camano Island—Whidbey Island Aquifer - Washington
Spokane-Rathdrum Aquifer - Washington and Idaho
Nassau and Suffolk counties - New York
Cape Cod - Massachusetts
Fresno - California
Ten Mile Creek - Maryland
Northern Guam Lens - Guam

The following eighteen are under consideration:

Arizona
- Santa Cruz, Upper Santa Cruz, Aura-Altar Basins

California
- Scotts Valley

Delaware
- New Castle County

Florida
- Volusia - Floridan Aquifer

Idaho
- Snake River Plain

Louisiana
- Baton Rouge
- DeSota Parish

New Jersey
- Coastal Plain
- Ridgewood
- Upper Rockaway

New York
- Kings and Queens counties
- Sardinia
- Schenectady
- Vestal

Pennsylvania
- Seven Valleys

Texas
- Carrizo-Wilcox Aquifer

Texas and New Mexico
- Delaware Basin

Wisconsin
- Niagara Aquifer



3.3.4 Underground Injection Control

The Underground Injection Control (UIC) program regulates the uses of underground injection wells to protect an underground source of drinking water (USDW). USDW means an aquifer or its portion which

1. supplies any public water system or contains a sufficient quantity of ground water to supply a public water system;
2. currently supplies drinking water for human consumption or contains less than 10,000 mg/liter total dissolved solids; and
3. is not an exempted aquifer (40 CFR 146.04 provides criteria for exemption).

SDWA requires any state designated by EPA as requiring a UIC program to develop and submit a state UIC program for EPA approval. EPA has designated each of the fifty states.

The federal program classifies injection wells as follows:

Class I—Wells used to inject hazardous waste, or other industrial and municipal disposal wells which inject fluids beneath the lower-most formation containing a USDW within one-quarter mile of the well bore.

Class II—Wells that inject fluids

1. which are brought to the surface as part of conventional oil or natural gas production and may be mixed with production waste waters from gas plants, unless those waters are classified as a hazardous waste at the time of injection;
2. for enhanced recovery of oil or natural gas; and
3. for storage of hydrocarbons which are liquid at standard temperature and pressure.

Class III—Wells that inject for extraction of minerals including

1. mining of sulfur by the Frasch process;
2. in situ production of uranium or other metals. This category includes only in situ production from ore bodies which have not been conventionally mined. Solution mining of conventional mines such as stopes leaching is included in Class V; and
3. solution mining of salts or potash.

Class IV—Wells used to dispose of hazardous or radioactive waste into or above a formation which contains a USDW within one-quarter mile of the well. Also, wells used to inject hazardous waste that cannot be classified as Class I or Class IV under the above criteria are Class IV wells.

Class V—All other injection wells (40 CFR 146.05(e) and 146.51 provide specific information and exemptions).

Underground injection is controlled through the permitting process. Construction, operation, monitoring and reporting activities are controlled. Individual state programs are based upon, and must be essentially equivalent to, the federal criteria and standards (40 CFR 146).

3.4 TOXIC SUBSTANCE CONTROL ACT

This statute (TSCA) authorizes EPA to restrict or prohibit the manufacture, distribution, and use of products which may result in unreasonable risk to health and the environment. Although ground water is not specifically named in the Act, EPA has taken the position that the protection of health and the environment includes the protection of ground water.

3.5 FEDERAL INSECTICIDE, FUNGICIDE, RODENTICIDE ACT

This statute (FIFRA) gives EPA the responsibility to control the sale and use of all pesticides to prevent unreasonable adverse environmental and health effects. The use and disposal of pesticide packages and containers is also regulated. In deciding whether to register, cancel, suspend, or change the classification of a pesticide, EPA considers a broad range of environmental impacts including those affecting ground water.



3.6 RESOURCE CONSERVATION AND RECOVERY ACT

The Solid Waste Disposal Act and the Resource Recovery Act of 1970, as amended by the Resource Conservation and Recovery Act of 1976 (RCRA), require EPA to establish a national program to regulate the management of waste materials.

3.6.1 Solid Waste

Subtitle D of RCRA established a broad-based national program to improve solid waste management through the development of state and regional solid waste management plans. The act offered federal financial assistance to states interested in developing and implementing a solid waste management plan. The state plans, under federal guidelines, identify respective responsibilities of local, state, and regional authorities, and encourage resource recovery and conservations and the application and enforcement of environmentally sound disposal practices.

A major element of the Subtitle D program is the open dump inventory. Section 4005 of RCRA prohibits open dumping. Federal criteria for classifying solid waste management facilities are provided in 40 CFR 257. EPA cannot approve a state solid waste management program with less stringent criteria. Solid waste management facilities failing to satisfy the criteria are considered open dumps. In order to satisfy these criteria, a facility or practice (in addition to other environmental considerations) shall not contaminate an underground drinking water source beyond the solid waste boundary or beyond an alternative boundary established by the state or in court persuant to the stipulations of 40 CFR 257.3-4. The federal criteria define contamination as an exceedence of the MCLs provided in the National Interim Primary Drinking Water Regulations or an increase in concentration of any parameter for which the ambient concentration exceed the MCL.

3.6.2 Hazardous Waste

EPA has issued a series of hazardous waste regulations under Subtitle C of RCRA (40 CFR 260 to 267 and 122 to 124). On 19 May 1980, EPA issued a comprehensive set of standards for generators and transporters of hazardous waste and "interim status" standards for facilities in existence on 19 November 1980, that treat, store, or dispose of hazardous waste. Such facilities were allowed to operate under interim status until they received an RCRA permit. Subsequently, EPA issued standards for granting RCRA permits to treatment and storage facilities. Standards for land disposal facilities were issued on 26 July 1982—virtually completing the program for controlling hazardous waste under RCRA.

The standards for permitting land disposal facilities were issued after a wide range of regulatory options were considered. Over a period of several years, EPA proposed two different sets of land disposal standards and solicited comments on various issues. On 13 February 1981, EPA issued temporary standards for new land disposal facilities. The 26 July regulations replace those temporary standards except for Class I underground injection wells. These will remain subject to the temporary standards until final standards are issued.

The regulations consist primarily of two complementary sets of performance standards:

1. A set of design and operating standards tailored to each of four types of facilities
2. Ground-water monitoring and response regulations applicable to all land disposal facilities

The design and operating standards implement a liquids management strategy that has two goals:

1. Minimize leachate generated at the facility
2. Remove leachate generated to minimize its chance of reaching ground water

The major requirements include

1. Liner
   - Requirement: design to prevent migration of waste out of the facility during its active life
   - Applicability: landfills, surface impoundments, and waste piles
2. Leachate collection and removal
   - Requirement: collect and remove leachate from the facility and ensure that leachate depth over the liner does not exceed 30 centimeters (1 foot)
   - Applicability: landfills and waste piles



3. Run-on and runoff control systems
   - Requirement: design tc control flow dunng at least 25-year storm
   - Applicability: landfills, waste piles, land treatment
4. Wind dispersal controls
   - Requirement: cover waste or otherwise manage unit to control wind dispersal
   - Applicability: landfills, waste piles, and land treatment units that contain particulate matter
5. Overtopping controls
   - Requirement: prevent overtopping or overfilling
   - Applicability: surface impoundments
6. Disposal unit closure
   - Requirement: final cover (cap) over waste unit designed to minimize infiltration of precipitation
   - Applicability: landfills and surface impoundments (if used for disposal)
7. Storage unit closure
   - Requirement: remove waste and decontaminate
   - Applicability: surface impoundments used for treatment or storage and waste piles
8. Postclosure Care
   - Maintain effectiveness of final cover
   - Operate leachate collection and removal system
   - Maintain ground-water monitoring system (and leak detection system where double liner is used)
   - Continue 30 years after closure

The goal of the ground-water monitoring and response program is to detect and correct any ground-water contamination. There are four main elements:

1. A detection monitoring program which requires the permittee to install a system to monitor ground water in the uppermost aquifer to determine if a leachate plume has reached the edge of the waste management area.
2. A ground-water protection standard is set when a hazardous constituent is detected. The standard specifies concentration limits, compliance point, and compliance period.
3. A compliance monitoring program determines if the facility is complying with its ground-water protection standard.
4. Corrective action is required when the ground-water protection standard is violated. The permittee must either remove the contamination or treat it in place to restore ground-water quality.

Until hazardous waste management facilities are issued permits, existing facilities will continue to operate under interim status standards. Facilities operating under interim status will be required to file Part B applications for final permits.

Under Subtitle C of RCRA, EPA approves state hazardous waste management programs in two phases. Phase I authorization gives states the right to control transportation and generation of hazardous wastes within their borders and to regulate existing treatment, storage, and disposal facilities. Phase II authorization includes the permitting of new facilities.

3.7 COMPREHENSIVE ENVIRONMENTAL RESPONSE, COMPENSATION, AND LIABILITY ACT

This statute (CERCLA), commonly referred to as Superfund, authorizes EPA to respond to releases or threatened releases into the environment, including ground water, of any hazardous substance which may present an imminent and substantial danger to public health. The act provides funds for emergency action and has cost recovery provisions.



CALIFORNIA

Classification–Ground water is included in the definition of "Waters of the State" as found in the California Water Quality Act. Ground water has been included in beneficial use classes developed as part of Basin Management Programs of the Water Resources Control Board and the Regional Boards.

Quality Standards–The general policy is a nondegradation policy to protect the present and possible future uses of ground water as a source of potable, industrial, and agricultural water supply. Quality standards are specific to each use class and Basin Program.

Drinking Water Standards–The California Water Resources Control Board has adopted the federal primary and secondary drinking water standards.

Appropriations–There are no state-wide permit requirements, however, see Controlled Use Areas below.

Controlled Use Areas–Several ground-water basins are being managed by local authorities in response to special legislative acts and court orders. These authorities regulate ground-water withdrawals within their jurisdictions. However, these areas account for less than five percent of all ground-water basins.

Well Construction–Local counties may adopt well construction standards and require drillers to be licensed. Approximately half of California's 58 counties have done so.

Underground Injection Control–California is in the process of submitting a UIC program for EPA approval. The Water Resources Control Board will be the lead agency in the program. Class II wells will be regulated by the Oil and Gas Division of the Department of Conservation.

Waste Management Facilities–The solid and hazardous waste management programs are administered by the Solid Waste Management Board. The Hazardous Waste Management Regulations are administered by the Department of Health Services.

> **Solid Waste**–The California Solid Waste Management Regulations require a ground-water monitoring system for disposal sites. Monitoring requirements are on a case-by-case basis.
>
> **Hazardous Waste**–California has received interim status authorization for its RCRA Phase I program and is seeking Phase II authority. Ground-water monitoring requirements are included in permit conditions and are generally equivalent to EPA requirements.

Sole Source Aquifers–The Fresno area aquifer has been designated as sole source by EPA. The Scotts Valley aquifer is under consideration by EPA.

Geological Surveys–

Division of Mines and Geology
Department of Conservation
1416 Ninth St.
Sacramento, CA 95814
916-445-1923
State Geologist:
Dr. James F. Davis

Water Resources Division
U.S. Geological Survey
Federal Bldg., Room W-2235
2800 Cottage Way
Sacramento, CA 95825
916-484-4606
District Chief:
T.J. Durbin

References–

California Water Quality Act
(California Water Code, Div. 7, Ch. 482)

California Solid Waste Management Regulations
(California Admin. Code, Title 14, Div. 7, Ch. 1-5 and 9)

California Hazardous Waste Management Regulations
(California Admin. Code, Title 22, Div. 4, Ch. 30)

Contacts–

Ms. Helen Joyce Peters
Department of Water Resources
P.O. Box 388
Sacramento, CA 95802
916-445-2182

Mr. Evan Nossoff
Water Resources Control Board
P.O. Box 100
Sacramento, CA 95801
916-322-8353

Revisions provided by Ms. Helen Joyce Peters in a letter received 11 April 1983.

CALIFORNIA

Parameter (mg/l unless noted)	Drinking Water Standards		Quality Standards	Monitoring Requirements	
	Federal	State		Solid Waste	Hazardous Waste
Arsenic	0.05	0.05			
Barium	1.0	1.0			
Cadmium	0.010	0.010			
Chromium	0.05	0.05			
Lead	0.05	0.05			
Mercury	0.002	0.002			
Selenium	0.01	0.01			
Silver	0.05	0.05			
Fluoride	1.4–2.4	1.4–2.4			
Nitrate (as N)	10.0	10.0			
Endrin	0.0002	0.0002			
Lindane	0.004	0.004			
Methoxychlor	0.1	0.1			
Toxaphene	0.005	0.005			
2,4-D	0.1	0.1			
2,4,5-TP Silvex	0.01	0.01			
Trihalomethanes	0.1	0.1			
Turbidity (TU)	1.0	1.0			
Coliform bacteria — membrane filter test (#/100 ml)	1.0	1.0			
Gross alpha (pCi/l)	15.0	15.0			
Combined Radium 226 and Radium 228	5.0	5.0			
Beta and photon particle activity (mrem/yr)	4.0	4.0			
Sodium	M	M			
Chloride	250.0	250.0			
Color (units)	15.0	15.0			
Copper	1.0	1.0			
Corrosivity	Noncorrosive	Noncorrosive			
Foaming agents	0.5	0.5			
Iron	0.3	0.3			
Manganese	0.05	0.05			
Odor (threshold no.)	3.0	3.0			
pH (units)	6.5–8.5	6.5–8.5			
Sulfate	250.0	250.0			
Total dissolved solids	500.0	500.0			
Zinc	5.0	5.0			
Phenols					
Specific conductance					
Total organic carbon					
Total organic halogen					

Note: "M" denotes monitoring requirement. See Section 4.3.

ENVIRONMENTAL PROTECTION AGENCY NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

(40 CFR 141; 40 FR 59565, December 24, 1975; Amended by 41 FR 28402, July 9, 1976; 44 FR 68641, November 29, 1979; Corrected by 45 FR 15542, March 11, 1980; 45 FR 57342, August 27, 1980)

Title 40—Protection of Environment
CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

PART 141—NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

Subpart A—General

Sec.
141.1 Applicability.
141.2 Definitions.
141.3 Coverage.
141.4 Variances and exemptions.
141.5 Siting requirements.
141.6 Effective dates.

Subpart B—Maximum Contaminant Levels

141.11 Maximum contaminant levels for inorganic chemicals.
141.12 Maximum contaminant levels for organic chemicals.
141.13 Maximum contaminant levels for turbidity.
141.14 Maximum microbiological contaminant levels.
141.15 Maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity in community water systems.
141.16 Maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides in community water systems.

Subpart C—Monitoring and Analytical Requirements

141.21 Microbiological contaminant sampling and analytical requirements.
141.22 Turbidity sampling and analytical requirements.
141.23 Inorganic chemical sampling and analytical requirements.
141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.
141.25 Analytical Methods for Radioactivity.
141.26 Monitoring Frequency for Radioactivity in Community Water Systems.
141.27 Alternative analytical techniques.
141.28 Approved laboratories.
141.29 Monitoring of consecutive public water systems.

Subpart D—Reporting Public Notification, and Record-keeping

141.31 Reporting requirements.
141.32 Public notification of variances, exemptions, and non-compliance with regulations.
141.33 Record maintenance.

Subpart E—Special Monitoring Regulations for Organic Chemicals

141.40 Special monitoring for organic chemicals.

Authority: Secs. 1412, 1414, 1445, and 1450 of the Public Health Service Act, 88 Stat. 1660 (42 U.S.C. 300g-1, 300g-3, 300j-4, and 300j-9).

Subpart A—General

§ 141.1 Applicability.

This part establishes primary drinking water regulations pursuant to section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93-523); and related regulations applicable to public water systems.

§ 141.2 Definitions.

As used in this part, the term:

(a) "Act" means the Public Health Service Act, as amended by the Safe Drinking Water Act, Pub. L. 93-523.

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Maximum contaminant level" means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system, except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

(d) "Person" means an individual, corporation, company, association, partnership, State, municipality, or Federal agency.

(e) "Public water system" means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system."

(i) "Community water system" means a public water system which serves at least 15 service connections used by year-round residents or regularly serves at least 25 year-round residents.

(ii) "Non-community water system" means a public water system that is not a community water system.

(f) "Sanitary survey" means an on-site review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

(g) "Standard sample" means that aliquot of finished drinking water that is examined for the presence of coliform bacteria.

(h) "State" means the agency of the State government which has jurisdiction over public water systems. During any period when a State does not have primary enforcement responsibility pursuant to Section 1413 of the Act, the term "State" means the Regional Administrator, U.S. Environmental Protection Agency.

(i) "Supplier of water" means any person who owns or operates a public water system.

(j) "Dose equivalent" means the product of the absorbed dose from ionizing radiation and such factors as account for differences in biological effectiveness due to the type of radiation and its distribution in the body as specified by the International Commission on Radiological Units and Measurements (ICRU).

(k) "Rem" means the unit of dose equivalent from ionizing radiation to the total body or any internal organ or organ system. A "millirem (mrem)" is 1/1000 of a rem.

(l) "Picocurie (pCi)" means that quantity of radioactive material producing 2.22 nuclear transformations per minute.

(m) "Gross alpha particle activity" means the total radioactivity due to alpha particle emission as inferred from measurements on a dry sample.

(n) "Man-made beta particle and photon emitters" means all radionuclides emitting beta particles and/or photon

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listed in Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure, NBS Handbook 69, except the daughter products of thorium-232, uranium-235 and uranium-238.

(o) "Gross beta particle activity" means the total radioactivity due to beta particle emission as inferred from measurements on a dry sample.

[41 FR 28402, July 9, 1976]
[141.2 (p)-(t) added by 44 FR 68641, November 29, 1979]

(p) "Halogen" means one of the chemical elements chlorine, bromine or iodine.

(q) "Trihalomethane" (THM) means one of the family of organic compounds, named as derivatives of methane, wherein three of the four hydrogen atoms in methane are each substituted by a halogen atom in the molecular structure.

(r) "Total trihalomethanes" (TTHM) means the sum of the concentration in milligrams per liter of the trihalomethane compounds (trichloromethane [chloroform], dibromochloromethane, bromodichloromethane and tribromomethane [bromoform]), rounded to two significant figures.

(s) "Maximum Total Trihalomethane Potential (MTP)" means the maximum concentration of total trihalomethanes produced in a given water containing a disinfectant residual after 7 days at a temperature of 25° C or above.

(t) "Disinfectant" means any oxidant, including but not limited to chlorine, chlorine dioxide, chloramines, and ozone added to water in any part of the treatment or distribution process, that is intended to kill or inactivate pathogenic microorganisms.

§ 141.3 Coverage.

This part shall apply to each public water system, unless the public water system meets all of the following conditions:

(a) Consists only of distribution and storage facilities (and does not have any collection and treatment facilities);

(b) Obtains all of its water from, but is not owned or operated by, a public water system to which such regulations apply;

(c) Does not sell water to any person; and

(d) Is not a carrier which conveys passengers in interstate commerce.

§ 141.4 Variances and exemptions.

Variances or exemptions from certain provisions of these regulations may be granted pursuant to Sections 1415 and 1416 of the Act by the entity with primary enforcement responsibility. Provisions under Part 142, *National Interim Primary Drinking Water Regulations Implementation*—subpart E (Variances) and subpart F (Exemptions)—apply where EPA has primary enforcement responsibility.

§ 141.5 Siting requirements.

Before a person may enter into a financial commitment for or initiate construction of a new public water system or increase the capacity of an existing public water system, he shall notify the State and, to the extent practicable, avoid locating part or all of the new or expanded facility at a site which:

(a) Is subject to a significant risk from earthquakes, floods, fires or other disasters which could cause a breakdown of the public water system or a portion thereof; or

(b) Except for intake structures, is within the floodplain of a 100-year flood or is lower than any recorded high tide where appropriate records exist.

The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public water systems siting which are made at the State or local government levels.

§ 141.6 Effective dates.

[141.6 revised by 44 FR 68641, November 29, 1979]

(a) Except as provided in paragraph (b) of this section, the regulations set forth in this part shall take effect on June 24, 1977.

(b) The regulations for total trihalomethanes set forth in § 141.12(c) shall take effect 2 years after the date of promulgation of these regulations for community water systems serving 75,000 or more individuals, and 4 years after the date of promulgation for communities serving 10,000 to 74,999 individuals.

(c) The regulations set forth in 141.11 (a), (c) and (d); 141.14(a)(1); 141.14(b)(1)(c); 141.14(b)(2)(i); 141.14(d); 141.21 (a), (c) and (i); 141.22 (a) and (e); 141.23 (a)(3) and (a)(4); 141.23(f); 141.24(a)(3); 141.24 (e) and (f); 141.25(e); 141.27(a); 141.28 (a) and (b); 141.31 (a), (c), (d) and (e); 141.32(b)(3); and 141.32(d) shall take effect immediately upon promulgation.

(d) The regulations set forth in 141.41 shall take effect 18 months from the date of promulgation. Suppliers must complete the first round of sampling and reporting within 12 months following the effective date.

(e) The regulations set forth in 141.42 shall take effect 18 months from the date of promulgation. All requirements in 141.42 must be completed within 12 months following the effective date.

[141.6 (c)-(e) added by 45 FR 57342, August 27, 1980]

Subpart B—Maximum Contaminant Levels

§ 141.11 Maximum contaminant levels for inorganic chemicals.

(a) The MCL for nitrate is applicable to both community water systems and non-community water systems except as provided by in paragraph (d). The levels for the other organic chemicals apply only to community water systems. Compliance with MCLs for inorganic chemicals is calculated pursuant to § 141.23.

[141.11(a) amended by 45 FR 57342, August 27, 1980]

(b) The following are the maximum contaminant levels for inorganic chemicals other than fluoride:

Contaminant	Level, milligrams per liter
Arsenic	0.05
Barium	1.
Cadmium	0.010
Chromium	0.05
Lead	0.05
Mercury	0.002
Nitrate (as N)	10.
Selenium	0.01
Silver	0.05

(c) When the annual average of the maximum daily air temperatures for the location in which the community water system is situated is the following, the maximum contaminant levels for fluoride are:

Temperature Degrees Fahrenheit	Degrees Celsius	Level, milligrams per liter
53.7 and below	12.0 and below	2.4
53.8 to 58.3	12.1 to 14.6	2.2
58.4 to 63.8	14.7 to 17.6	2.0
63.9 to 70.6	17.7 to 21.4	1.8
70.7 to 79.2	21.5 to 26.2	1.6
79.3 to 90.5	26.3 to 32.5	1.4

(c) Fluoride at optimum levels in drinking water has been shown to have beneficial effects in reducing the occurrence of tooth decay.

[141.11 (c) amended by 45 FR 57342, August 27, 1980]

(d) At the discretion of the State, nitrate levels not to exceed 20 mg/l may be allowed in a non-community water system if the supplier of water demonstrates to the satisfaction of the State that:

(1) Such water will not be available to children under 6 months of age; and

(2) There will be continuous posting of the fact that nitrate levels exceed 10 mg/l and the potential health effects of exposure; and

(3) Local and State public health authorities will be notified annually of nitrate levels that exceed 10 mg/l; and

(4) No adverse health effects shall result.

[141.11 (d) added by 45 FR 57342, August 27, 1980]

§ 141.12 Maximum contaminant levels for organic chemicals.

[141.12 revised by 44 FR 68641, November 29, 1979]

The following are the maximum contaminant levels for organic chemicals. The maximum contaminant levels for organic chemicals in paragraphs (a) and (b) of this section apply to all community water systems. Compliance with the maximum contaminant levels in paragraphs (a) and (b) is calculated pursuant to § 141.24. The maximum contaminant level for total trihalomethanes in paragraph (c) of this section applies only to community water systems which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process. Compliance with the maximum contaminant level for total trihalomethanes is calculated pursuant to § 141.30.

	Level, milligrams per liter
(a) Chlorinated hydrocarbons:	
Endrin (1,2,3,4,10, 10-hexachloro-6,7-epoxy-1,4, 4a,5,6,7,8,8a-octahydro-1,4-endo, endo-5,8-dimethano naphthalene).	0.0002
Lindane (1,2,3,4,5,6-hexachlorocyclohexane, gamma isomer).	0.004
Methoxychlor (1,1,1-Trichloro-2, 2-bis [p-methoxyphenyl] ethane).	0.1
Toxaphene ($C_{10}H_{10}Cl_8$-Technical chlorinated camphene, 67-69 percent chlorine).	0.005
(b) Chlorophenoxys:	
2,4-D, (2,4-Dichlorophenoxyacetic acid).	0.1
2,4,5-TP Silvex (2,4,5-Trichlorophenoxypropionic acid).	0.01

(c) Total trihalomethanes (the sum of the concentrations of bromodichloromethane, dibromochloromethane, tribromomethane (bromoform) and trichloromethane (chloroform) 0.10 mg/l.

[141.12(c) added by 44 FR 68641, November 29, 1979]

§ 141.13 Maximum contaminant levels for turbidity.

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems using surface water sources in whole or in part. The maximum contaminant levels for turbidity in drinking water, measured at a representative entry point(s) to the distribution system, are:

(a) One turbidity unit (TU), as determined by a monthly average pursuant to § 141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not do any of the following:

(1) Interfere with disinfection;

(2) Prevent maintenance of an effective disinfectant agent throughout the distribution system; or

(3) Interfere with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 141.22.

§ 141.14 Maximum microbiological contaminant levels.

The maximum contaminant levels for coliform bacteria, applicable to community water systems and non-community water systems, are as follows:

(a) When the membrane filter technique pursuant to § 141.21(a) is used, the number of coliform bacteria shall not exceed any of the following:

[141.14(a)(1) revised by 45 FR 57342, August 27, 1980]

(1) One per 100 milliliters as the arithmetic mean of all samples examined per compliance period pursuant to § 141.21(b) or (c), except that, at the primacy Agency's discretion systems required to take 10 or fewer samples per month may be authorized to exclude one positive routine sample per month from the monthly calculation if: (i) as approved on a case-by-case basis the State determines and indicates in writing to the public water system that no unreasonable risk to health existed under the conditions of this modification. This determination should be based upon a number of factors not limited to the following: (A) the system provided and had maintained an active disinfectant residual in the distribution system, (B) the potential for contamination as indicated by a sanitary survey, and (C) the history of the water quality at the public water system (e.g. MCL or monitoring violations); (ii) the supplier initiates a check sample on each of two consecutive days from the same sampling point within 24 hours after notification that the routine sample is positive, and each of these check samples is negative; and (iii) the original positive routine sample is reported and recorded by the supplier pursuant to § 141.31(a) and § 141.33(a). The supplier shall report to the State its compliance with the conditions specified in this paragraph and a summary of the corrective action taken to resolve the prior positive sample result. If a positive routine sample is not used for the monthly calculation, another routine sample must be analyzed for compliance purposes. This provision may be used only once during two consecutive compliance periods.

(2) Four per 100 milliliters in more than one sample when less than 20 are examined per month; or

(3) Four per 100 milliliters in more than five percent of the samples when 20 or more are examined per month.

(b)(1) When the fermentation tube method and 10 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

[141.14(b)(1)(i) revised by 45 FR 57342, August 27, 1980]

(i) More than 10 percent of the portions (tubes) in any one month pursuant to § 141.21 (b) or (c) except that, at the State's discretion, systems required to take 10 or fewer samples per month may be authorized to exclude one positive routine sample resulting in one or more positive tubes per month from the monthly calculation if: (A) as approved on a case-by-case basis the State determines and indicates in writing to the public water system that no unreasonable risk to health existed under the conditions of this modification. This determination should be based upon a number of factors not limited to the following: (*1*) the system provided and had maintained an active disinfectant residual in the distribution system, (*2*) the potential for contamination as indicated by a sanitary survey, and (*3*) the history of the water quality at the public water system (e.g. MCL or monitoring violations); (B) the supplier initiates a check sample on each of two consecutive days from the sampling point within 24 hours after notification that the routine sample is positive, and each of these check samples is negative; and (C) the original positive routine sample is reported and recorded by the supplier pursuant to § 141.31(a) and § 141.33(a). The supplier shall report to the State its compliance with the conditions specified in this paragraph and report the action taken to resolve the prior positive sample result. If a positive routine sample is not used for the monthly calculation, another routine sample must be analyzed for compliance purposes. This provision may be used only once during two consecutive compliance periods.

(ii) three or more portions in more than one sample when less than 20 samples are examined per month; or

(iii) three or more portions in more than five percent of the samples when 20 or more samples are examined per month.

(2) When the fermentation tube

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Part V

Environmental Protection Agency

Water Quality Criteria Documents; Availability

ENVIRONMENTAL PROTECTION AGENCY

[FRL 1623-3]

Water Quality Criteria Documents; Availability

AGENCY: Environmental Protection Agency.

ACTION: Notice of Water Quality Criteria Documents.

SUMMARY: EPA announces the availability and provides summaries of water quality criteria documents for 64 toxic pollutants or pollutant categories. These criteria are published pursuant to section 304(a)(1) of the Clean Water Act.

AVAILABILITY OF DOCUMENTS: Summaries of both aquatic-based and health-based criteria from the documents are published below. Copies of the complete documents for individual pollutants may be obtained from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161, (703-487-4650). A listing of the NTIS publication order numbers for all 64 criteria documents is published below. These documents are also available for public inspection and copying during normal business hours at the Public Information Reference Unit, U.S. Environmental Protection Agency, Room 2404 (rear), 401 M St., S.W., Washington, D.C. 20460. As provided in 40 CFR Part 2, a reasonable fee may be charged for copying services. Copies of these documents are also available for review in the EPA Regional Office libraries.

Some of the documents are not available from the EPA office listed below. Requests sent to that office will be forwarded to NTIS or returned to the sender.

1. Acenaphthene, PB81-117269.
2. Acrolein, PB81-117277.
3. Acrylonitrile, PB81-117285.
4. Aldrin/Dieldrin, PB81-117301.
5. Antimony, PB81-117319.
6. Arsenic, PB81-117327.
7. Asbestos, PB81-117335.
8. Benzene, PB81-117293.
9. Benzidine, PB81-117343.
10. Beryllium, PB81-117350.
11. Cadmium, PB81-117368.
12. Carbon Tetrachloride, PB81-117376.
13. Chlordane, PB81-117384.
14. Chlorinated benzenes, PB81-117392.
15. Chlorinated ethanes, PB81-117400.
16. Chloroalkyl ethers, PB81-117418.
17. Chlorinated naphthalene, PB81-117426.
18. Chlorinated phenols, PB81-117434.
19. Chloroform, PB81-117442.
20. 2-chlorophenol, PB81-117459.
21. Chromium, PB81-117467.
22. Copper, PB81-117475.
23. Cyanides, PB81-117483.
24. DDT, PB81-117491.
25. Dichlorobenzenes, PB81-117509.
26. Dichlorobenzidine, PB81-117517.
27. Dichloroethylenes, PB81-117525.
28. 2,4-dichlorophenol, PB81-117533.
29. Dichloropropanes/propenes, PB81-117541.
30. 2,4-dimethylphenol, PB81-117558.
31. Dinitrotoluene, PB81-117566.
32. Diphenylhydrazine, PB81-117731.
33. Endosulfan, PB81-117574.
34. Endrin, PB81-117582.
35. Ethylbenzene, PB81-117590.
36. Fluoranthene, PB81-117608.
37. Haloethers, PB81-117616.
38. Halomethanes, PB81-117624.
39. Heptachlor, PB81-117632.
40. Hexachlorobutadiene, PB81-117640.
41. Hexachlorocyclohexane, PB81-117657.
42. Hexachlorocyclopentadiene, PB81-117665.
43. Isophorone, PB81-117673.
44. Lead, PB81-117681.
45. Mercury, PB81-117699.
46. Naphthalene, PB81-117707.
47. Nickel, PB81-117715.
48. Nitrobenzene, PB81-117723.
49. Nitrophenols, PB81-117749.
50. Nitrosamines, PB81-117756.
51. Pentachlorophenol, PB81-117764.
52. Phenol, PB81-117772.
53. Phthalate esters, PB81-117780.
54. Polychlorinated biphenyls (PCBs), PB81-117798.
55. Polynuclear aromatic hydrocarbons, PB81-117806.
56. Selenium, PB81-117814.
57. Silver, PB81-117822.
58. Tetrachloroethylene, PB81-117830.
59. Thallium, PB81-117848.
60. Toluene, PB81-117855.
61. Toxaphene, PB81-117863.
62. Trichloroethylene, PB81-117871.
63. Vinyl chloride, PB81-117889.
64. Zinc, PB81-117897.

FOR FURTHER INFORMATION CONTACT: Dr. Frank Gostomski, Criteria and Standards Division (WH-585), United States Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460, (202) 245-3032.

SUPPLEMENTARY INFORMATION:

Background

Pursuant to section 304(a)(1) of the Clean Water Act, 33 U.S.C. 1314(a)(1), EPA is required to periodically review and publish criteria for water quality accurately reflecting the latest scientific knowledge:

(A) on the kind and extent of all identifiable effects on health and welfare including, but not limited to, plankton, fish, shellfish, wildlife, plant life, shorelines, beaches, esthetics, and recreation which may be expected from the presence of pollutants in any body of water, including groundwater. (B) on the concentration and dispersal of pollutants, or their byproducts, through biological, physical, and chemical processes, and (C) on the effects of pollutants on biological community diversity, productivity, and stability, including information on the factors affecting rates of eutrophication and rates of organic and inorganic sedimentation for varying types of receiving waters.

EPA is today announcing the availability of criteria documents for 64 of the 65 pollutants designated as toxic under section 307(a)(1) of the Act. The document on TCDD (Dioxin) will be published within the next month after review of recent studies. Criteria for the section 307(a)(1) toxic pollutants being published today will replace the criteria for those same pollutants found in the EPA publication, *Quality Criteria for Water*, (the "Red Book.") Criteria for all other pollutants and water constituents found in the "Red Book" remain valid. The criteria published today have been derived using revised methodologies for determining pollutant concentrations that will, when not exceeded, reasonably protect human health and aquatic life. Draft criteria documents were made available for public comment (44 FR 15926, March 15, 1979, 44 FR 43660, July 25, 1979, 44 FR 58628, October 1, 1979). These final criteria have been derived after consideration of all comments received.

These criteria documents are also issued in satisfaction of the Settlement Agreement in *Natural Resources Defense Council, et al.* v. *Train*, 8 E.R.C. 2120 (1976), modified, 12 E.R.C. 1833 (D.D.C. 1979). Pursuant to paragraph 11 of that agreement, EPA is required to publish criteria documents for the 65 pollutants which Congress, in the 1977 amendments to the Act, designated as toxic under section 307(a)(1). These documents contain recommended maximum permissible pollutant concentrations consistent with the protection of aquatic organisms, human health, and some recreational activities. Although paragraph 11 imposes certain obligations on the Agency, it does not create additional authority.

The Development of Water Quality Criteria

Section 304(a)(1) criteria contain two essential types of information: (1) discussions of available scientific data on the effects of pollutants on public health and welfare, aquatic life and recreation, and (2) quantitative concentrations or qualitative assessments of the pollutants in water which will generally ensure water

quality adequate to support a specified water use. Under section 304(a)(1), these criteria are based solely on data and scientific judgments on the relationship between pollutant concentrations and environmental and human health effects. Criteria values do not reflect considerations of economic or technological feasibility.

Publication of water quality criteria of this type has been an ongoing process which EPA, and its predecessor Agency, the Federal Water Pollution Control Administration, have been engaged in since 1968. At that time the first Federal compilation of water quality criteria, the so-called "Green Book" *(Water Quality Criteria)*, was published. As now, these criteria contained both narrative discussions of the environmental effects of pollutants on a range of possible uses and concentrations of pollutants necessary to support these uses. Since that time, water quality criteria have been revised and expanded with publication of the "Blue Book" *(Water Quality Criteria 1972)* in 1973 and the "Red Book" *(Quality Criteria for Water)* in 1976.

Since publication of the Red Book there have been substantial changes in EPA's approach to assessing scientific data and deriving section 304(a)(1) criteria. Previous criteria were derived from a limited data base. For many pollutants, an aquatic life criterion was derived by multiplying the lowest concentration known to have acute lethal effect on half of a test group of an aquatic species (the LC50 value) by an application factor in order to protect against chronic effects. If data showed a substance to be bioaccumulative or to have other significant long-term effects, a factor was used to reduce the indicated concentrations to a level presumed to be protective. Criteria for the protection of human health were similarly derived by considering the pollutants' acute, chronic, and bioaccumulative effects on non-human mammals and humans.

Although a continuation of the process of criteria development, the criteria published today were derived using revised methodologies (Guidelines) for calculating the impact of pollutants on human health and aquatic organisms. These Guidelines consist of systematic methods for assessing valid and appropriate data concerning acute and chronic adverse effects of pollutants on aquatic organisms, non-human mammals, and humans. By use of these data in prescribed ways, criteria are formulated to protect aquatic life and human health from exposure to the pollutants. For some pollutants, bioconcentration properties are used to formulate criteria protective of aquatic life uses. For almost all of the pollutants, bioconcentration properties are used to assess the relative extent of human exposure to the pollutant either directly through ingestion of water or indirectly through consumption of aquatic organisms. Human health criteria for carcinogens are presented as incremental risks to man associated with specific concentrations of the pollutant in ambient water. The Guidelines used to derive criteria protective of aquatic life and human health are fully described in appendices B and C, respectively, of this Notice.

The Agency believes that these Guidelines provide criteria which more accurately reflect the effects of these pollutants on human health and on aquatic organisms and their uses. They are based on a more rational and consistent approach for using scientific data. These Guidelines were developed by EPA scientists in consultation with scientists from outside the Agency and they have been subjected to intensive public comment.

Neither the Guidelines nor the criteria are considered inflexible doctrine. Even at this time, EPA is taking action to employ the resources of peer review groups, including the Science Advisory Board, to evaluate recently published data, and EPA is conducting its own evaluation of new data to determine whether revisions to the criteria documents would be warranted.

The criteria published today are based solely on the effect of a single pollutant. However, pollutants in combination may have different effects because of synergistic, additive, or antagonistic properties. It is impossible in these documents to quantify the combined effects of these pollutants, and persons using criteria should be aware that site-specific analysis of actual combinations of pollutants may be necessary to give more precise indications of the actual environmental impacts of a discharge.

Relationship of the Section 304(a)(1) Criteria to Regulatory Programs

Section 304(a)(1) criteria are not rules and they have no regulatory inpact. Rather, these criteria present scientific data and guidance on the enviromental effect of pollutants which can be useful to derive regulatory requirements based on considerations of water quality impacts. Under the Clean Water Act, these regulatory requirements may include the promulgation of water quality-based effluent limitations under section 302, water quality standards under section 303, or toxic pollutant effluent standards under section 307. States are encouraged to begin to modify or, if necessary, develop new programs necessary to support the implementation of regulatory controls for toxic pollutants. As appropriate, States may incorporate criteria for toxic pollutants, based on this guidance, into their water quality standards.

Section 304(a)(1) criteria have been most closely associated with the development of State water quality standards, and the "Red Book" values have, in the past, been the basis for EPA's assessments of the adequacy of State requirements. However, EPA is now completing a major review of its water quality standards policies and regulations. After consideration of comments received on an Advance Notice of Proposed Rulemaking (43 FR 29588, July 10, 1978) and the draft criteria documents, the Agency intends to propose, by the end of this year, a revised water quality standards regulation which will clarify the Agency's position on a number of significant standards issues.

With the publication of these criteria however, it is appropriate to discuss EPA's current thinking on standards issues relating to their use. This discussion does not establish new regulatory requirements and is intended as guidance on the possible uses of these criteria and an indication of future rulemaking the Agency may undertake. No substantive requirements will be established without further opportunity for public comment.

Water Quality Standards

Section 303 of the Clean Water Act provides that water quality standards be developed for all surface waters. A water quality standard consists basically of two parts: (1) A "designated use" for which the water body is to be protected (such as "agricultural," "recreation" or "fish and wildlife"), and (2) "criteria" which are numerical pollutant concentration limits or narrative statements necessary to preserve or achieve the designated use. A water quality standard is developed through State or Federal rulemaking proceedings and must be translated into enforceable effluent limitations in a point source (NPDES) permit or may form the basis of best management practices applicable to nonpoint sources under section 208 of the Act.

Relationship of Section 304(a)(1) Criteria to the Criteria Component of State Water Quality Standards:

In the ANPRM, EPA announced a policy of "presumptive applicability" for

section 304(a)(1) criteria codified in the "Red Book." Presumptive applicability meant that a State had to adopt a criterion for a particular water quality parameter at least as stringent as the recommendation in the Red Book unless the State was able to justify a less stringent criterion based on: natural - background conditions, more recent scientific evidence, or local, site-specific information. EPA is rescinding the policy of presumptive applicability because it has proven to be too inflexible in actual practice.

Although the section 304(a)(1) criteria represent a reasonable estimate of pollutant concentrations consistent with the maintenance of designated water uses, States may appropriately modify these values to reflect local conditions. In certain circumstances, the criteria may not accurately reflect the toxicity of a pollutant because of the effect of local water quality characteristics or varying sensitivities of local populations. For example, in some cases, ecosystem adaptation may enable a viable, balanced aquatic population to exist in waters with high natural background levels of certain pollutants. Similarly, certain compounds may be more or less toxic in some waters because of differences in alkalinity, temperature, hardness, and other factors.

Methods for adjusting the section 304(a)(1) criteria to reflect these local differences are discussed below.

Relationship of Section 304(a)(1) Criteria to Designated Water Uses:

The criteria published today can be used to support the designated uses which are generally found in State standards. The following section discusses the relationship between the criteria and individual use classifications. Where a water body is designated for more than one use, criteria necessary to protect the most sensitive use should be applied.

1. *Recreation:* Recreational uses of water include such activities as swimming, wading, boating and fishing. Although insufficient data exist on the effects of toxic pollutants resulting from exposure through such primary contact as swimming, section 304(a)(1) criteria based on human health effects may be used to support this designated use where fishing is included in the State definition of "recreation." In this situation only the portion of the criterion based on fish consumption should be used.

2. *Protection and Propagation of Fish and Other Aquatic Life:* The section 304(a)(1) criteria based on toxicity to aquatic life may be used directly to support this designated use.

3. *Agricultural and Industrial Uses:* The section 304(a)(1) criteria were not specifically developed to reflect the impact of pollutants on agricultural and industrial uses. However, the criteria developed for human health and aquatic life are sufficiently stringent to protect these other uses. States may establish criteria specifically designed to protect these uses.

4. *Public Water Supply:* The drinking water exposure component of the human health effects criteria can apply directly to this use classification or may be appropriately modified depending upon whether the specific water supply system falls within the auspices of the Safe Drinking Water Act's (SDWA) regulatory control, and the type and level of treatment imposed upon the supply before delivery to the consumer. The SDWA controls the presence of toxic pollutants in finished ("end-of-tap") drinking water. A brief description of relevant sections of this Act is necessary to explain how the SDWA will work in conjunction with section 304(a)(1) criteria in protecting human health from the effects of toxics due to consumption of water.

Pursuant to section 1412 of the SDWA, EPA has promulgated "National Interim Primary Drinking Water Standards" for certain organic and inorganic substances. These standards establish "maximum contaminant levels" ("MCLs") which specify the maximum permissible level of a contaminant in water which may be delivered to a user of a public water system now defined as serving a minimum of 25 people. MCLs are established based on consideration of a range of factors including not only the health effects of the contaminants but also technological and economic feasibility of the contaminants' removal from the supply. EPA is required to establish revised primary drinking water regulations based on the effects of a contaminant on human health, and include treatment capability, monitoring availability, and costs. Under Section 1401(1)(D)(i) of the SDWA, EPA is also allowed to establish the minimum quality criteria for water which may be taken into a public water supply system.

Section 304(a)(1) criteria provide estimates of pollutant concentrations protective of human health, but do not consider treatment technology, costs and other feasibility factors. The section 304(a)(1) criteria also include fish bioaccumulation and consumption factors in addition to direct human drinking water intake. These numbers were not developed to serve as "end of tap" drinking water standards, and they have no regulatory significance under the SDWA. Drinking water standards are established based on considerations, including technological and economic feasibility, not relevant to section 304(a)(1) criteria. Section 304(a)(1) criteria may be analogous to the recommended maximum contaminant levels (RMCLs) under section 1412(b)(1)(B) of the SDWA in which, based upon a report from the National Academy of Sciences, the Administrator should set target levels for contaminants in drinking water at which "no known or anticipated adverse effects occur and which allows an adequate margin of safety". RMCLs do not take treatment, cost, and other feasibility factors into consideration. Section 304(a)(1) criteria are, in concept, related to the health-based goals specified in the RMCLs. Specific mandates of the SDWA such as the consideration of multi-media exposure, as well as different methods for setting maximum contaminant levels under the two Acts, may result in differences between the two numbers.

MCLs of the SDWA, where they exist, control toxic chemicals in finished drinking water. However, because of variations in treatment and the fact that only a relatively small number of MCLs have been developed, ambient water criteria may be used by the States as a supplement to SDWA regulations. States will have the option of applying MCLs, section 304(a)(1) human health effects criteria, modified section 304(a)(1) criteria or controls more stringent than these three to protect against the effects of toxic pollutants by ingestion from drinking water.

For untreated drinking water supplies, States may control toxics in the ambient water through either use of MCLs (if they exist for the pollutants of concern), section 304(a)(1) human health effects criteria, or a more strigent contaminant level than the former two options.

For treated drinking water supplies serving less than 25 people, States may choose toxics control through application of MCLs (if they exist for the pollutants of concern and are attainable by the type of treatment) in the finished drinking water. States also have the options to control toxics in the ambient water by choosing section 304(a)(1.) criteria, adjusted section 304(a)(1) criteria resulting from the reduction of the direct drinking water exposure component in the criteria calculation to the extent that the treatment procedure reduces the level of pollutants, or a more stringent contaminant level than the former three options.

For treated drinking water supplies serving 25 people or greater, States *must* control toxics down to levels at least as stringent as MCLs (where they exist for

the pollutants of concern) in the finished drinking water. However, States also have the options to control toxics in the ambient water by choosing section 304(a)(1) criteria, adjusted section 304(a)(1) criteria resulting from the reduction of the direct drinking water exposure component in the criteria calculation to the extent that the treatment process reduces the level of pollutants, or a more stringent contaminant level than the former three options.

Inclusion of Specific Pollutants in State Standards:

To date, EPA has not required that a State address any specific pollutant in its standards. Although all States have established standards for most conventional pollutants, the treatment of toxic pollutants has been much less extensive. In the ANPRM, EPA suggested a policy under which States would be required to address a set of pollutants and incorporate specific toxic pollutant criteria into water quality standards. If the State failed to incorporate these criteria, EPA would promulgate the standards based upon these criteria pursuant to section 303(c)(4)(B).

In the forthcoming proposed revision to the water quality standard regulations, a significant change in policy will be proposed relating to the incorporation of certain pollutants in State water quality standards. This proposal will differ from the proposal made in the ANPRM. The ANPRM proposed an EPA-published list of pollutants for which States would have had to develop water quality standards. This list might have contained some (or all) of the 65 toxic pollutants. However, the revised water quality standards regulation will propose a process by which EPA will assist States in identifying specific toxic pollutants required for assessment for possible inclusion in State water quality standards. For these pollutants, States will have the option of adopting the published criteria or of adjusting those criteria based on site-specific analysis.

These pollutants would generally represent the greatest threat to sustaining a healthy, balanced ecosystem in water bodies or to human health due to exposure directly or indirectly from water. EPA is currently developing a process to determine which pollutants a State must assess for possible inclusion in its water quality standards. Relevant factors might include the toxicity of the pollutant, the frequency and concentration of its discharge, its geographical distribution, the breadth of data underlying the scientific assessment of its aquatic life and human health effects, and the technological and economic capacity to control the discharge of the pollutant. For some of the pollutants, all States may be required to assess them for possible inclusion in their standards. For others, assessment would be restricted to States or limited to specific water bodies where the pollutants pose a particular site-specific problem.

Criteria Modification Process

Flexibility is available in the application of these and any other valid water quality criteria to regulatory programs. Although in some cases they may be used by the States as developed, the criteria may be modified to reflect local environmental conditions and human exposure patterns before incorporation into programs such as water quality standards. If significant impacts of site-specific water quality conditions in the toxicities of pollutants can be demonstrated or significantly different exposure patterns of these pollutants to humans can be shown, section 304(a)(1) criteria may be modified to reflect these local conditions. The term "local" may refer to any appropriate geographic area where common aquatic environmental conditions or exposure patterns exist. Thus, "local" may signify a Statewide, regional, river reach, or entire river basin area. On the other hand, the criteria of some pollutants might be applicable nationwide without the need for adaptation to reflect local conditions. The degree of toxicity toward aquatic organisms and humans characteristic of these pollutants would not change significantly due to local water quality conditions.

EPA is examining a series of environmental factors or water quality parameters which might realistically be expected to affect the laboratory-derived water quality criterion recommendation for a specific pollutant. Factors such as hardness, pH, suspended solids, types of aquatic organisms present, etc. could impact on the chemical's effect in the aquatic environment. Therefore, local information can be assembled and analyzed to adjust the criterion recommendation if necessary.

The Guidelines for deriving criteria for the protection of aquatic life suggest several approaches for modifying the criteria. First, toxicity data, both acute and chronic, for local species could be substituted for some or all of the species used in deriving criteria for the water quality standard. The minimum data requirements should still be fulfilled in calculating a revised criterion. Second, criteria may be specifically tailored to a local water body by use of data from toxicity tests performed with that ambient water. A procedure such as th would account for local environmental conditions in formulating a criterion relevant to the local water body. Third site-specific water quality characteristics resulting in either enhancement or mitigation of aquatic life toxicity for the pollutant could be factored into final formulation of the criterion. Finally, the criteria may be made more stringent to ensure protection of an individual species no otherwise adequately protected by an of the three modification procedures previously mentioned.

EPA does not intend to have States assess every local stream segment an lake in the country on an individual basis before determining if an adjustment is necessary. Rather, it is envisioned that water bodies having similar hydrological, chemical, physical, and biological properties will be grouped for the purpose of criteria adjustment. The purpose of this effort to assist States in adapting the section 304(a) criteria to local conditions where needed, thereby precluding the setting arbitrary and perhaps unnecessarily stringent or underprotective criteria in a water body. In all cases, EPA will still be required, pursuant to section 303(c to determine whether the State water quality standards are consistent with the goals of the Act, including a determination of whether State-established criteria are adequate to support a designated use.

Criteria for the Protection of Aquatic Life

Interpretation of the Criteria

The aquatic life criteria issued toda are summarized in Appendix A of thi Federal Register notice. Criteria have been formulated by applying a set of Guidelines to a data base for each pollutant. The criteria for the protecti of aquatic life specify pollutant concentrations which, if not exceeded, should protect most, but not necessar all, aquatic life and its uses. The Guidelines specify that criteria shoul be based on an array of data from organisms, both plant and animal, occupying various trophic levels. Bas on these data, criteria can be derived which should be adequate to protect the types of organisms necessary to suppo an aquatic community.

The Guidelines are not designed t derive criteria which will protect all life stages of all species under all conditions. Generally some life stage one or more tested species, and

probably some untested species, will have sensitivities below the maximum value or the 24-hour average under some conditions and would be adversely affected if the highest allowable pollutant concentrations and the worst conditions existed for a long time. In actual practice, such a situation is not likely to occur and thus the aquatic community as a whole will normally be protected if the criteria are not exceeded. In any aquatic community there is a wide range of individual species sensitivities to the effects of toxic pollutants. A criterion adequate to protect the most susceptible life stage of the most sensitive species would in many cases be more stringent than necessary to protect the overall aquatic community.

The aquatic life criteria specify both maximum and 24-hour average values. The combination of the two values is designed to provide adequate protection of aquatic life and its uses from acute and chronic toxicity and bioconcentration without being as restrictive as a one-number criterion would have to be to provide the same amount of protection. A time period of 24 hours was chosen in order to ensure that concentrations not reach harmful levels for unacceptably long periods. Averaging for longer periods, such as a week or a month for example, could permit high concentrations to persist long enough to produce significant adverse effects. A 24-hour period was chosen instead of a slightly longer or shorter period in recognition of daily fluctuations in waste discharges and of the influence of daily cycles of sunlight and darkness and temperature on both pollutants and aquatic organisms.

The maximum value, which is derived from acute toxicity data, prevents significant risk of adverse impact to organisms exposed to concentrations above the 24-hour average. Merely specifying the average value over a specified time period is insufficient because concentrations of chemicals higher than the average value can kill or cause irreparable damage in short periods. Furthermore, for some chemicals the effect of intermittent high exposures is cumulative. It is therefore necessary to place an upper limit on pollutant concentrations to which aquatic organisms might be exposed. The two-number criterion is intended to describe the highest average ambient water concentration which will produce a water quality generally suited to the maintenance of aquatic life while restricting the extent and duration of the excursions over that average to levels which will not cause harm. The only way to assure the same degree of protection with a one-number criterion would be to use the 24-hour average as a concentration that is not to be exceeded at any time in any place.

Since some substances may be more toxic in freshwater than in saltwater, or vice versa, provision is made for deriving separate water quality criteria for freshwater and for saltwater for each substance. However, for some substances sufficient data may not be available to derive one or both of these criteria using the Guidelines.

Specific aquatic life criteria have not been developed for all of the 65 toxic pollutants. In those cases where there were insufficient data to allow the derivation of a criterion, narrative descriptions of apparent threshold levels for acute and/or chronic effects based on the available data are presented. These descriptions are intended to convey a sense of the degree of toxicity of the pollutant in the absence of a criterion recommendation.

Summary of the Aquatic Life Guidelines

The Guidelines for Deriving Water Quality Criteria for the Protection of Aquatic Life and its Uses were developed to describe an objective, internally consistent, and appropriate way of ensuring that water quality criteria for aquatic life would provide, on the average, a reasonable amount of protection without an unreasonable amount of overprotection or underprotection. The resulting criteria are not intended to provide 100 percent protection of all species and all uses of aquatic life all of the time, but they are intended to protect most species in a balanced, healthy aquatic community. The Guidelines are published as Appendix B of this Notice. Responses to public comments on these Guidelines are attached as Appendix D.

Minimum data requirements are identified in four areas: acute toxicity to animals (eight data points), chronic toxicity to animals (three data points), toxicity to plants, and residues. Guidance is also given for discarding poor quality data.

Data on acute toxicity are needed for a variety of fish and invertebrate species and are used to derive a Final Acute Value. By taking into account the number and relative sensitivities of the tested species, the Final Acute Value is designed to protect most, but not necessarily all, of the tested and untested species.

Data on chronic toxicity to animals can be used to derive a Final Chronic Value by two different means. If chronic values are available for a specified number and array of species, a final chronic value can be calculated directly. If not, an acute-chronic ratio is derived and then used with the Final Acute Value to obtain the Final Chronic Value.

The Final Plant Value is obtained by selecting the lowest plant toxicity value based on measured concentrations.

The Final Residue Value is intended to protect wildlife which consume aquatic organisms and the marketability of aquatic organisms. Protection of the marketability of aquatic organisms is, in actuality, protection of a use of that water body ("commercial fishery"). Two kinds of data are necessary to calculate the Final Residue Value: a bioconcentration factor (BCF) and a maximum permissible tissue concentration, which can be an FDA action level or can be the result of a chronic wildlife feeding study. For lipid soluble pollutants, the BCF is normalized for percent lipids and then the Final Residue Value is calculated by dividing the maximum permissible tissue concentration by the normalized BCF and by an appropriate percent lipid value. BCFs are normalized for percent lipids since the BCF measured for any individual aquatic species is generally proportional to the percent lipids in that species.

If sufficient data are available to demonstrate that one or more of the final values should be related to a water quality characteristic, such as salinity, hardness, or suspended solids, the final value(s) are expressed as a function of that characteristic.

After the four final values (Final Acute Value, Final Chronic Value, Final Plant Value, and Final Residue Value) have been obtained, the criterion is established with the Final Acute Value becoming the maximum value and the lowest of the other three values becoming the 24-hour average value. All of the data used to calculate the four final values and any additional pertinent information are then reviewed to determine if the criterion is reasonable. If sound scientific evidence indicates that the criterion should be raised or lowered, appropriate changes are made as necessary.

The present Guidelines have been revised from the earlier published versions (43 FR 21506, May 18, 1978; 43 FR 29028, July 5, 1978; 44 FR 15926, March 15, 1979). Details have been added in many places and the concept of a minimum data base has been incorporated. In addition, three adjustment factors and the species sensitivity factor have been deleted. These modifications were the result of the Agency's analysis of public comments and comments received from the Science Advisory Board on earlier

versions of the Guidelines. These comments and the Resultant modifications are addressed fully in Appendix D to this notice.

Criteria for the Protection of Human Health

Interpretation of the Human Health Criteria

The human health criteria issued today are summarized in Appendix A of this Federal Register notice. Criteria for the protection of human health are presented for 62 of the 65 pollutants based on their carcinogenic, toxic, or organoleptic (taste and odor) properties. The meanings and practical uses of the criteria values are distinctly different depending on the properties on which they are based.

The objective of the health assessment portions of the criteria documents is to estimate ambient water concentrations which, in the case of non-carcinogens, prevent adverse health effects in humans, and in the case of suspect or proven carcinogens, represent various levels of incremental cancer risk.

Health assessments typically contain discussions of four elements: Exposure, pharmacokinetics, toxic effects, and criterion formulation.

The exposure section summarizes information on exposure routes: ingestion directly from water, indirectly from consumption of aquatic organisms found in ambient water, other dietary sources, inhalation, and dermal contact. Exposure assumptions are used to derive human health criteria. Most criteria are based solely on exposure from consumption of water containing a specified concentration of a toxic pollutant and through consumption of aquatic organisms which are assumed to have bioconcentrated pollutants from the water in which they live. Other multimedia routes of exposure such as air, non-aquatic diet, or dermal are not factored into the criterion formulation for the vast majority of pollutants due to lack of data. The criteria are calculated using the combined aquatic exposure pathway and also using the aquatic organism ingestion exposure route alone. In criteria reflecting both the water consumption and aquatic organism ingestion routes of exposure, the relative exposure contribution varies with the propensity of a pollutant to bioconcentrate, with the consumption of aquatic organisms becoming more important as the bioconcentration factor (BCF) increases. As additional information on total exposure is assembled for pollutants for which criteria reflect only the two specified aquatic exposure routes, adjustments in water concentration values may be made. The Agency intends to publish guidance which will permit the States to identify significantly different exposure patterns for their populations. If warranted by the demonstration of significantly different exposure patterns, this will become an element of a process to adapt/modify human health-based criteria to local conditions, somewhat analogous to the aquatic life criteria modification process discussed previously. It is anticipated that States at their discretion will be able to set appropriate human health criteria based on this process.

The pharmacokinetics section reviews data on absorption, distribution, metabolism, and excretion to assess the biochemical fate of the compounds in the human and animal system. The toxic effects section reviews data on acute, subacute, and chronic toxicity, synergistic and antagonistic effects, and specific information on mutagenicity, teratogenicity, and carcinogenicity. From this review, the toxic effect to be protected against is identified taking into account the quality, quantity, and weight of evidence characteristic of the data. The criterion formulation section reviews the highlights of the text and specifies a rationale for criterion development and the mathematical derivation of the criterion number.

Within the limitations of time and resources, current published information of significance was incorporated into the human health assessments. Review articles and reports were used for data evaluation and synthesis. Scientific judgment was exercised in reviewing and evaluating the data in each criteria document and in identifying the adverse effects for which protective criteria were published.

Specific health-based criteria are developed only if a weight of evidence supports the occurrence of the toxic effect and if dose/response data exist from which criteria can be estimated.

Criteria for suspect or proven carcinogens are presented as concentrations in water associated with a range of incremental cancer risks to man. Criteria for non-carcinogens represent levels at which exposure to a single chemical is not anticipated to produce adverse effects in man. In a few cases, organoleptic (taste and odor) data form the basis for the criterion. While this type of criterion does not represent a value which directly affects human health, it is presented as an estimate of the level of a pollutant that will not produce unpleasant taste or odor either directly from water consumption or indirectly by consumption of aquatic organisms found in ambient waters. A criterion developed in this manner is judged to be as useful as other types of criteria in protecting designated water uses. In addition, where data are available, toxicity-based criteria are also presented for pollutants with derived organoleptic criteria. The choice of criteria used in water quality standards for these pollutants will depend upon the designated use to be protected. In the case of a multiple use water body, the criterion protecting the most sensitive use will be applied. Finally, for several pollutants no criteria are recommended due to a lack of information sufficient for quantitative criterion formulation.

Risk Extrapolation

Because methods do not now exist to establish the presence of a threshold for carcinogenic effects, EPA's policy is that there is no scientific basis for estimating "safe" levels for carcinogens. The criteria for carcinogens, therefore, state that the recommended concentration for maximum protection of human health is zero. In addition, the Agency has presented a range of concentrations corresponding to incremental cancer risks of 10^{-7} to 10^{-5} (one additional case of cancer in populations ranging from ten million to 100,000, respectively). Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Summary of the Human Health Guidelines

The health assessments and corresponding criteria published today were derived based on *Guidelines and Methodology Used in the Preparation of Health Effect Assessment Chapters of the Consent Decree Water Criteria Documents* (the Guidelines) developed by EPA's Office of Reserch and Development. The estimation of health risks associated with human exposure to environmental pollutants requires predicting the effect of low doses for up to a lifetime in duration. A combination of epidemiological and animal dose/response data is considered the preferred basis for quantitative criteria derivation. The complete Guidelines are presented as Appendix C. Major issues associated with these Guidelines and responses to public comments are presented as Appendix E.

No-effect (non-carcinogen) or specified risk (carcinogen) concentrations were estimated by extrapolation from animal toxicity or

human epidemiology studies using the following basic exposure assumptions: a 70-kilogram male person (*Report of the Task Group on Reference Man*, International Commission for Radiation Protection, November 23, 1957) as the exposed individual; the average daily consumption of freshwater and estuarine fish and shellfish products equal to 6.5 grams/day; and the average ingestion of two liters/day of water (*Drinking Water and Health*, National Academy of Sciences, National Research Council, 1977). Criteria based on these assumptions are estimated to be protective of an adult male who experiences average exposure conditions.

Two basic methods were used to formulate health criteria, depending on whether the prominent adverse effect was cancer or other toxic manifestations. The following sections detail these methods.

Carcinogens

Extrapolation of cancer responses from high to low doses and subsequent risk estimation from animal data is performed using a linearized multi-stage model. This procedure is flexible enough to fit all monotonically-increasing dose response data, since it incorporates several adjustable parameters. The multi-stage model is a linear non-threshold model as was the "one-hit" model originally used in the proposed criteria documents. The linearized multi-stage model and its characteristics are described fully in Appendix C. The linear non-threshold concept has been endorsed by the four agencies in the Interagency Regulatory Liaison Group and is less likely to underestimate risk at the low doses typical of environmental exposure than other models that could be used. Because of the uncertainties associated with dose response, animal-to-human extrapolation and other unknown factors, because of the use of average exposure assumptions, and because of the serious public health consequences that could result if risk were underestimated, EPA believes that it is prudent to use conservative methods to estimate risk in the water quality criteria program. The linearized multistage model is more systematic and invokes fewer arbitrary assumptions than the "one-hit" procedure previously used.

It should be noted that extrapolation models provide estimates of risk since a variety of assumptions are built into any model. Models using widely different assumptions may produce estimates ranging over several orders of magnitude. Since there is at present no way to demonstrate the scientific validity of any model, the use of risk extrapolation models is a subject of debate in the scientific community. However, risk extrapolation is generally recognized as the only tool available at this time for estimating the magnitude of health hazards associated with non-threshold toxicants and has been endorsed by numerous Federal agencies and scientific organizations, including EPA's Carcinogen Assessment Group, the National Academy of Sciences, and the Interagency Regulatory Liaison Group as a useful means of assessing the risks of exposure to various carcinogenic-pollutants.

Non-Carcinogens

Health criteria based on toxic effects of pollutants other than carcinogenicity are estimates of concentrations which are not expected to produce adverse effects in humans. They are based upon Acceptable Daily Intake (ADI) levels and are generally derived using no-observed-adverse-effect-level (NOAEL) data from animal studies although human data are used wherever available. The ADI is calculated using safety factors to account for uncertainties inherent in extrapolation from animal to man. In accordance with the National Research Council recommendations (*Drinking Water and Health*, National Academy of Sciences, National Research Council, 1977), safety factors of 10, 100, or 1,000 are used depending on the quality and quantity of data. In some instances extrapolations are made from inhalation studies or limits to approximate a human response from ingestion using the Stokinger-Woodward model (Journal of American Water Works Association, 1958). Calculations of criteria from ADIs are made using the standard exposure assumptions (2 liters of water, 6.5 grams of edible aquatic products, and an average body weight of 70 kg).

Dated: October 24, 1980.

Douglas M. Costle,

Administrator.

Appendix A—Summary of Water Quality Criteria

Acenaphthene

Freshwater Aquatic Life

The available data for acenaphthene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 1,700 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of acenaphthene to sensitive freshwater aquatic animals but toxicity to freshwater algae occur at concentrations as low as 520 μg/l.

Saltwater Aquatic Life

The available data for acenaphthene indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 970 and 710 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested. Toxicity to algae occurs at concentrations as low as 500 μg/l.

Human Health

Sufficient data is not available for acenaphthene to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 20 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Acrolein

Freshwater Aquatic Life

The available data for acrolein indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 68 and 21 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for acrolein indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 55 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of acrolein to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of acrolein ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 320 μg/l.

For the protection of human health from the toxic properties of acrolein ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 780 μg/l.

Acrylonitrile

Freshwater Aquatic Life

The available data for acrylonitrile indicate that acute toxicity to freshwater aquatic life occurs at concentrations as

low as 7,550 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of acrylonitrile to sensitive freshwater aquatic life but mortality occurs at concentrations as low as 2,600 μg/l with a fish species exposed for 30 days.

Saltwater Aquatic Life

Only one saltwater species has been tested with acrylonitrile and no statement can be made concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of acrylonitrile through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .58 μg/l, .058 μg/l and .006 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 6.5 μg/l, .65 μg/l, and .065 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Aldrin-Dieldrin

Dieldrin

Freshwater Aquatic Life

For dieldrin the criterion to protect fresh water aquatic life as derived using the Guidelines is 0.0019 μg/l as a 24-hour average and the concentration should not exceed 2.5 μg/l at any time.

Saltwater Aquatic Life

For dieldrin the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0019 μg/l as a 24-hour average and the concentration should not exceed 0.71 μg/l at any time.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of dieldrin through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .71 ng/l, .071 ng/l, and .0071 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are .76 ng/l, .076 ng/l, and .0076 ng/l respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Aldrin

Freshwater Aquatic Life

For freshwater aquatic life the concentration of aldrin should not exceed 3.0 μg/l at any time. No data are available concerning the chronic toxicity of aldrin to sensitive freshwater aquatic life.

Saltwater Aquatic Life

For saltwater aquatic life the concentration of aldrin should not exceed 1.3 μg/l at any time. No data are available concerning the chronic toxicity of aldrin to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of aldrin through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .74 ng/l, .074 ng/l, and .0074 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are .79 ng/l, .079 ng/l, and .0079 ng/l, respectively. Other concentrations respresenting different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Antimony

Freshwater Aquatic Life

The available data for antimony indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 9,000 and 1,6 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested. Toxicity to algae occurs at concentrations as low as 610 μg/l.

Saltwater Aquatic Life

No saltwater organisms have been adequately tested with antimony, and no statement can be made concerning acute or chronic toxicity.

Human Health

For the protection of human health from the toxic properties of antimony ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 146 μg/l.

For the protection of human health from the toxic properties of antimony ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 45,000 μg

Arsenic

Freshwater Aquatic Life

For freshwater aquatic life the concentration of total recoverable trivalent inorganic arsenic should not exceed 440 μg/l at any time. Short-te effects on embryos and larvae of aqu vertebrate species have been shown occur at concentrations as low as 40 μg/l.

Saltwater Aquatic Life

The available data for total recoverable trivalent inorganic arsenic indicate that acute toxicity to saltwa aquatic life occurs at concentrations low as 508 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concer the chronic toxicity of trivalent inorganic arsenic to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogen effects due to exposure of arsenic through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration sh be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels w h may result in incremental increase c cancer risk over the lifetime are

estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 22 ng/l, 2.2 ng/l, and .22 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 175 ng/l, 17.5 ng/l, and 1.75 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Asbestos

Freshwater Aquatic Life

No freshwater organisms have been tested with any asbestiform mineral and no statement can be made concerning acute or chronic toxicity.

Saltwater Aquatic Life

No saltwater organisms have been tested with any asbestiform mineral and no statement can be made concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of asbestos through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 300,000 fibers/1, 30,000 fibers/1, and 3,000 fibers/1, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Benzene

Freshwater Aquatic Life

The available data for benzene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 5,300 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of benzene to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for benzene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 5,100 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of benzene to sensitive saltwater aquatic life, but adverse effects occur at concentrations as low as 700 μg/l with a fish species exposed for 168 days.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of benzene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 6.6 μg/l, .66 μg/l, and .066 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 400 μg/l, 40.0 μg/l, and 4.0 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Benzidine

Freshwater Aquatic Life

The available data for benzidine indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 2,500 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of benzidine to sensitive freshwater aquatic life.

Saltwater Aquatic Life

No saltwater organisms have been tested with benzidine and no statement can be made concerning acute and chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of benzidine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 1.2 ng/1, .12 ng/1, and .01 ng/1, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 5.3 ng/1, .53 ng/1, and .05 ng/1, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Beryllium

Freshwater Aquatic Life

The available data for beryllium indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 130 and 5.3 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested. Hardness has a substantial effect on acute toxicity.

Salt water Aquatic Life

The limited saltwater data base available for beryllium does not permit any statement concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of beryllium through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 37 ng/l, 3.7 ng/l, and .37 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 641 ng/l, 64.1 ng/l, and 6.41 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Cadmium

Freshwater Aquatic Life

For total recoverable cadmium the criterion (in μg/l) to protect freshwater aquatic life as derived using the Guidelines is the numerical value given

by $e^{(1.00\ [\ln(\text{hardness})]-3.51)}$ as a 24-hour average and the concentration (in μg/l) should not exceed the numerical value given by $e^{(1.00\ [\ln(\text{hardness})]-2.73)}$ at any time. For example, a hardnesses of 50, 100, and 200 mg/l as $CaCO_3$ the criteria are 0.012, 0.025, and 0.051 μg/l, respectively, and the concentration of total recoverable cadmium should not exceed 1.5, 3.0 and 6.3 μg/l, respectively, at any time.

Saltwater Aquatic Life

For total recoverable cadmium the criterion to protect saltwater aquatic life as derived using the Guidelines is 4.5 μg/l as a 24-hour average and the concentration should not exceed 59 μg/l at any time.

Human Health

The ambient water quality criterion for cadmium is recommended to be identical to the existing drinking water standard which is 10 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Carbon Tetrachloride

Freshwater Aquatic Life

The available date for carbon tetrachloride indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 35,200 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of carbon tetrachloride to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for carbon tetrachloride indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 50,000 μg/l and would occur at lower concentrations among species that are more sensitive that those tested. No data are available concerning the chronic toxicity of carbon tetrachloride to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of carbon tetrachloride through ingestion of contaminated water and contaminated aquatic organisms the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 4.0μg/l, .40 μg/l, and .04 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 69.4 μg/l, 6.94 μg/l, and .69 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Chlordane

Freshwater Aquatic Life

For chlordane the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.0043 μg/l as a 24-hour average and the concentration should not exceed 2.4 μg/l at any time.

Saltwater Aquatic Life

For chlordane the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0040 μg/l as a 24-hour average and the concentration should not exceed 0.09 μg/l at any time.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of chlordane through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 4.6 ng/l, .46 ng/l, and .046 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 4.8 ng/l, .48 ng/l, and .048 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Chlorinated Benzenes

Freshwater Aquatic Life

The available data for chlorinated benzenes indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 250 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of the more toxic of the chlorinated benzenes to sensitive freshwater aquatic life but toxicity occurs at concentrations as low as 50 μg/l for a fish species exposed for 7.5 days.

Saltwater Aquatic Life

The available data for chlorinated benzenes indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 160 and 129 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of hexachlorobenzene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6} and 10^{-7}. The corresponding recommended criteria are 7.2 ng/l, .72 ng/l, and .072 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 7.4 ng/l, .74 ng/l, and .074 ng/l, respectively.

For the protection of human health from the toxic properties of 1,2,4,5-tetrachlorobenzene ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 38 μg/l.

For the protection of human health from the toxic properties of 1,2,4,5-tetrachlorobenzene ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 48 μg/l.

For the protection of human health from the toxic properties of pentachlorobenzene ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 74 μg/l.

For the protection of human health from the toxic properties of pentachlorobenzene ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 85 μg/l.

Using the present guidelines, a satisfactory criterion cannot be deriv-

at this time due to the insufficiency in the available data for trichlorobenzene.

For comparison purposes, two approaches were used to derive criterion levels for monochlorobenzene. Based on available toxicity data, for the protection of public health, the derived level is 488 μg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 20 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Chlorinated Ethanes

Freshwater Aquatic Life

The available freshwater data for chlorinated ethanes indicate that toxicity increases greatly with increasing chlorination, and that acute toxicity occurs at concentrations as low as 118,000 μg/l for 1,2-dichloroethane, 18,000 μg/l for two trichloroethanes, 9,320 μg/l for two tetrachloroethanes, 7,240 μg/l for pentachloroethane, and 980 μg/l for hexachloroethane. Chronic toxicity occurs at concentrations as low as 20,000 μg/l for 1,2-dichloroethane, 9,400 μg/l for 1,1,2-trichloroethane, 2,400 μg/l for 1,1,2,2,-tetrachloroethane, 1,100 μg/l for pentachloroethane, and 540 μg/l for hexachloroethane. Acute and chronic toxicity would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available saltwater data for chlorinated ethanes indicate that toxicity increases greatly with increasing chlorination and that acute toxicity to fish and invertebrate species occurs at concentrations as low as 113,000 μg/l for 1,2-dichloroethane, 31,200 μg/l for 1,1,1-trichloroethane, 9,020 μg/l for 1,1,2,2-tetrachloroethane, 390 μg/l for pentachloroethane, and 940 μg/l for hexachloroethane. Chronic toxicity occurs at concentrations as low as 281 μg/l for pentachloroethane. Acute and chronic toxicity would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 1,2-dichloroethane through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 9.4 μg/l, .94 μg/l, and .094 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 2,430 μg/l, 243 μg/l, and 24.3 μg/l respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the protection of human health from the toxic properties of 1,1,1-trichloroethane ingested through water and contaminated aquatic organism, the ambient water criterion is determined to be 18.4 mg/l.

For the protection of human health from the toxic properties of 1,1,1,-trichloroethane ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 1.03 g/l.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 1,1,2-trichloroethane through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 6.0 μg/l, .6 μg/l, and .06 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 418 μg/l, 41.8 μg/l, and 4.18 μg/l respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 1,1,2,2-tetrachloroethane through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 1.7 μg/l, .17 μg/l, and .017 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 107 μg/l, 10.7 μg/l, and 1.07 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of hexachloroethane through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 19 μg/l, 1.9 μg/l, and .19 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 87.4 μg/l, 8.74 μg/l, and .87 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for monochloroethane.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for 1,1,-dichloroethane.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for 1,1,1,2-tetrachloroethane.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for pentachloroethane.

Chlorinated Naphthalenes

Freshwater Aquatic Life

The available data for chlorinated naphthalenes indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 1,600 μg/l and would occur at lower concentrations among species that are

more sensitive than those tested. No data are available concerning the chronic toxicity of chlorinated naphthalenes to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for chlorinated napthalenes indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 7.5 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of chlorinated naphthalenes to sensitive saltwater aquatic life.

Human Health

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for chlorinated napthalenes.

Chlorinated Phenols

Freshwater Aquatic Life

The available freshwater data for chlorinated phenols indicate that toxicity generally increases with increasing chlorination, and that acute toxicity occurs at concentrations as low as 30 μg/l for 4-chloro-3-methylphenol to greater than 500,000 μg/l for other compounds. Chronic toxicity occurs at concentrations as low as 970 μg/l for 2,4,6-trichlorophenol. Acute and chronic toxicity would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available saltwater data for chlorinated phenols indicate that toxicity generally increases with increasing chlorination and that acute toxicity occurs at concentrations as low as 440 μg/l for 2,3,5,6-tetrachlorophenol and 29,700 μg/l for 4-chlorophenol. Acute toxicity would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of chlorinated phenols to sensitive saltwater aquatic life.

Human Health

Sufficient data is not available for 3-monochlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 0.1 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 4-monochlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 0.1 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 2,3-dichlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is .04 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 2,5-dichlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is .5 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 2,6-dichlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is .2 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 3,4-dichlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is .3 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 2,3,4,6-tetrachlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 1 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

For comparison purposes, two approaches were used to derive criterion levels for 2,4,5-trichlorophenol. Based on available toxicity data, for the protection of public health, the derived level is 2.6 mg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 1.0 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 2,4,6-trichlorophenol through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 12 μg/l, 1.2 μg/l, and .12 μg/l respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 36 μg/l, 3.6 μg/l, and .36 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 2 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 2-methyl-4-chlorophenol to derive a level which would protect against any potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 1800 μg/l. It should be

recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 3-methyl-4-chlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 3000 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Sufficient data is not available for 3-methyl-6-chlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 20 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Chloroalkyl Ethers

Freshwater Aquatic Life

The available data for chloroalkyl ethers indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 238,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of chloroalkyl ethers to sensitive freshwater aquatic life.

Saltwater Aquatic Life

No saltwater organisms have been tested with any chloroalkyl ether and no statement can be made concerning acute and chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of bis-(chloromethyl)-ether through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .038 ng/l, .0038 ng/l, and .00038 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 18.4 ng/l, 1.84 ng/l, and .184 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of bis (2-chloroethyl) ether through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .3 μg/l, .03 μg/l, and .003 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 13.6 μg/l, 1.36 μg/l, and .136 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the protection of human health from the toxic properties of bis (2-chloroisopropyl) ether ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 34.7 μg/l.

For the protection of human health from the toxic properties of bis (2-chloroisopropyl) ether ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 4.36 mg/l.

Chloroform

Freshwater Aquatic Life

The available data for choloroform indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 28,900 μg/l, and would occur at lower concentrations among species that are more sensitive than the three tested species. Twenty-seven-day LC50 values indicate that chronic toxicity occurs at concentrations as low as 1,240 μg/l, and could occur at lower concentrations among species or other life stages that are more sensitive than the earliest life cycle stage of the rainbow trout.

Saltwater Aquatic Life

The data base for saltwater species is limited to one test and no statement can be made concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of chloroform through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 1.90 μg/l, .19 μg/l, and .019 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 157 μg/l, 15.7 μg/l, and 1.57 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

2-Chlorophenol

Freshwater Aquatic Life

The availabe data for 2-chlorophenol indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 4,380 μg/l and would occur at lower concentrations among species that are more sensitive that those tested. No definitive data are available concerning the chronic toxicity of 2-chlorophenol to sensitive freshwater aquatic life but flavor impairment occurs in one species of fish at concentrations as low as 2,000 μg/l.

Saltwater Aquatic Life

No saltwater organisms have been tested with 2-chlorophenol and no statement can be made concerning acute and chronic toxicity.

Human Health

Sufficient data is not available for 2-chlorophenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 0.1 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no

demonstrated relationship to potential adverse human health effects.

Chromium

Freshwater Aquatic Life

For total recoverable hexavalent chromium the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.29 μg/l as a 24-hour average and the concentration should not exceed 21 μg/l at any time.

For freshwater aquatic life the concentration (in μg/l) of total recoverable trivalent chromium should not exceed the numerical value given by "e(1.08[ln(hardness)]+3.48)" at any time. For example, at hardnesses of 50, 100 and 200 mg/l as $CaCO_3$ the concentration of total recoverable trivalent chromium should not exceed 2,200, 4,700, and 9,900 μg/l, respectively, at any time. The available data indicate that chronic toxicity to freshwater aquatic life occurs at concentrations as low a 44 μg/l and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

For total recoverable hexavalent chromium the criterion to protect saltwater aquatic life as derived using the Guidelines is 18 μg/l as a 24-hour average and the concentration should not exceed 1,260 μg/l at any time.

For total recoverable trivalent chromium, the availabe data indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 10,300 μg/l, and would occur at lower concentrations amoung species that are more sensitive than those tested. No data are available concerning the chronic toxicity of trivalent chromium to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of Chromium III ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 170 mg/l.

For the protection of human health from the toxic properties of Chromium III ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 3433 mg/l.

The ambient water quality criterion for total Chromium VI is recommended to be identical to the existing drinking water standard which is 50 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Copper

Freshwater Aquatic Life

For total recoverable copper the criterion to protect freshwater aquatic life as derived using the Guidelines is 5.6 μg/l as a 24-hour average and the concentration (in μg/l) should not exceed the numerical value given by e(0.94[ln(hardness)]-1.23) at any time. For example, at hardnesses of 50, 100, and 200 mg/l $CaCO_3$ the concentration of total recoverable copper should not exceed 12, 22, and 43 μg/l at any time.

Saltwater Aquatic Life

For total recoverable copper the criterion to protect saltwater aquatic life as derived using the Guidelines is 4.0 μg/l as a 24-hour average and the concentration should not exceed 23 μg/l at any time.

Human Health

Sufficient data is not available for copper to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 1 mg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Cyanide

Freshwater Aquatic Life

For free cyanide (sum of cyanide present as HCN and CN^-, expressed as CN) the criterion to protect freshwater aquatic life as derived using the Guidelines is 3.5 μg/l as a 24-hour average and the concentration should not exceed 52 μg/l at any time.

Saltwater Aquatic Life

The available data for free cyanide (sum of cyanide present as HCN and CN^-, expressed as CN) indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 30 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. If the acute-chronic ratio for saltwater organisms is similar to that for freshwater organisms, chronic toxicity would occur at concentrations as low as 2.0 μg/l for the tested species and at lower concentrations among species that are more sensitive than those tested.

Human Health

The ambient water quality criterion for cyanide is recommended to be identical to the existing drinking water standard which is 200 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

DDT and Metabolites

Freshwater Aquatic Life

DDT

For DDT and its metabolites the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.0010 μg/l as a 24-hour average and concentration should not exceed 1.1 μg/l at any time.

TDE

The available data for TDE indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 0.6 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of TDE to sensitive freshwater aquatic life.

DDE

The available data for DDE indicate that acute toxicity to freshwater aquat life occurs at concentrations as low as 1,050 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of DDE to sensitive freshwater aquatic life.

Saltwater Aquatic Life

DDT

For DDT and its metabolites the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0010 μg/l as a 24-hour average and the concentration should not exceed 0.13 μg/l at any time.

TDE

The available data for TDE indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 3.6 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the

chronic toxicity of TDE to sensitive saltwater aquatic life.

DDE

The available data for DDE indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 14 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of DDE to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of DDT through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .24 ng/l, .024 ng/l, and .0024 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are .24 ng/l, .024 ng/l, and .0024 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment of an "acceptable" risk level.

Dichlorobenzenes

Freshwater Aquatic Life

The available data for dichlorobenzenes indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 1,120 and 763 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for dichlorobenzenes indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 1,970 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of dichlorobenzenes to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of dichlorobenzenes (all isomers) ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 400 μg/l.

For the protection of human health from the toxic properties of dichlorobenzenes (all isomers) ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 2.6 mg/l.

Dichlorobenzidines

Freshwater Aquatic Life

The data base available for dichlorobenzidines and freshwater organisms is limited to one test on bioconcentration of 3,3'-dichlorobenzidine and no statement can be made concerning acute or chronic toxicity.

Saltwater Aquatic Life

No saltwater organisms have been tested with any dichlorobenzidine and no statement can be made concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of dichlorobenzidine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero base on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .103 μg/l, .0103 μg/l, and .00103 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are .204 μg/l, .0204 μg/l, and .00204 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Dichloroethylenes

Freshwater Aquatic Life

The available data for dichloroethylenes indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 11,600 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of dichlorethylenes to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for dichlorethylenes indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 224,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity dichloroethylenes to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 1,1-dichloroethylene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .33 μg/l, .033 μg/l, and .0033 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 18.5 μg/l, 1.85 μg/l, and .185 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficency in the available data for 1,2-dichloroethylene.

2,4-Dichlorophenol

Freshwater Aquatic Life

The available data for 2,4-dichlorophenol indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 2,020 and 365 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive that those tested. Mortality to early life stages of one species of fish occurs at concentrations as low as 70 μg/l.

Saltwater Aquatic Life

Only one test has been conducted with saltwater organisms on 2,4-dichlorophenol and no statement can be made concerning acute or chronic toxicity.

Human Health

For comparison purposes, two approaches were used to derive criterion levels for 2,4-dichlorophenol.

Based on available toxicity data, for the protection of public health, the derived level is 3.09 mg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 0.3 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Dichloropropanes/Dichloropropenes

Freshwater Aquatic Life

The available data for dichloropropanes indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 23,000 and 5,700 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

The available data for dichloropropenes indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 6,060 and 244 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for dichloropropanes indicate that acute and chronic toxicity to saltwater aquatic life occurs at concentrations as low as 10,300 and 3,040 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

The available data for dichloropropenes indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low a as 790 μg/l, and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of dichloropropenes to sensitive saltwater aquatic life.

Human Health

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for dichloropropanes.

For the protection of human health from the toxic properties of dichloropropenes ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 87 μg/l.

For the protection of human health from the toxic properties of dichloropropenes ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 14.1 mg/l.

2,4-Dimethylphenol

Freshwater Aquatic Life

The available data for 2,4-dimethylphenol indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 2,120 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of dimethylphenol to sensitive freshwater aquatic life.

Saltwater Aquatic Life

No saltwater organisms have been tested with 2,4-dimethylphenol and no statement can be made concerning acute and chronic toxicity.

Human Health

Sufficient data are not available for 2,4-dimethylphenol to derive a level which would protect against the potential toxicity of this compound. Using available organoleptic data, for controlling undersirable taste and odor quality of ambient water, the estimated level is 400 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

2,4-Dinitrotoluene

Freshwater Aquatic Life

The available data for 2,4-dinitrotoluene indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 330 and 230 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for 2,4-dinitrotoluenes indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 590 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of 2,4-dinitrotoluenes to sensitive saltwater aquatic life but a decrease in algal cell numbers occurs at concentrations as low as 370 μg/l.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 2,4-dinitrotoluene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 1.1 μg/l, 0.11 μg/l, and 0.011 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 91 μg/l, 9.1 μg/l, and 0.91 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

1,2-Diphenylhydrazine

Freshwater Aquatic Life

The available data for 1,2-diphenylhydrazine indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 270 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of 1,2-diphenylhydrazine to sensitive freshwater aquatic life.

Saltwater Aquatic Life

No saltwater organisms have been tested with 1,2-diphenylhydrazine and no statement can be made concerning acute and chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of 1,2-diphenylhydrazine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 422 ng/l, 42 ng/l, and 4 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 5.8 μg/l, 0.58 μg/l, and 0.058 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not

represent an Agency judgment on an "acceptable" risk level.

Endosulfan

Freshwater Aquatic Life

For endosulfan the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.056 μg/l as a 24-hour average and the concentration should not exceed 0.22 μg/l at any time.

Saltwater Aquatic Life

For endosulfan the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0087 μg/l as a 24-hour average and the concentration should not exceed 0.034 μg/l at any time.

Human Health

For the protection of human health from the toxic properties of endosulfan ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 74 μg/l.

For the protection of human health from the toxic properties of endosulfan ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 159 μg/l.

Endrin

Freshwater Aquatic Life

For endrin the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.0023 μg/l as a 24-hour average and the concentration should not exceed 0.18 μg/l at any time.

Saltwater Aquatic Life

For endrin the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0023 μg/l as a 24-hour average and the concentration should not exceed 0.037 μg/l at any time.

Human Health

The ambient water quality criterion for endrin is recommended to be identical to the existing drinking water standard which is 1 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Ethylbenzene

Freshwater Aquatic Life

The available data for ethylbenzene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 32,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of ethylbenzene to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for ethylbenzene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 430 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of ethylbenzene to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of ethylbenzene ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 1.4 mg/l.

For the protection of human health from the toxic properties of ethylbenzene ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 3.28 mg/l.

Fluoranthene

Freshwater Aquatic Life

The available data for fluoranthene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 3980 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of fluoranthene to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for fluoranthene indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 40 and 16 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For the protection of human health from the toxic properties of fluoranthene ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 42 μg/l.

For the protection of human health from the toxic properties of fluoranthene ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 54 μg/l.

Haloethers

Freshwater Aquatic Life

The available data for haloethers indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 360 and 122 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

No saltwater organisms have been tested with any haloether and no statement can be made concerning acute or chronic toxicity.

Human Health

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for haloethers.

Halomethanes

Freshwater Aquatic Life

The available data for halomethanes indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 11,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of halomethanes to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for halomethanes indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 12,000 and 8,400 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested. A decrease in algal cell numbers occurs at concentrations as low as 11,500 μg/l.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of chloromethane, bromomethane, dichloromethane, bromodichloromethane, tribromomethane, dichlorodifluoromethane, trichlorofluoromethane, or combinations of these chemicals through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6} and 10^{-7}. The corresponding criteria are

1.9 μg/l, 0.19 μg/l, and 0.019 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 157 μg/l, 15.7 μg/l, and 1.57 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Heptachlor

Freshwater Aquatic Life

For heptachlor the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.0038 μg/l as a 24-hour average and the concentration should not exceed 0.52 μg/l at any time.

Saltwater Aquatic Life

For heptachlor the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.0036 μg/l as a 24-hour average and the concentration should not exceed 0.053 μg/l at any time.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of heptachlor through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 2.78 ng/l, .28 ng/l, and .028 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 2.85 ng/l, .29 ng/l, and .029 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Hexachlorobutadiene

Freshwater Aquatic Life

The available data for hexachlorobutadiene indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 90 and 9.3 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for hexachlorobutadiene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 32 μg/l and would occur at lower concentrations among species that are more sensitive that those tested. No data are available concerning the chronic toxicity of hexachlorobutadiene to sensitive saltwater aquatic life

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of hexachlorobutadiene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 4.47 μg/l, 0.45 μg/l, and 0.045 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 500 μg/l, 50 μg/l, and 5 μg/l respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Hexachlorocyclohexane

Lindane

Freshwater Aquatic Life

For Lindane the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.080 μg/l as a 24-hour average and the concentration should not exceed 2.0 μg/l at any time.

Saltwater Aquatic Life

For saltwater aquatic life the concentration of lindane should not exceed 0.16 μg/l at any time. No data are available concerning the chronic toxicity of lindane to sensitive saltwater aquatic life.

BHC

Freshwater Aquatic Life

The available data for a mixture of isomers of BHC indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 100 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of a mixture of isomers of BHC to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for a mixture of isomers of BHC indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 0.34 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of a mixture of isomers of BHC to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of alpha-HCH through ingestion of contaminated wat and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 92 ng/l, 9.2 ng/l, and .92 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 310 ng/l, 31.0 ng/l, and 3.1 ng/l respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of huma health from the potential carcinogenic effects due to exposure of beta-HCH through ingestion of contaminated wate and contaminated aquatic organisms, the ambient water concentration shoul be zero based on the non-threshold assumption for this chemical. However zero level may not be attainable at the present time. Therefore, the levels whi may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 163 ng/l, 16.3 ng/l, and 1.63 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only excluding consumption of water, the levels are 547 ng/l, 54.7 ng/l, and 5.47 ng/l, respectively. Other concentrations representing different risk levels may b calculated by use of the Guidelines. Th risk estimate range is presented for information purposes and does not

represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of tech-HCH through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 123 ng/l, 12.3 ng/l, and 1.23 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 414 ng/l, 41.4 ng/l, and 4.14 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of gamma-HCH through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentrations should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 186 ng/l, 18.6 ng/l, and 1.86 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 625 ng/l, 62.5 ng/l, 6.25 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for delta-HCH.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for epsilon-HCH.

Hexachlorocyclopentadiene

Freshwater Aquatic Life

The available data for hexachlorocyclopentadiene indicate that acute and chronic toxicity to freshwater aquatic life occurs at concentrations as low as 7.0 and 5.2 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data to hexachlorocyclopentadiene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 7.0 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of hexachlorocyclopentadiene to sensitive saltwater aquatic life.

Human Health

For comparison purposes, two approaches were used to derive criterion levels for hexachlorocyclopentadiene. Based on available toxicity data, for the protection of public health, the derived level is 206 μg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 1.0 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Isophorone

Freshwater Aquatic Life

The available data for isophorone indicate that acute toxicity to freshwater aquatic life ocurs at concentrations as low as 117,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of isophorone to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for isophorone indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 12,900 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of isophorone to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of isophorone ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 5.2 mg/l.

For the protection of human health from the toxic properties of isophorone ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 520 mg/l.

Lead

Freshwater Aquatic Life

For total recoverable lead the criterion (in μg/l) to protect freshwater aquatic life as derived using the Guidelines is the numerical value given by $e^{(2.35[\ln(\text{hardness})]-9.48)}$ as a 24-hour average and the concentration (in μg/l) should not exceed the numerical value given by $e^{(1.22[\ln(\text{hardness})]-0.47)}$ at any time. For example, at hardnesses of 50, 100, and 200 mg/l as $CaCO_3$ the criteria are 0.75, 3.8, and 20 μg/l, respectively, as 24-hour averages, and the concentrations should not exceed 74, 170, and 400 μg/l, respectively, at any time.

Saltwater Aquatic Life

The available data for total recoverable lead indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 668 and 25 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

The ambient water quality criterion for lead is recommended to be identical to the existing drinking water standard which is 50 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective to human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Mercury

Freshwater Aquatic Life

For total recoverable mercury the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.00057 μg/l as a 24-hour average and the concentration should not exceed 0.0017 μg/l at any time.

Saltwater Aquatic Life

For total recoverable mercury the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.025 μg/l as a 24-hour average and the concentration should not exceed 3.7 μg/l at any time.

Human Health

For the protection of human health from the toxic properties of mercury

ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 144 ng/l.

For the protection of human health from the toxic properties of mercury ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 146 ng/l.

Note.—These values include the consumption of freshwater, estuarine, and marine species.

Naphthalene

Freshwater Aquatic Life

The available data to naphthalene indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 2,300 and 620 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for naphthalene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 2,350 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of naphthalene to sensitive saltwater aquatic life.

Human Health

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for naphthalene.

Nickel

Freshwater Aquatic Life

For total recoverable nickel the criterion (in μg/l) to protect freshwater aquatic life as derived using the Guidelines is the numerical value given by e(0.76 [ln (hardness)] +1.06) as a 24-hour average and the concentration (in μg/l) should not exceed the numerical value given by e(0.76[ln (hardness)] + 4.02) at any time. For example, at hardnesses of 50, 100, and 200 mg/l as $CaCO_3$ the criteria are 56, 96, and 160 μg/l, respectively, as 24-hour averages, and the concentrations should not exceed 1,100, 1,800, and 3,100 μg/l, respectively, at any time.

Saltwater Aquatic Life

For total recoverable nickel the criterion to protect saltwater aquatic life as derived using the Guidelines is 7.1 μg/l as a 24-hour average and the concentration should not exceed 140 μg/l at any time.

Human Health

For the protection of human health from the toxic properties of nickel ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 13.4 μg/l.

For the protection of human health from the toxic properties of nickel ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 100 μg/l.

Nitrobenzene

Freshwater Aquatic Life

The available data for nitrobenzene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 27,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No definitive data are available concerning the chronic toxicity of nitrobenzene to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for nitrobenzene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 6,680 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of nitrobenzene to sensitive saltwater aquatic life.

Human Health

For comparison purposes, two approaches were used to derive criterion levels for nitrobenzene. Based on available toxicity data, for the protection of public health, the derived level is 19.8 mg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 30 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have no demonstrated relationship to potential adverse human health effects.

Nitrophenols

Freshwater Aquatic Life

The available data for nitrophenols indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 230 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of nitrophenols to sensitive freshwater aquatic life but toxicity to one species of algae occurs at concentrations as low as 150 μg/l.

Saltwater Aquatic Life

The available data for nitrophenols indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 4,850 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of nitrophenols to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of 2,4-dinitro-o-cresol ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 13.4 μg/l.

For the protection of human health from the toxic properties of 2,4-dinitro-o-cresol ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 765 μg/l.

For the protection of human health from the toxic properties of dinitrophenol ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 70 μg/l.

For the protection of human health from the toxic properties of dinitrophenol ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 14.3 mg/l.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for mononitrophenol.

Using the present guidelines, a satisfactory criterion cannot be derived at this time due to the insufficiency in the available data for tri-nitrophenol.

Nitrosamines

Freshwater Aquatic Life

The available data for nitrosamines indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 5,850 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of nitrosamines to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for nitrosamines indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 3,300,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of nitrosamines to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of n-nitrosodimethylamine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 14 ng/l, 1.4 ng/l, and .14 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 160,000 ng/l, 16,000 ng/l, and 1,600 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of n-nitrosodiethylamine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 8 ng/l, 0.8 ng/l, and 0.08 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 12,400 ng/l, 1,240 ng/l, and 124 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure of n-nitrosodi-n-butylamine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 64 ng/l 6.4 ng/l and .064 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 5,868 ng/l, 587 ng/l, and 58.7 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure in n-nitrosodiphenylamine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 49,000 ng/l 4,900 ng/l and 490 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 161,000 ng/l, 16,100 ng/l, and 1,610 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

For the maximum protection of human health from the potential carcinogenic effects due to exposure in n-nitrosopyrrolidine through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk, over the lifetimes are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 180 ng/l 18.0 ng/l and 1.80 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 919,000 ng/l, 91,900 ng/l, and 9,190 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Pentachlorophenol

Freshwater Aquatic Life

The available data for pentachlorophenol indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 55 and 3.2 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for pentachlorophenol indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 53 and 34 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For comparison purposes, two approaches were used to derive criterion levels for pentachlorophenol. Based on available toxicity data, for the protection of public health, the derived level is 1.01 mg/l. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is 30 μg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Phenol

Freshwater Aquatic Life

The available data for phenol indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 10,200 and 2,560 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for phenol indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 5,800 ug/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of phenol to sensitive saltwater aquatic life.

Human Health

For comparison purposes, two approaches were used to derive criterion levels for phenol. Based on available toxicity data, for the protection of public health, the derived level is 3.5 mg/l. Using available organoleptic data, for controlling

undesirable taste and odor quality of ambient water, the estimated level is 0.3 mg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criterion have limitations and have no demonstrated relationship to potential adverse human health effects.

Phthalate Esters

Freshwater Aquatic Life

The available data for phthalate esters indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 940 and 3 µg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for phthalate esters indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 2944 µg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of phthalate esters to sensitive saltwater aquatic life but toxicity to one species of algae occurs at concentrations as low as 3.4 µg/l.

Human Health

For the protection of human health from the toxic properties of dimethylphthalate ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 313 mg/l.

For the protection of human health from the toxic properties of dimethylphthalate ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 2.9 g/l.

For the protection of human health from the toxic properties of diethylphthalate ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 350 mg/l.

For the protection of human health from the toxic properties of diethylphthalate ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 1.8 g/l.

For the protection of human health from the toxic properties of dibutylphthalate ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 34 mg/l.

For the protection of human health from the toxic properties of dibutylphthalate ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 154 mg/l.

For the protection of human health from the toxic properties of di-2-ethylhexyl-phthalate ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 15 mg/l.

For the protection of human health from the toxic properties of di-2-ethylhexyl-phthalate ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 50 mg/l.

Polychlorinated Biphenyls

Freshwater Aquatic Life

For polychlorinated biphenyls the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.014 µg/l as a 24-hour average. The available data indicate that acute toxicity to freshwater aquatic life probably will only occur at concentrations above 2.0 µg/l and that the 24-hour average should provide adequate protection against acute toxicity.

Saltwater Aquatic Live

For polychlorinated biphenyls the criterion to protect saltwater aquatic life as derived using the Guidelines is 0.030 µg/l as a 24-hour average. The available data indicate that acute toxicity to saltwater aquatic life probably will only occur at concentrations above 10 µg/l and that the 24-hour average should provide adequate protection against acute toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of PCBs through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are .79 ng/l, 0.79 ng/l, and .0079 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are .79 ng/l, .079 ng/l, and .0079 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Polynuclear Aromatic Hydrocarbons (PAHs)

Freshwater Aquatic Life

The limited freshwater data base available for polynuclear aromatic hydrocarbons, mostly from short-term bioconcentration studies with two compounds, does not permit a statement concerning acute or chronic toxicity.

Saltwater Aquatic Life

The available data for polynuclear aromatic hydrocarbons indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 300 ug/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of polynuclear aromatic hydrocarbons to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of PAHs through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 28 ng/l, 2.8 ng/l, and .28 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 311 ng/l, 31.1 ng/l, and 3.11 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Selenium

Freshwater Aquatic Life

For total recoverable inorganic selenite the criterion to protect freshwater aquatic life as derived using the Guidelines is 35 µg/l as a 24-hour average and the concentration should not exceed 260 µg/l at any time.

The available data for inorganic selenate indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 760 µg/l and would occur at lower concentrations among species that are more sensitive

than those tested. No data are available concerning the chronic toxicity of inorganic selenate to sensitive freshwater aquatic life.

Saltwater Aquatic Life

For total recoverable inorganic selenite the criterion to protect saltwater aquatic life as derived using the Guidelines is 54 μg/l as a 24-hour average and the concentration should not exceed 410 μg/l at any time.

No data are available concerning the toxicity of inorganic selenate to saltwater aquatic life.

Human Health

The ambient water quality criterion for selenium is recommended to be identical to the existing drinking water standard which is 10 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Silver

Freshwater Aquatic Life

For freshwater aquatic life the concentration (in μg/l) of total recoverable silver should not exceed the numerical value given by "e[1.72(ln (hardness)-6.52)]" at any time. For example, at hardnesses of 50, 100, 200 mg/l as $CaCO_3$ the concentration of total recoverable silver should not exceed 1.2, 4.1, and 13 μg/l, respectively, at any time. The available data indicate that chronic toxicity to freshwater aquatic life may occur at concentrations as low as 0.12 μg/l.

Saltwater Aquatic Life

For saltwater aquatic life the concentration of total recoverable silver should not exceed 2.3 μg/l at any time. No data are available concerning the chronic toxicity of silver to sensitive saltwater aquatic life.

Human Health

The ambient water quality criterion for silver is recommended to be identical to the existing drinking water standard which is 50 μg/l. Analysis of the toxic effects data resulted in a calculated level which is protective of human health against the ingestion of contaminated water and contaminated aquatic organisms. The calculated value is comparable to the present standard. For this reason a selective criterion based on exposure solely from consumption of 6.5 grams of aquatic organisms was not derived.

Tetrachloroethylene

Freshwater Aquatic Life

The available data for tetrachloroethylene indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 5,280 and 840 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Saltwater Aquatic Life

The available data for tetrachloroethylene indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations low as 10,200 and 450 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of tetrachloroethylene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 8 μg/l, .8 μg/l, and .08 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 88.5 μg/l, 8.85 μg/l, and .88 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Thallium

Freshwater Aquatic Life

The available data for thallium indicate that acute and chronic toxicity to freshwater aquatic life occur at concentrations as low as 1,400 and 40 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested. Toxicity to one species of fish occurs at concentrations as low as 20 μg/l after 2,600 hours of exposure.

Saltwater Aquatic Life

The available data for thallium indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 2,130 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of thallium to sensitive saltwater aquatic life.

Human Health

For the protection of human health from the toxic properties of thallium ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 13 μg/l.

For the protection of human health from the toxic properties of thallium ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 48 μg/l.

Toluene

Freshwater Aquatic Life

The available data for toluene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 17,500 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of toluene to sensitive freshwater aquatic life.

Saltwater Aquatic Life

The available data for toluene indicate that acute and chronic toxicity to saltwater aquatic life occur at concentrations as low as 6,300 and 5,000 μg/l, respectively, and would occur at lower concentrations among species that are more sensitive than those tested.

Human Health

For the protection of human health from the toxic properties of toluene ingested through water and contaminated aquatic organisms, the ambient water criterion is determined to be 14.3 mg/l.

For the protection of human health from the toxic properties of toluene ingested through contaminated aquatic organisms alone, the ambient water criterion is determined to be 424 mg/l.

Toxaphene

Freshwater Aquatic Life

For toxaphene the criterion to protect freshwater aquatic life as derived using the Guidelines is 0.013 μg/l as a 24-hour average and the concentration should not exceed 1.8 μg/l at any time.

Saltwater Aquatic Life

For saltwater aquatic life the concentration of toxaphene should not exceed 0.070 μg/l at any time. No data

are available concerning the chronic toxicity of toxaphene to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of toxaphene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 7.1 ng/l, .71 ng/l, and .07 ng/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 7.3 ng/l, .73 ng/l, and .07 ng/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Trichloroethylene

Freshwater Aquatic Life

The available data for trichloroethylene indicate that acute toxicity to freshwater aquatic life occurs at concentrations as low as 45,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of trichloroethylene to sensitive freshwater aquatic life but adverse behavioral effects occurs to one species at concentrations as low as 21,900 μg/l.

Saltwater Aquatic Life

The available data for trichloroethylene indicate that acute toxicity to saltwater aquatic life occurs at concentrations as low as 2,000 μg/l and would occur at lower concentrations among species that are more sensitive than those tested. No data are available concerning the chronic toxicity of trichloroethylene to sensitive saltwater aquatic life.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of trichloroethylene through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 27 μg/l, 2.7 μg/l, and .27 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 807 μg/l, 80.7 μg/l, and 8.07 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Vinyl Chloride

Freshwater Aquatic Life

No freshwater organisms have been tested with vinyl chloride and no statement can be made concerning acute or chronic toxicity.

Saltwater Aquatic Life

No saltwater organisms have been tested with vinyl chloride and no statement can be made concerning acute or chronic toxicity.

Human Health

For the maximum protection of human health from the potential carcinogenic effects due to exposure of vinyl chloride through ingestion of contaminated water and contaminated aquatic organisms, the ambient water concentration should be zero based on the non-threshold assumption for this chemical. However, zero level may not be attainable at the present time. Therefore, the levels which may result in incremental increase of cancer risk over the lifetime are estimated at 10^{-5}, 10^{-6}, and 10^{-7}. The corresponding criteria are 20 μg/l, 2.0 μg/l, and .2 μg/l, respectively. If the above estimates are made for consumption of aquatic organisms only, excluding consumption of water, the levels are 5,246 μg/l, 525 μg/l, and 52.5 μg/l, respectively. Other concentrations representing different risk levels may be calculated by use of the Guidelines. The risk estimate range is presented for information purposes and does not represent an Agency judgment on an "acceptable" risk level.

Zinc

Freshwater Aquatic Life

For total recoverable zinc the criterion to protect freshwater aquatic life as derived using the Guidelines is 47 μg/l as a 24-hour average and the concentration (in μg/l) should not exceed the numerical value given by $e^{(0.83\,(\ln(\text{hardness}))\,+\,1.95)}$ at any time. For example, at hardnesses of 50, 100, and 200 mg/l as $CaCO_3$ the concentration total recoverable zinc should not exceed 180, 320, and 570 μg/l at any time.

Saltwater Aquatic Life

For total recoverable zinc the criterion to protect saltwater aquatic life as derived using the Guidelines is 58 μg/l as a 24-hour average and the concentration should not exceed 170 μg/l at any time.

Human Health

Sufficient data is not available for zinc to derive a level which would protect against the potential toxicity this compound. Using available organoleptic data, for controlling undesirable taste and odor quality of ambient water, the estimated level is mg/l. It should be recognized that organoleptic data as a basis for establishing a water quality criteria have limitations and have not demonstrated relationship to potential adverse human health effects.

Appendix B—Guidelines for Deriving Water Quality Criteria for the Protection of Aquatic Life and Its Uses

Introduction

This version of the Guidelines provides clarifications, additional details, and technical and editorial changes in the last version published in the Federal Register [44 FR 15970 (March 15, 1979)]. This version incorporates changes resulting from comments on previous versions and from experience gained during U.S. EPA's use of the previous versions. Future versions of the Guidelines will incorporate new ideas and data as their usefulness is demonstrated.

Criteria may be expressed in several forms. The numerical form is commonly used, but descriptive and procedural forms can be used if numerical criteria are not possible or desirable. The purpose of these Guidelines is to describe an objective, internally consistent and appropriate way of deriving numerical water quality criteria for the protection of the uses of, as well as the presence of, aquatic organisms.

A numerical criterion might be thought of as an estimate of the highest concentration of a substance in water which does not present a significant risk to the aquatic organisms in the water and their uses. Thus the Guidelines are intended to derive criteria which will protect aquatic communities by protecting most of the species and their uses most of the time, but not

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