Quality Assurance Project Plan

Onalaska Municipal Landfill
Onalaska, Wisconsin

Groundwater Remedial Action

WA 38-5NL5/Contract No. 68-W8-0040

May 1992

GLT272/030.51

Section 1

Title Page

Remedial Planning Activities
(ARCS V)
Contract No. 68-W8-0040
Quality Assurance Project Plan (QAPP)

Project Title: Groundwater Remedial Action
Onalaska Municipal Landfill
Onalaska, Wisconsin

EPA No: WA 38-5NL5

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Project Description

3.1 Introduction

The United States Environmental Protection Agency (EPA) requires that all EPA contractors participate in a centrally managed quality assurance (QA) program. That requirement applies to all environmental monitoring and measurement efforts mandated or supported by the EPA. Each contractor generating data has the responsibility to implement minimum procedures to see that the precision, accuracy, completeness, and representativeness of its data are known and documented. To see that this responsibility is met uniformly, each EPA contractor must prepare a written Quality Assurance Project Plan (QAPjP) addressing each project it is contracted to perform.

This QAPjP is prepared as part of Work Assignment No. 38-5NL5 under ARCS V (Contract No. 68-W8-0040) which authorizes CH2M HILL to complete the remedial design for the Onalaska Municipal Landfill in Onalaska, Wisconsin. This QAPjP presents the organization, objectives, functional activities, and specific QA and quality control (QC) activities associated with groundwater sampling as part of the groundwater monitoring plan to implement the selected remedial action for groundwater extraction and treatment at the Onalaska Municipal Landfill site.

3.2 Site Description

The Onalaska Municipal Landfill is in La Crosse County, Wisconsin, about 10 miles north of the City of La Crosse near the confluence of the Mississippi and Black Rivers and within 400 feet of the Black River (Figure 1). Several homes are located within 500 feet of the site, and a subdivision of about 50 homes is located 1.25 miles southeast of the site. The area is generally rural. The sand and gravel aquifer is used as the water supply.

The 11-acre site was mined as a sand and gravel quarry in the early 1960s (see Figure 2). In the mid-1960s the quarry operation ceased, and the Town of Onalaska began using the quarry as a municipal landfill. Between 1969 and 1980, municipal trash and chemical wastes were disposed of in the landfill. The landfill was capped between 1980 and 1982. The site is not fenced, but two gates restrict vehicular access to it.

3.3 Site History and Background

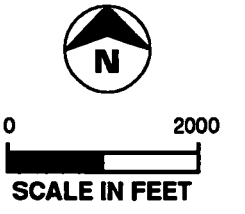
3.3.1 Site History

The Town of Onalaska owned and was licensed to operate the Onalaska Municipal Landfill from 1969 to 1980. The Wisconsin Department of Natural Resources (DNR) ordered its closure in 1980. During 11 years of operation, the Onalaska Landfill provided waste disposal for residential, commercial, and industrial generators located within the township and nonresidents with written permits. The landfill also accepted refuse from other townships.

Landfill operations were informal. During the first 3 years of operation, there was no attendant at the landfill. Later, operating hours were posted and an operator was present to cover incoming waste and measure the nonresidential waste for billing purposes. The landfill boundaries were defined by a cable or fence partially enclosing the site. A gate was installed at the site in early 1971 to restrict site access. However, keys were readily provided to clients who wished to use the landfill outside the posted operating hours.

Seven acres of the Onalaska Landfill were reportedly reserved for using the compaction and cover method of waste disposal. The landfill was regularly inspected by the DNR. Early DNR records indicate that open burning was practiced at the site in late 1970. The DNR prohibited all open burning in January 1971 after receiving several complaints about noxious odors and sooty, black smoke from the burning of naphtha, an oily industrial solvent waste. Consequently, the DNR required that an area be designated for the disposal of industrial solvents and wastes delivered to the site. Several industrial firms are known to have used the landfill for waste disposal.

Outers Laboratories and Metallics, Inc. (two companies owned by one person), contributed significant quantities of industrial wastes to the site. Daily landfill operation reports indicate that these two companies were disposing of industrial waste oils and solvents as early as July 7, 1970. Early DNR records report that Outers delivered liquid solvent residues to the site for burning. The waste solvents consisted primarily of naphtha, toluene, and paint residues. Initially, both Outers and Metallics hauled solvent wastes in 55-gallon barrels. Once a week, 20 to 25 barrels of industrial wastes from the companies were hauled to the landfill. The barrels were emptied and the waste was burned. After burning was banned, the liquid waste was dumped in the designated area and poured into excavated holes for immediate burial. Occasionally, full barrels were left at the site if they could not be easily emptied or if they were damaged or leaking. In later years, the liquid waste was hauled in a 500-gallon truck instead of barrels. At that time, about 300 barrels were additionally mass buried at the landfill.



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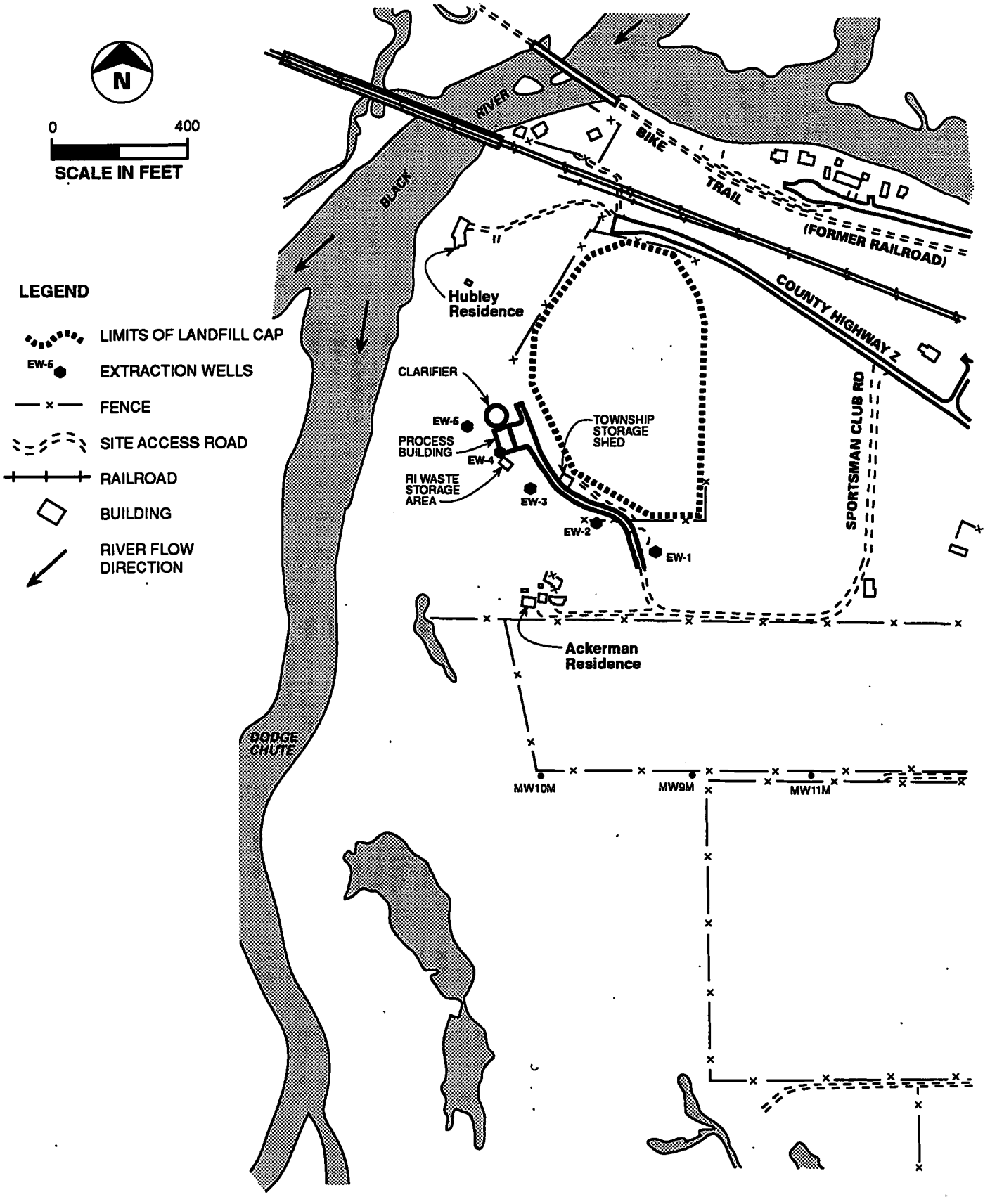
SOURCE: U.S. Geological Survey, Trempealeau, 15' Quadrangle.

FIGURE 1
SITE LOCATION MAP
ONALASKA
GWMP and QAPP



GLO\$5602.DS.GM F2 SITE MAP 4-20-92mms/ds

FIGURE 2
SITE MAP
ONALASKA LANDFILL
GWMP and QAPP

Reportedly, on one occasion, when a tank truck hauling the waste could not be drained because the discharge outlet was plugged with hardened paint resin and solvent, the truck was buried in the south section of the landfill. In August 1975, the DNR recommended that Outers find alternative methods to dispose of its naphtha waste. Outers investigated and eventually implemented a reclamation process to recover some of the raw materials from the waste. In April 1976, Outers informed the DNR that it was no longer disposing of liquid wastes in the landfill.

On February 9, 1978, the DNR issued an order to the township to submit an infield conditions report for the landfill because the site did not meet Wisconsin solid waste codes. Warzyn Engineering investigated the site for the township and submitted a report to the DNR on April 17, 1978. Warzyn recommended phased abandonment of the site. In June 1978, the DNR reported that the average distance between the groundwater table and the base of the refuse pile was 1 foot. Studies showed that the seasonal fluctuations in water levels sometimes allowed the groundwater to be in direct contact with a portion of the waste for extended periods of time.

On October 19, 1978, Warzyn Engineering submitted a plan of operation for phased abandonment of the landfill. On May 4, 1979, the DNR issued a plan approval and ordered the landfill closed by September 30, 1979. On May 30, 1980, the DNR modified the order to close the landfill by September 30, 1980. Closure proceeded in phases, and the final cap was placed in July 1982.

In September 1982, the DNR sampled and analyzed water from monitoring wells and private wells for compliance with drinking water standards for organic and inorganic constituents. The investigations indicated that groundwater contamination had occurred. The barium concentrations in the water from Cecil Miller's residential well south of the site exceeded the drinking water standard, and five organic compounds were detected above background levels. In January 1983, the Town of Onalaska replaced Mr. Miller's well with a deep well.

On May 2, 1983, an EPA Potential Hazardous Waste Site inspection report was submitted. In September 1984 the Onalaska Landfill was placed on the National Priorities List with a hazard ranking of 42.97.

3.3.2 Background

Except for the industrial waste solvents from Outers Laboratories and Metallics, Inc. There is little indication that the wastes within the 7 acres used for open pit disposal were segregated. Industrial, commercial, and municipal wastes are considered to be mixed throughout the fill area. Outers and Metallics used a specific area designated for liquid industrial waste disposal according to DNR correspondence and license applications. However, the designated disposal area was not strictly limited to the

industrial wastes from Outers and Metallics. Records indicate that other commercial wastes were deposited simultaneously in the area designated for liquid industrial waste disposal in October 1981 and October 1982.

For a time, open burning occurred at the site. Until early 1971 when open burning was banned, the industrial solvents from Outers and Metallics were burned regularly at apparently random locations throughout the landfill. Some refuse was also burned bimonthly. Open burning reportedly continued, even though banned, until as late as 1979.

Liquid industrial wastes consisted primarily of naphtha-based solvents used in a metal cleaning process and solvent wastes from paint spray, gun cleaning, and machine shop cleaning fluids. At least two kinds of naphtha were disposed of at the site—high-flash naphtha and VM&P or Stoddard naphtha. High-flash naphtha is a coal-tar derivative consisting primarily of a mixture of aromatic hydrocarbons. It was probably used as a degreasing agent or a general solvent. The VM&P or Stoddard naphthas, derived from petroleum, are slightly more volatile. They consist of a mixture of aliphatic hydrocarbons, naphthenes, and alkyl benzenes. They are used as universal solvents for general cleaning and as paint thinners. These naphthas were probably used in a paint cleaning process at one of the plants and as general solvents. Both the petroleum and coal-tar derived naphthas are less dense than water and would float on the surface of the water table if they reached the aquifer.

Some of the organic compounds detected in the groundwater during past analyses may have been derived from naphtha wastes floating on the water table. The liquid naphtha waste could generate a complex mixture of dissolved organic compounds in the groundwater over a period of time. Both types of naphtha would each produce a different suite of degradation products of varying composition. It is impossible to predict the exact composition of each mixture, but generally naphtha degradation products consist of aliphatic and aromatic carboxylic acids, toluene, and other complex mixtures of aromatic and aliphatic hydrocarbons.

3.4 Target Compounds

Contaminant concentrations in the groundwater at individual monitoring well locations within the landfill or at the landfill boundary contained contaminant concentrations that exceed one or more standards or criteria. The Safe Drinking Water Act maximum contaminant levels (MCLs) for arsenic, barium, benzene, 1,1-dichloroethene, toluene, 1,1,1-trichloroethane, trichloroethene, and xylene were exceeded at one or more monitoring well locations.

A series of shallow groundwater samples were collected during the RI and were analyzed using a close support laboratory. The primary objectives of the shallow groundwater analysis were to locate the extent of the floating non-aqueous phase and to help select groundwater monitoring well locations. The close support laboratory analyzed a total of 81 samples for the following organic compounds:

- Toluene
- Total xylenes
- 1,1,1-TCA
- TCE
- PCE

These compounds were selected on the basis of historical groundwater analysis, site history, and their chemical properties (e.g., mobility). Concentrations of toluene were observed as high as 43,000 μg/L. Of the three chlorinated compounds analyzed for, 1,1,1-TCA was the most prevalent, and was found at concentrations as high as 730 μg/L.

Two rounds of groundwater sampling for Contract Laboratory analysis were conducted. These samples were analyzed for the complete Target Compound List (TCL) and 13 Special Analytical Services (SAS) parameters.

Volatile Organic Compounds (VOCs) were generally observed to be present at concentrations much greater than semivolatile organics (sometimes more than an order of magnitude greater). The majority of the VOCs detected during the Remedial Investigation were found in shallow monitoring wells (MW-5S and MW-3S and B4S) and were BTEX compounds. The vertical extent of BTEX and chlorinated compounds contamination is mostly confined to the upper 10 to 20 feet of the aquifer. Ethylbenzene, 1,1-DCA and chloroethane were detected, however, at depths up to 50 to 60 feet below the water table. The vertical extent of semivolatile organic compounds (SVOCs) contamination is also mostly confined to the upper 10 to 20 feet of the aquifer. There were no SVOCs detected in any of the deep monitoring wells.

Monitoring wells along the southwestern edge of the landfill and southwest of the landfill have the most occurrence of inorganic chemicals above background. These are primarily shallow and medium wells that included MW-2S, MW-2M, MW-3S, MW-4S, MW-B4S, MW-5S, and MW-8S. Four chemicals: barium, iron, manganese, and sodium, were detected above background with greater frequency than the other inorganic chemicals. The higher concentrations of these four chemical tends to occur in wells along the southwestern edge of the landfill or southwest of the landfill.

Under the remedy selected in the ROD, the following cleanup standards were adopted:

- Groundwater contaminant plume located at any point beyond the property boundary or DMZ:
  - Preventive Action Limits (PALs) from Wisconsin Administrative Code Chapter NR 140
- Groundwater contaminant plume located at landfill waste boundary:
  - Maximum Contaminant Levels (MCLs) from the Safe Drinking Water Act, 40 CFR 141.61 and 40 CFR 143
  - Maximum Contaminant Level Goals (MCLGs) above zero Safe Drinking Water Act, 40 CFR 141.50

The ROD requires that the more stringent Wisconsin standards promulgated in NR 140, WAC, be achieved "at any point beyond the property boundary or beyond the three-dimensional design management zone, whichever is closer to the waste boundary." The DMZ as defined in NR 140 is a three-dimensional boundary surrounding a regulated facility. The boundary extends from the ground surface through all saturated geological strata. The DMZ defined for the Onalaska site extends 250 feet horizontally from the waste boundary as shown in Figure 3. Because the property boundary generally is closer to the waste boundary than to the DMZ, the property boundary is considered the point at which PALs apply. As the plume is reduced in size, however, the MCLs or MCLGs could apply.

Tables 1 and 2 present a summary of monitoring well concentrations that exceeded U.S. EPA drinking water standards, criteria and guidelines, or Wisconsin groundwater protection standards.

Groundwater samples from the monitoring and extraction wells will be analyzed for the target compounds listed in Table 3. Table 3 consists of the chemical parameters identified in Table 3B of the ROD as the "Chemicals of Concern" and additional parameters were added to the list in order to better monitor the landfill or as a means to meet Town of Onalaska sampling requirements.

- Chloride and total dissolved solids (TDS) analyses will be used to assess the relative strength of the leachate contributed by the landfill.
- Total organic carbon (TOC) analyses will be used to determine the amount of organic constituents in the leachate and to monitor the contaminant concentrations in the plume.

Table 1
Summary of Monitoring Well Concentrations That Exceed
Wisconsin Groundwater Protection Standards
Onalaska Landfill Site

Well	Chemical	Detected Concentration (µg/L)	Criteria[a] Exceeded	Criteria Level (µg/L)
MW02S-01	Benzene	5	ES	0.67
			PAL	0.067
	Arsenic	9.5	PAL	5
	Chromium	24.8	PAL	5
MW02M-01	Arsenic	19.4	PAL	5
	Barium	1390	ES	1000
			PAL	200
MW03S-01	1,1-Dichloroethene	15	ES	0.24
			PAL	0.024
	Benzene	13	ES	0.67
			PAL	0.067
	1,1,1-Trichloroethane	240	ES	200
			PAL	40
	Trichloroethene	11	ES	1.8
			PAL	0.18
	Toluene	8300	ES	343
			PAL	68.6
	Xylene	2300	ES	620
			PAL	124
	Arsenic	19.4	PAL	5
	Barium	593	ES	1000
MW03M-01	Arsenic	68.4	ES	50
			PAL	5
	Barium	2760	ES	1000
			PAL	200
MW04S-01	Toluene	530	ES	343
			PAL	68.6
	Arsenic	6.9	PAL	5
	Barium	1140	ES	1000
MW05S-01	Benzene	7	ES	0.67
			PAL	0.067
	Toluene	8300	ES	343
			PAL	68.6
	Xylene	1400	ES	620
			PAL	124
	Arsenic	8	PAL	5
	Barium	347	ES	1000
MW06M-01	Barium	1370	ES	1000
			PAL	200
MW08M-01	Barium	600	PAL	200
W21S-01	Barium	201	PAL	200

Note: The public welfare PAL and ES for iron was exceeded for all wells except MW06M, MW08D, MW08M, MW08S, MW10M, MW12S, and MW13S. The public welfare PAL and ES for manganese exceed in all wells.

[a]Criteria abbreviation:
ES Enforcement Standard
PAL Protective Action Limit

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Table 2
Summary of Monitoring Well Concentrations That Exceed U.S. EPA Drinking Water Standards, Criteria, and Guidelines
Onalaska Site

Well	Chemical	Detected Concentration (µg/L)	Criteria[a] Exceeded	Criteria Level (µg/L)
MW02S-01	Benzene	5	MCL	5
			MCLG	0
			WQC-Risk	0.67
	Ethylbenzene	210	MCL2°	30
MWO2M-01	Barium	1,390	MCL	1,000
MW03S-01	Benzene	13	MCL	5
			MCLG	0
			WQC-Risk	0.67
	1,1-Dichloroethene	15	MCL	7
			MCLG	7
			WQC-Risk	0.033
	1,1,1-Trichloroethane	240	MCL	200
			MCLG	200
			DWLHA	200
	Trichloroethene	11	MCL	5
			MCLG	0
			WQC-Risk	2.8
	Toluene	8,300	MCL-Prop	2,000
			MCL2°	40
			MCLG-Prop	2,000
			DWLHA	2,040
	Xylene	2,300	DWLHA	400
			MCL2°	20
MW03M-01	Arsenic	68.4	MCL	50
			DWLHA	50
			WQC-Risk	0.0025
	Barium	2,760	MCL	1,000
			DWLHA	1,500
MW04S-01	Ethylbenzene	42	MCL2°	30
	Toluene	530	MCL2°	40
MW05S-01	Benzene	7	MCL	5
			MCLG	0
			WQC-Risk	0.67
	Ethylbenzene	160	MCL2°	30
	Toluene	8,300	MCL-Prop	2,000
			MCL2°	40
			MCLG-Prop	2,000
			DWLHA	2,040
	Xylene	1,400	DWLHA	400
			MCL2°	20
MW06M-01	Barium	1,370	MCL	1,000

Note: The secondary MCL for mangenese exceeded in all wells except MW125 and MW135. The secondary MCL for iron exceeded in all wells except MW06M, MW08D, MW08M, MW08S, MW10M, MW12S, MW13S, MW21S

[a]Criteria abbreviations:
MCL - Maximum Contaminant Level
MCL2° - Secondary Maximum Contaminant Level
MCLG - Maximum Contaminant Level Goal
WQC-RISK - Water Quality Criteria at 10^{-6} risk level
DWLHA - Drinking Water Lifetime Healthy Advisory
Prop - Proposed

Table 3
Analyte List and Action Levels

Target Analytes	PAL (µg/L)	MCL (µg/L)	MCLG (µg/L)	Detection Limit[a] (µg/L)
ROD Compounds				
Benzene	0.067	5	0	0.03
Toluene	68.6	1,000[b]	1,000[b]	0.08
Xylenes	124	10,000[b]	10,000[b]	0.02
Ethylbenzene	272	700	700	0.03
Trichloroethene	0.18	5	0	0.02
1,1-Dichloroethane	85			0.03
1,1,1-Trichloroethane	40	200	200	0.04
1,1-Dichloroethene	0.024	7	7	0.05
Arsenic	5	50		10
Barium	0.2	2,000	2,000	200
Lead	5	15[c]	0.0	3
Others				
1,1,2,2-Tetrachloroethylene	125,000			0.2
Chloride	1,000			5,000
Total Organic Carbon (TOC)	200,000			500
Total Dissolved solids (TDS)				20,000
Oil and Grease				400
Alkalinity				2,000
Hardness	150			1,000
Iron	25			100
Manganese				10
Chemical Oxygen Demand (COD)				5,000
Color	7.5[d]			1[d]
Turbidity				0[e]
Odor	1.5[f]			1[f]

[a] Based on SAS detection limits
[b] Standard effective July 30, 1992
[c] Action level
[d] Color Units
[e] Nephelometric Turbidity Units
[f] Threshold Odor No.

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- Oil and grease analyses will be used to monitor or the presence of the nonaqueous phase detected in the remedial investigation.

- 1,1,2,2-tetrachloroethylene, total alkalinity as $CaCO_3$, manganese, iron, COD, color, odor, and turbidity were added to Table 3 to fulfill a Town of Onalaska requirement for semi-annual monitoring of these parameters.

The groundwater monitoring plan (Appendix A) includes monthly and quarterly sampling of monitoring wells and extraction wells and collection of monthly groundwater elevation data from the six piezometers. In addition, annual surface water and sediment samples will be collected in triplicate from two locations during the June or third quarter sampling event.

Groundwater, surface water, and sediment samples will be analyzed for the listed compounds (Table 3) using the Contract Laboratory Program's (CLP's) special analytical services (SAS) for select volatile organic compounds (VOCs), select inorganic constituents, and select conventional parameters. Field temperature, pH, and specific conductance will also will be recorded for each well during sampling. Field sampling procedures, methods of analyses, and QA/QC protocols for CLP analyses will be followed in accordance with the QAPjP.

3.5 Project Objectives

The objectives of the groundwater monitoring program are to:

- Provide data to determine if groundwater contaminant concentrations in the aquifer have been reduced to below the cleanup criteria

- Provide data to verify that a hydraulic gradient is being maintained by the extraction system in order to contain and collect contaminated groundwater

- Provide data to determine if groundwater contaminant concentrations in the aquifer between the landfill and the Black River are being reduced by the extraction system

- Monitor water levels in the wetlands adjacent to the site to make sure that the extraction system is not lowering water levels to such a level as to adversely affect the wetlands

3.5.1 Intended Data Usage

These data shall be used to evaluate the effectiveness of the remedial action design and determine when groundwater extraction may cease. The data will be used to:

- Determine the change in extent or movement of the groundwater contaminant plume
- Monitor the safety of field sampling personnel and to select proper personal protective equipment by screening VOC concentrations with an HNu or organic vapor analyzer (OVA)
- Support decisions related to operation of the groundwater extraction system (such as extraction rate per well)

3.5.2 Data Quality Objectives

Data quality objectives (DQOs) define and specify the quality of data required for the intended use of the data. The degree of certainty of a data set with respect to precision, accuracy, representativeness, completeness, and comparability is an indication of the data quality.

There are five levels of Analytical Date Quality, and they are defined as follows:

1. Level I—Field Screening. The objective of this level of analysis is to generate data to be used in refining sampling plans and determining gross extent of contamination at the site. This type of data also provides real time monitoring for health and safety.

2. Level II—Field Analysis. The objective of this level of analysis is to provide real-time data for ongoing field activities. This level of analysis also provides preliminary data used to decide what additional laboratory analyses should be performed. Analyses include the use of an onsite close support laboratory.

3. Level III—Laboratory Analysis. This level of analysis is designed to provide laboratory analyses using standard EPA-approved procedures other than the current Contract Laboratory Programs (CLP) Routine Analytical Services (RAS). This level provides data for site characterization, environmental monitoring, and confirmation of field data; and to support engineering studies.

4. Level IV—CLP RAS. This level of analysis provides for the highest level of data quality with full CLP analytical, QC, and validation procedures in accordance with EPA protocols. The data is used for risk assessment, confirmation of field analysis data, and to obtain highly documented data.

5. Level V—Nonstandard Methods, CLP SAS. The objective of this level of analysis is to provide data not obtained through standard avenues of analytical support. This usually involves modification of existing methods or method development. The level of quality control is usually similar to that of Level IV data.

Levels I and V analytical data will be generated during groundwater monitoring at the Onalaska Municipal Landfill. DQO Level I data to be generated include field measurements of groundwater pH, temperature, and specific conductance and HNu/ OVA readings. The laboratory analyses requested include the Level V chemical analyses. Level V data will be needed to provide the rigorous QA/QC required to track and monitor the groundwater contaminant plume, to determine if any remedial action is required because of risks to public health, and because nonstandard procedures are required to meet the lower detection limits of VOC and inorganic analyses.

3.6 Sample Network Design and Rationale

The groundwater monitoring network consists of six new water table piezometers, one new monitoring well, six existing monitoring wells, and five new extraction wells. The groundwater monitoring network was designed to provide groundwater quality data for the site and adjacent area, and to facilitate evaluation of the hydraulic gradient control. Detailed rationale for selection of each well and piezometer is summarized in the groundwater monitoring plan. Well and piezometer locations are shown in Figure 3.

3.6.1 Monitoring Well / Piezometer Selection / Installation

The monitoring wells (MW-1S, MW-6S, MW-6M, MW-8S, MW-8M, MW-12S, and MW-14S) shall be used to monitor:

- If contaminated groundwater has been captured successfully (i.e., contaminants of concern are below action levels at point of compliance)

- Groundwater quality changes downgradient of the collection system capture zone (i.e., how quickly concentrations of contaminants of concern decrease after the extraction system is functioning)

- Hydraulic gradient control (i.e., whether contaminated groundwater plume is moving toward the extraction wells)

Shallow wells MW-6S (new) and MW-8S and intermediate wells MW-6M and MW-8M will be used to monitor groundwater quality downgradient of the landfill and extraction well network. These wells are outside of the capture zone. MW-12S and MW-14S are located on the periphery of the capture zone and will be used to monitor groundwater quality and hydraulic gradient control east and west of the extraction well network. MW-1S will be used to monitor background groundwater quality upgradient of the landfill.

Six shallow water piezometers were installed to monitor hydraulic gradient control during the remedial action.

3.6.2 Extraction Well Selection / Installation

A series of five extraction wells have been installed in locations to capture the contaminant plume prior to offsite groundwater discharge. The extraction well network has been designed to extract about 800 gallons per minute (gpm) of contaminated groundwater for treatment. Groundwater from the extraction wells will be monitored to assess the effectiveness of the remedial action.

3.6.3 Surface Water and Sediments

The groundwater beneath the site generally flows in a south-southwesterly direction toward the wetlands bordering the Black River. Although no site-derived contamination was detected in the surface water and sediment samples collected during the remedial investigation, surface water and sediments will be sampled annually during the remedial action to monitor for potential offsite contaminant migration. Surface water and sediment grab samples will be collected from the wetland area and Dodge Chute. Sample locations are shown in Figure 3.

3.6.4 Background / Baseline Monitoring

The monitoring program will begin with the collection and analysis of four discrete samples from all 17 wells in the monitoring program to develop baseline concentrations. The samples will be analyzed for the parameters listed in Table 3. The analytical results will be compared to background concentrations.

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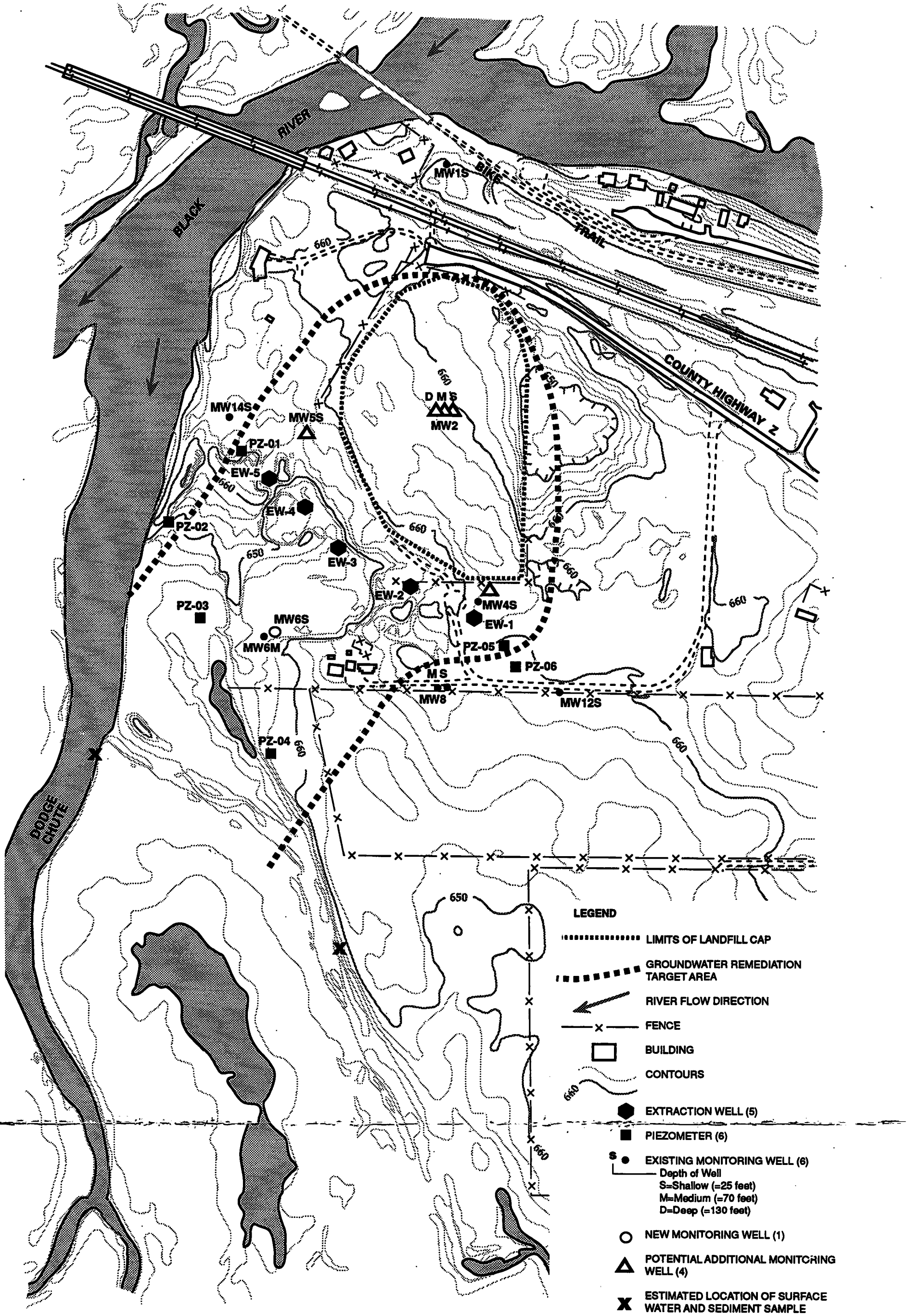

FIGURE 3
MONITORING WELL, EXTRACTION WELL, AND PIEZOMETER NETWORK
ONALASKA
GWMP and QAPP

3.6.5 Sampling

3.6.5.1 Monthly

Monthly groundwater samples will be collected during the first year of system operation. The primary purpose of the monthly sampling is to monitor the efficacy of the newly-implemented groundwater extraction and treatment system and identify needed refinements and corrections. Monthly groundwater samples and elevation data will be collected from the five extraction wells (EW-01 through EW-05) and six monitoring wells (MW-06S, MW-06M, MW-08S, MW-8M, MW-12S, and MW-14S) during the first year of remedy implementation. Monthly groundwater elevation data also will be collected from the six piezometers and all existing onsite monitoring wells for the first year.

3.6.5.2 Quarterly Sampling

The primary purpose of the quarterly sampling is to continue to evaluate the groundwater extraction and treatment system for reliable operation and to monitor the reduction in contaminant concentrations in the aquifer. The quarterly sampling will also identify any seasonal fluctuations in groundwater quality.

Quarterly sampling will commence at the beginning the second year and continue through the fifth year of system operation or until the groundwater contaminant concentrations approach the groundwater cleanup standards.

3.6.6 Annual Surface Water and Sediment Sampling

Surface water and sediment samples will be collected annually from the two locations shown in Figure 3. The primary purpose of these samples is to monitor for any unusual increase in contaminant concentrations that may be attributed to remedy implementation activities.

Table 4 lists the total number of samples for analysis by sampling event, matrix, and planned measurements.

3.7 Project Schedule

The monthly groundwater level measurements and groundwater samples shall be collected during the first week of each month. After the first year of monthly sampling and system evaluation, samples will be taken quarterly, unless unanticipated problems indicate that continued monthly sampling is warranted.

The quarterly sampling will consist of collecting groundwater samples and taking elevation measurements from the six monitoring wells and five extraction wells. The samples will be collected during the first week of March, June, September, and December. Elevation measurements will continue to be collected on a monthly basis from the piezometers, monitoring wells, and extraction wells. The need for elevation data from the other existing monitoring wells will be assessed after the first year. Depending on the analytical results from the quarterly sampling and the absence of operational problems, the sampling schedule may be modified further.

The groundwater quality will be evaluated at the end of the fifth year to determine if the groundwater standards have been met. The ROD estimates that 95 percent of the contaminants will be removed from the groundwater plume within the 5-year timeframe. If the groundwater goals (or WACLs, if established) have not been met, sampling and remediation will continue until the cleanup goals are achieved. The frequency of sampling will be evaluated based on the trends observed in the first 5 years. If an applicable and appropriate requirement (ARAR) waiver is established, the groundwater goals and the need or frequency of further sampling will be addressed as part of the waiver process.

The annual surface water and sediment samples shall be collected during the first week of June each year.

TABLE 4

SAMPLING AND ANALYSIS SUMMARY FOR BASELINE MONITORING

ONALASKA MUNICIPAL LANDFILL

TASK	SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLE			FIELD DUPLICATES			FIELD BLANKS			TRIP BLANKS			MATRIX SPIKE(S)			MATRIX TOTAL
				No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	
1	GROUNDWATER	pH	CHLORIDE	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
		TEMPERATURE	ODOR	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
		CONDUCTIVITY	COLOR	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
		HNu/OVA	COD	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
			ALKALINITY	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
			HARDNESS	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
			TDS	17	1	17	2	1	2	2	1	2	0	0	0	0	0	0	21
			TOC	17	1	17	2	1	2	2	1	2	0	0	0	1	1	1	22
			OIL & GREASE	17	1	17	2	1	2	2	1	2	0	0	0	1	1	1	22
			VOCs	17	1	17	2	1	2	2	1	2	1	1	1	2	1	2	24
			METALS	17	1	17	2	1	2	2	1	2	0	0	0	1	1	1	22

TABLE 4

SAMPLING AND ANALYSIS SUMMARY FOR MONTHLY MONITORING

(YEARLY BASIS)

ONALASKA MUNICIPAL LANDFILL

		FIELD	LABORATORY	SAMPLE			FIELD DUPLICATES			FIELD BLANKS			TRIP BLANKS			MATRIX SPIKE(S)			MATRIX
TASK	SAMPLE MATRIX	PARAMETERS	PARAMETERS	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	TOTAL
2	GROUNDWATER	pH	CHLORIDE	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
		TEMPERATURE	ODOR	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
		CONDUCTIVITY	COLOR	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
		HNu/OVA	COD	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
			ALKALINITY	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
			HARDNESS	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
			TDS	11	12	132	1	12	12	1	12	12	0	0	0	0	0	0	156
			TOC	11	12	132	1	12	12	1	12	12	0	0	0	1	12	12	168
			OIL & GREASE	11	12	132	1	12	12	1	12	12	0	0	0	1	12	12	168
			VOCs	11	12	132	1	12	12	1	12	12	1	12	12	2	12	24	192
			SELECT METALS	11	12	132	1	12	12	1	12	12	0	0	0	1	12	12	168

TABLE 4

ANNUAL SAMPLING AND ANALYSIS SUMMARY FOR QUARTERLY MONITORING

(YEARLY BASIS)

ONALASKA MUNICIPAL LANDFILL

		FIELD	LABORATORY	SAMPLE			FIELD DUPLICATES			FIELD BLANKS			TRIP BLANKS			MATRIX SPIKE(S)			MATRIX
TASK	SAMPLE MATRIX	PARAMETERS	PARAMETERS	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	TOTAL
3	GROUNDWATER	pH	CHLORIDE	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
		TEMPERATURE	ODOR	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
		CONDUCTIVITY	COLOR	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
		HNu/OVA	COD	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
			ALKALINITY	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
			HARDNESS	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
			TDS	11	4	44	1	4	4	1	4	4	0	0	0	0	0	0	52
			TOC	11	4	44	1	4	4	1	4	4	0	0	0	1	4	4	56
			OIL & GREASE	11	4	44	1	4	4	1	4	4	0	0	0	1	4	4	56
			VOCs	11	4	44	1	4	4	1	4	4	1	4	4	2	4	8	64
			SELECT METALS	11	4	44	1	4	4	1	4	4	0	0	0	1	4	4	56

TABLE 4

SAMPLING AND ANALYSIS SUMMARY FOR ANNUAL MONITORING

(YEARLY BASIS)

ONALASKA MUNICIPAL LANDFILL

				SAMPLE			FIELD DUPLICATES			FIELD BLANKS			TRIP BLANKS			MATRIX SPIKE(S)			MATRIX
TASK	SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	No.	FREQ.	TOTAL	TOTAL
4	SURFACEWATER	pH	CHLORIDE	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
		TEMPERATURE	ODOR	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
		CONDUCTIVITY	COLOR	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
		HNu/OVA	COD	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
			ALKALINITY	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
			HARDNESS	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
			TDS	2	1	2	1	1	1	1	1	1	0	0	0	0	0	0	4
			TOC	2	1	2	1	1	1	1	1	1	0	0	0	1	1	1	5
			OIL & GREASE	2	1	2	1	1	1	1	1	1	0	0	0	1	1	1	5
			VOCs	2	1	2	1	1	1	1	1	1	1	1	1	2	1	2	7
			SELECT METALS	2	1	2	1	1	1	1	1	1	0	0	0	1	1	1	5
	SEDIMENTS		VOCs	2	1	2	1	1	1	1	1	1	1	1	1	2	1	2	7
			SELECT METALS	2	1	2	1	1	1	1	1	1	0	0	0	1	1	1	5

Section 4
Project Organization and Responsibility

At the direction of the Region 5 Remedial Project Manager (RPM), with final authority by the Region 5 Regional Project Officer (RPO), CH2M HILL has overall responsibility for all phases of the groundwater monitoring plan, including overall management and QA/QC for all activities within their control. CH2M HILL will perform the field sample collection, field screening, and prepare the study report. The project organization chart is included as Figure 4.

4.1 Management Responsibilities

Project management will be conducted through CH2M HILL's regional office in Milwaukee. Contact will be maintained with the EPA's RPM during all phases of the project.

Monthly reports will be submitted to keep the EPA apprised of the technical, financial, and schedule status of the project. Other CH2M HILL responsibilities include controlling budgets and schedules; selecting, coordinating, and scheduling staff and subcontractors for task assignments; and maintaining project QA/QC programs.

Operational responsibilities involving execution and direct management of the technical and administrative aspects of this project have been assigned as follows:

- Regional Project Officer
  Stephen Nathan (U.S. EPA Region 5)
- Remedial Project Manager
  Kevin Adler (U.S. EPA Region 5)
- State Project Manager (SPM)
  Robin Schmidt (WDNR)
- Site Manager (SM)
  Stevan Keith (CH2M HILL)
- Program Manager (PM)
  Alpheus Sloan (CH2M HILL)

The responsibilities of the aforementioned personnel are described below.

Remedial Project Manager

The RPM has the responsibility for the implementation of the Remediation Plan.

State Project Manager

The State Project Manager has responsibility for ensuring that the Remedial implementation meets WDNR regulations and guidelines.

Program Manager

The PM has overall responsibility for seeing that the project meets EPA and state objectives and CH2M HILL's quality standards. In addition, he is responsible for technical quality control and project oversight, and will provide the site manager with access to corporate management.

Site Manager

The Site Manager (SM) is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The SM's primary function is to see that technical, financial, and scheduling objectives are achieved. The SM will report directly to the RPM and SPM and will provide the major point of contact and control for matters concerning the project. The SM will:

- Define project objectives and develop a detailed work plan schedule
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task
- Acquire and apply technical and corporate resources as needed to maintain performance within budget and schedule constraints
- Orient all field leaders and support staff concerning the project's special considerations
- Monitor and direct the field leaders
- Develop and meet ongoing project and/or task staffing requirements, and develop mechanisms to review and evaluate each task product
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness

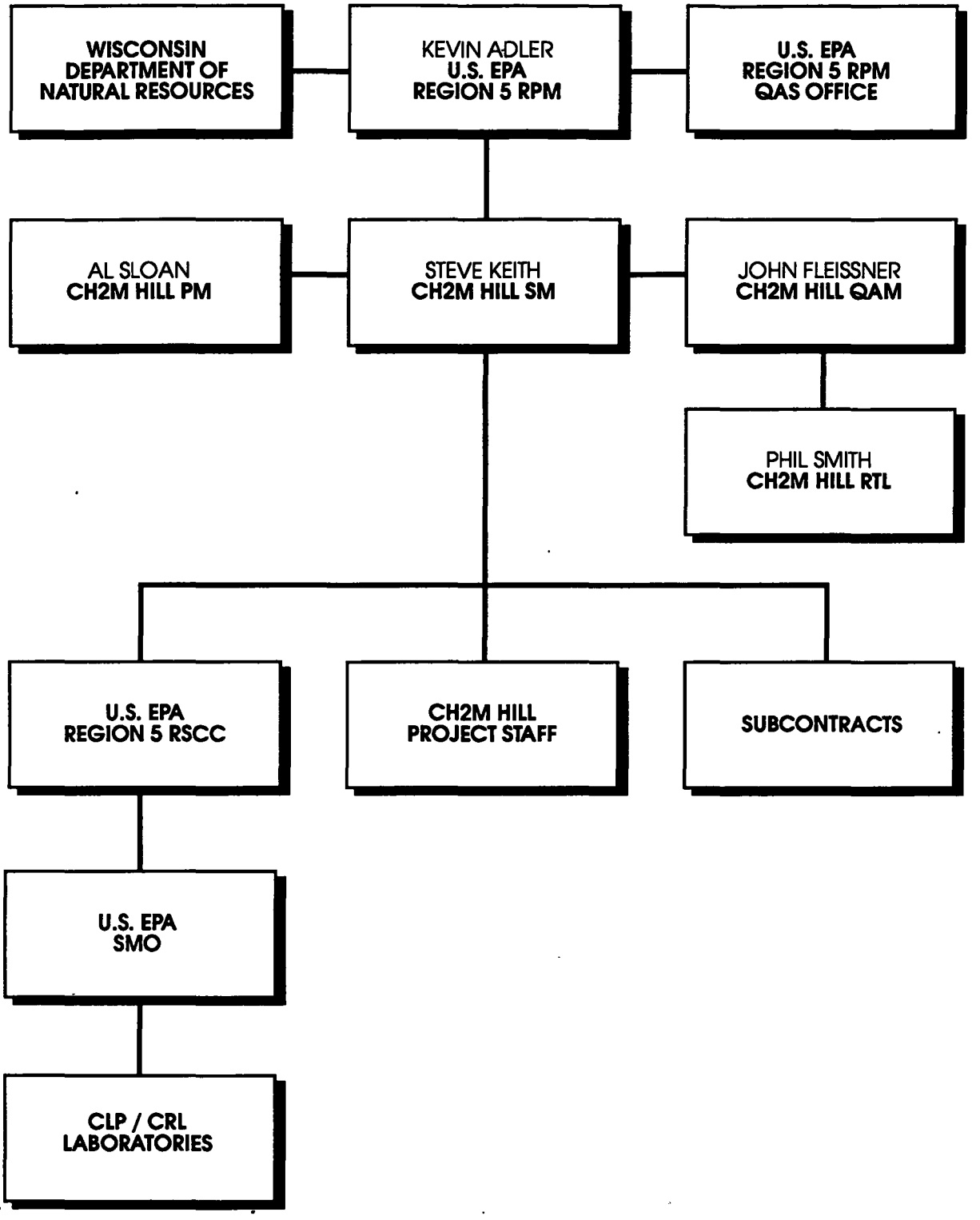

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FIGURE 4
PROJECT ORGANIZATION
ONALASKA
GWMP and QAPP

- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all external reports (deliverables) before their submission to EPA Region 5 and WDNR
- Be responsible for preparation and quality of interim and final reports
- Represent the project team at meetings and public hearings

4.2 Quality Assurance Organization

Responsibilities for management and execution of QA aspects of this project are assigned as follows:

Tasks	Responsible Organization/Personnel
• Final review and approval of QAPjP	Kevin Adler, U.S. EPA Region 5 RPM U.S. EPA Region 5 QA Officer
• QA program for CLP, SAS performance and systems audits for CLP SAS	U.S. EPA Headquarters U.S. EPA Sample Management Office U.S. EPA Region 5 CRL LSSS U.S. EPA EMSL–Las Vegas, QA Division

• QA review and approval of reports, plans and procedures, and field activities; and identifying and controlling non-conformance for corrective action while providing technical assistance to project staff. The QAD will remain independent of direct job involvement and day-to-day operations, and will have direct access to corporate executive staff as necessary to resolve any QA dispute.	John Fleissner, CH2M HILL, Quality Assurance Director (QAD)
• Evidence audits of field records	John Fleissner, CH2M HILL, QAD NEIC Evidence Audit Team (Techlaw, Inc.)
• Performance and systems audits of U.S. EPA CRL	U.S. EPA Region 5 QC CRL Coordinator U.S. EPA Region 5 QA Officer
• Approval of QA programs and laboratory SAS procedures	U.S. EPA Region 5 QAO U.S. EPA Region 5 CRL

4.3 Field Operations

Responsibilities for field operations tasks including both management and execution of the field work, are assigned as follows:

Tasks	Responsible Organization/Personnel
• Sample Collections	Stevan Keith, CH2M HILL, SM Kevin Adler, U.S. EPA Region 5 RPM
• Field measurements	Stevan Keith, CH2M HILL, SM Kevin Adler, U.S. EPA Region 5 RPM
• External Field Audits	U.S. EPA Region 5 CRL

- Internal Field Audits — Stevan Keith, CH2M HILL, SM

The responsibilities of the field team leader and field team members are described below.

Field Team Leaders

The field team leader is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The field team leader is a professional with extensive environmental experience who will report directly to the SM. Specific field team leader responsibilities include:

- Day-to-day coordination with the SM on field activities
- Coordination and management of field staff including sampling and drilling
- Coordination and oversight of technical efforts of subcontractors assisting the field team
- Review all field activities to ensure proper custody procedures are followed
- Implementation of QC for technical data provided by the field staff including field measurement data
- Identification of problems at the field team level, discussion of resolutions with the site manager, and communication between field team and upper management
- Adherence to work schedules provided by the SM
- Participation in the preparation of the final report

Field Team Members

The field team members for this project will be drawn from CH2M HILL's pool of resources. The technical team staff will gather and analyze data and prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

4.4 Laboratories

Organizations and personnel responsible for requesting services, administration of CLP laboratories, and QA/QC tasks associated with the laboratories are assigned as follows:

CLP SAS Tasks	Responsible Organization/Personnel
• Initiation of request	Dave Shekoski, CH2M HILL*
• Preparation of SAS	Daniel MacGregor, CH2M HILL*
• Contact for CLP SAS services	U.S. EPA Region 5 CRL Laboratory Scientific Support Section (LSSS)
• Review and approval of CLP SAS	U.S. EPA Region 5 CRL LSSS U.S. EPA Region 5 Quality Assurance Office Kevin Adler, U.S. EPA, Region 5 RPM
• Data validation of CLP SAS	U.S. EPA Region 5 CRL LSSS
• Data assessment of CLP SAS	Daniel MacGregor, CH2M HILL*

* Contractor's personnel for these tasks may change, subject to staff availability. If a change is made, the EPA RPM will be notified.

Section 5

Quality Assurance Objectives for Measurement Data

The overall QA objectives are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide the quality of data required for monitoring and tracking the groundwater contaminants. Specific procedures to be used for sampling, chain of custody, calibration of field instruments, laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in other sections of this QAPjP and the groundwater monitoring plan (Appendix A). This section addresses the objectives of data precision, accuracy, completeness, representativeness, and comparability.

Precision measures the reproducibility of measurements under a given set of conditions. It is a measure of the variability of a group of measurements compared to an average value. Accuracy measures the bias in a measurement system. Possible sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques. Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or environmental conditions. Completeness is defined as the percentage of measurements made that are judged to be valid measurements. Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another.

5.1 Level of Quality Control Effort

Field blank, trip blank, duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks, consisting of distilled water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the site which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes (MSs) provide information about the effect

of the sample matrix on the extraction/digestion and measurement methodology. Whereas, inorganic analyses require one MS sample, volatile organic matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

The general level of the QC effort will be collecting one field duplicate and one field blank for every 10 or fewer investigative samples. One volatile organic analysis (VOA) trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOA samples.

Aqueous MS/MSD samples must be collected at triple the volume for VOCs and aqueous MS samples must be collected at double the volume for metals, oil and grease, and TOC analyses. Sediment MS/MSD samples require no extra volume for VOCs or inorganic compound analyses. One MS and MS/MSD sample, as appropriate, will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, sediment). Sampling procedures are specified in the Groundwater Monitoring Plan (Appendix A).

The sediment and groundwater samples will be sent to the Contract Laboratory Program/Central Regional Laboratory for analysis. The level of laboratory QC effort for SAS analyses is outlined individually in each SAS contained in Appendix B.

The QC effort for the field measurement of pH consists of pre-measurement calibration and a post-measurement verification using two standard reference solutions. This procedure will be performed daily. The QC effort for field conductivity measurements will include daily calibration of the instrument using standard solutions of known conductivity. The QC effort for temperature will consist of checking the thermometer on the conductivity meter against a certified thermometer. These procedures are described in more detail in the field equipment measurement SOPs (Appendix C).

5.1.1 Accuracy, Precision, and Sensitivity of Analysis

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

The accuracy, precision, and sensitivity requirements for SAS for the CLP are specified in each individual SAS request contained in Appendix B. The standard

operating procedures (SOPs) for the field equipment to measure pH, conductivity, and temperature are outlined in Appendix C. QA requirements for field screening analyses are also included in SOPs found in Appendix C.

5.1.2 Completeness, Representativeness and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the field measurement and CLP laboratories will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{completeness (\%)} = \frac{\text{(number of valid data)}}{\text{(number of sample collected for each parameter analyzed for)}} \times 100$$

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During design and development of the sampling network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale of the sampling network is discussed in detail in the Groundwater Monitoring Plan (Appendix A). Representativeness will be satisfied by seeing that the Groundwater Monitoring Plan is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by analyzing of field duplicated samples.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may

not be directly comparable to existing data because of differences in procedures and QA objectives.

5.2 Method Detection Limits

Contract-required detection limits for the SAS are given in Appendix B.

Section 6

Sampling Procedures

Detailed sampling procedures are provided in the Groundwater Monitoring Plan (Appendix A). Table 4 of Section 3 provides a summary of sample matrices and the parameters to be sampled for.

Section 7

Sample Custody Procedures

7.1 Introduction

It is EPA and Region 5's policy to follow the EPA Region 5 sample custody or chain-of-custody protocols as described in *NEIC Policies and Procedures*, EPA-330/ 9-78-001-R, revised June 1985. This custody is in three parts: field custody procedures, laboratory custody procedures, and final evidence files.

A sample or evidence file is under your custody if the documents:

- Are in your possession
- Are in your view after being in your possession
- Were in your possession and you placed them in a secured location
- Are in a designated secure area

7.2 Field Custody Procedures

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact. The protocol for sample numbering is included in the Groundwater Monitoring Plan.

7.2.1 Field Procedures

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As FEW people as possible should handle the samples.

(b) Each sample bottle will have an EPA Region 5 sample tag attached which will contain the sample number, the case or SAS number, and station location.

(c) Sample tags are to be completed for each sample using waterproof ink.

7.2.2 Sample Documentation Procedures

Sample packaging and shipping procedures are based on U.S. EPA Specifications and Department of Transportation (DOT) regulations (40 CFR). The procedures vary according to sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples will be shipped within 24 hours of collection. Shipping containers must be insulated, durable, and watertight. Sample bottles are to be cushioned within the shipping container with vermiculite packing material (zonolite). To prevent contamination of samples, all containers regardless of size and type must be placed inside sealed plastic bags before being packed in vermiculite or zonolite. Preformed poly-foam cooler liners may be used for shipment of low-concentration samples only. Following shipment, airbill numbers must be called in to the SMO and to the sample documentation coordinator. Field packing and shipping procedures are as follows:

1. Assemble a list of the samples to be packaged and shipped on the same day by their respective analytes and the names of the assigned laboratories.

2. Enter the case number, CRL number, matrix, sample numbers, laboratory, date sampled, and date shipped for each sample on the sample I.D. matrix.

   Note: If portions of a given sample are to be shipped to different laboratories (e.g., for organic and inorganic analysis), two entry lines will be required for that sample number to accommodate the chain-of-custody record, airbill, and traffic report numbers corresponding to each portion of the sample.

3. Obtain the QC lot numbers of the prelabeled containers for each sample and enter them on the sample I.D. matrix.

4. Determine the number of shipping containers (coolers) required for the day's shipment. This will depend on the number of samples to be shipped, the number of containers per sample, the number of sample containers that will fit in each cooler, and the number of laboratories to be used.

Note: A group of containers for a single sample should not be split between coolers unless the portions of the sample are to be sent to more than one laboratory for different types of analysis.

5. Complete an airbill for each laboratory address.

   Note: Several coolers may be shipped to the same address under one airbill.

   Shipment of medium and high concentration samples requires the use of a special airbill, including a shipper's certification for restricted articles.

6. Enter the airbill numbers on the sample I.D. matrix.

7. Assign a chain-of-custody record to each cooler and determine which sample containers will be shipped in each.

   Note: More than one chain-of-custody record may be needed to accommodate the number of samples to be shipped in one cooler.

8. Assign CRL Log numbers to each sample by entering these numbers on the matrix.

   Reminder: Portions of samples for organic and inorganic analysis will usually be sent to separate laboratories. Use one line on the sample I.D. matrix for the organic portion and another line for the inorganic portion.

9. Determine the number of traffic labels that will be needed for organics and inorganics.

10. Assign traffic report numbers from the labels to each sample and enter the numbers on the sample I.D. matrix.

11. Record the tag numbers on each sample container and enter the numbers on the sample I.D. matrix.

12. Complete separate traffic reports for each laboratory each day (or SAS packing lists) based on the information provided on the matrix.

13. Complete sample tags according to the information provided on the sample I.D. matrix and the parameters of analysis. Place tags in groups by sample number.

14. Complete the chain-of-custody records based on the information provided on the sample I.D. matrix.

15. Assign two EPA custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form (if the new combined chain-of-custody/traffic report forms are used, enter the serial numbers in the appropriate box) and temporarily clip seals to the form.

16. Group all the paperwork associated with each cooler in a separate clip.

17. Obtain full signatures of the Sample Team Leader (STL) and initials of significant field team members (including yourself) on the sample tags and at the top of the chain-of-custody forms.

18. Prepare samples for shipment.

All original data recorded on traffic report forms, sample identification tags, chain-of-custody records, and receipt for sample forms will be written with waterproof ink.

Step-by-step instructions for completing each form, plus example forms, are found in Appendix D.

7.2.3 Transfer of Custody Procedures

Transfer of custody procedures are as follows:

1. Samples must be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. The field sampler is personally responsible for the care and custody of the samples until they are transferred or

properly dispatched. As few people as possible should handle the samples. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to or from a secure storage area.

2. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

3. Whenever samples are split with a source or government agency, a separate sample receipt is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "received by" space.

4. All shipments will be accompanied by the chain-of-custody record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to the sampling office.

5. If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

7.2.4 Field Log Book

All information pertinent to a field survey or sampling effort will be recorded in a bound log book or equivalent standard form. Each page or form will be consecutively numbered and will be at least 4½ inches by 7 inches in size. All entries will be made in indelible ink or, if weather conditions dictate, in hard lead pencil, and all corrections will consist of line-out deletions that are initialed and dated. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- End date

At a minimum, the log book will contain the following:

- Purpose of sampling
- Location, description, and log of photographs of the sampling point
- Details of the sampling site (e.g., the elevation of the casing, casing diameter and depth, integrity of the casing, etc.)
- Weather conditions
- Name and address of field contact
- Documentation of procedures for preparation of reagents supplied that become an integral part of the sample (e.g., filters and absorbing reagents)
- Identification of sampling crew members
- Type of sample (e.g., groundwater, soil, sludge, wastewater)

- Suspected waste composition
- Number and volume of samples taken
- Sampling methodology, including distinction between grab and composite samples
- Modifications from Field Sampling Plan
- Sample preservation
- Date and time of collection
- Sample identification designation and tag numbers
- Sample distribution and how transported (i.e., name of the laboratory and transporting agent)
- References such as maps of the sample site
- All field measurements data (e.g., pH, specific conductance, temperature, and water depth)
- Method of field measurement data reduction
- Signature and date by the personnel responsible for observations
- Decontamination procedures

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a log book or standardized form. However, records will contain sufficient information so that someone can reconstruct the sampling activity without relying on the sample collector's memory. The log book and standardized forms will be kept under strict chain of custody.

7.2.5 Corrections to Documentation

No accountable serialized documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document. If an error is made on an accountable document assigned to one individual, that individual shall make corrections by making a single line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

7.2.6 Distribution of Completed Documents

Final disposition of the completed documents is as follows:

- Shipped with samples:
  - Chain-of-custody form, original (if new combined chain-of-custody traffic reports are used, two copies will be required)
  - Traffic report forms, two copies
  - SAS packing list, two copies
  - Sample tags
- Retained by project manager:
  - Sample identification matrix
  - Field log books (at completion of project)
- Sent to CH2M HILL documentation coordinator:
  - Chain-of-custody form, two copies
  - Traffic report forms, original and one copy
  - SAS packing list, original and one copy

7.2.7 Site Manager's Responsibility

The site manager will review all field activities to determine whether proper custody procedures were followed during the fieldwork and decide if additional samples are required. He or she should notify the U.S. EPA Remedial Project Manager of a breach or irregularity in chain-of-custody procedures.

7.3 Laboratory Custody Procedures for The Contract Laboratory

The chain-of-custody procedures for the Contract Laboratory Program (CLP) as described in the U.S. EPA Contract Laboratory Program Statements of Work (OLM 01.0) for organic compounds and the U.S. EPA CLP Statements of Work (ILM 01.0) for inorganic compounds will be followed for the SAS analyses.

Laboratory custody will conform to procedures established for the CLP. These procedures include:

- Designation of a sample custodian
- Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt)
- Laboratory sample tracking and documentation procedures
- Secure sample storage (of the appropriate environment—refrigerated, dry, etc.)
- Proper data logging and documentation procedures including custody of all original laboratory records

7.4 Final Evidence Files Custody Procedures

The final evidence files from the CRL and the CLP are maintained by the U.S. EPA Region 5 CRL. This includes all CRL and/or CLP SAS analytical deliverables, data validation reports, SMO/laboratory telephone conversation records, and purge file records including lab chain-of-custody and sample tags. These files are maintained under document control in a secure area.

The contractor maintains the files along with all relevant records, reports, logs, field notebooks with field measurements and HNu/OVA screenings, pictures, subcontractor reports, and LSSS data reviews in a secured, limited access area and under custody of the contractor's site manager.

Section 8
Calibration Procedures and Frequency

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or scheduled, periodic basis.

8.1 Special Analytical Services

For CLP SAS analysis, the calibration procedures and frequency are presented in the SAS request forms in Appendix B.

8.2 Field Instruments

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used doing the field sampling will be examined to certify that it is operating condition. This includes checking the manufacturing's operating manual and the instruction for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment problem are not overlooked, and all necessary repairs to equipment have been carried out.

Calibration procedures and frequency for field instruments including the OVA and HNu; the pH, specific conductivity, and temperature meters are found in Appendix C.

Section 9
Analytical Procedures

All samples collected during field sampling will be analzed by the CLP or CRL.

9.1 Special Analytical Services

For CLP SAS analyses, the analytical procedures are presented in the SAS request forms in Appendix B. SAS analysis will be performed by CLP/CRL. Also specified in the SAS requests are calibration procedures, frequency of calibration, and the internal quality control checks required for each analysis.

9.2 Field Instruments

Analytical procedures for field instruments including the OVA and HNu, pH, specific conductivity, and temperature meters are found in Appendix C.

Section 10
Internal Quality Control Checks

10.1 Special Analytical Services

For CLP SAS analyses, the analytical QC procedures are presented in the SAS request forms in Appendix B. Section 5 of this QAPjP provides examples of QC checks used for laboratory measurement and analysis.

10.2 Field Instruments

Field analyses will be performed onsite and will not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy that is consistent with that capable of the analytical methodologies and sufficient to meet the intended use of the data. Field QC procedures will be limited to checking the reproducibility of measurements, by taking multiple readings and by verifying accuracy and precision through instrument calibration and calibration checks. The field measurement SOPs in Appendix C describe the QC checks for the field measurements.

Section 11
Data Reduction, Validation, and Reporting

11.1 Data Reduction

11.1.1 Laboratory Analysis

All samples collected at the Onalaska Landfill will be sent to the CLP or CRL. Data reduction, evaluation, and reporting for samples analyzed by the CLP will be performed according to specifications outlined in the CLP RAS SOW (OLM01.1) or the most current version for the organics and SOW (ILM01.1) or the most current version for inorganics. The data will then be sent to the EPA, Region 5, Laboratory Scientific Support Section (LSSS) for data validation. If the CRL is used for analytical services, the data reduction will be performed by the CRL according to specifications outlined in the CRL's SOP on data reduction.

11.1.2 Field Measurements

Raw data from field measurements and sample collection activities will be recorded in the field laboratory notebook. The method of reduction will also be documented in the laboratory notebook.

11.2 Data Validation

11.2.1 Laboratory Analysis

Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements described in the SAS methods. Raw data such as: GC/MS Total Ion Current (TIC) chromatograms, GC/MS mass spectra, FAA data reports, and conventional analyses data station printouts will be examined to ensure that reported results are accurate. The U.S. EPA Region 5 Laboratory Scientific Support Section (LSSS) will be responsible for data validation. The protocol for RAS analyte data validation as presented in the Functional Guidelines (referenced below) will be used to validate the SAS data:

- *Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses*—U.S. EPA, February 1988.

- *Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses*—U.S. EPA, July 1988.

Data validated by the U.S. EPA LSSS will be assessed by CH2M HILL to determine if project objectives and intended data usage requirements were met. If project objectives or data usage requirements are not being met, the program will be modified in such a manner that project requirements are fulfilled.

11.2.2 Field Measurements

Data validation of field measurements will be the responsibility of the Field Team Leader. The field measurement data validation will consist of: the field notebooks being checked to verify that the QC procedures specified in the field measurement SOP in Appendix C were performed, and that the QC procedures were performed correctly and that all calculations are correct. The computer spreadsheet data and results will be proofed against the field notebooks to ensure no transcription errors occurred.

11.3 Data Reporting

11.3.1 Laboratory Analysis

The CRL or SAS analytical laboratories will prepare and submit full analytical and QC reports to EPA Region 5 in compliance with requirements of the CLP to include the following (as applicable):

1. Narrative including statement of samples received, description of any deviations from SAS procedures, explanation of qualifications regarding data quality, and any other significant problems encountered during analysis.

2. An organic QA/QC report including Forms I through X, surrogate spike results for each sample, MS/MSD results, method blank results, and initial and continuing calibration checks.

3. An inorganic QA/QC report including Forms I through XIII, spike and duplicate results, method blank results, MS results, and initial and continuing calibration checks.

4. Field and laboratory chain-of-custody documentation pertaining to each sample delivery group analyzed.

11.3.2 Field Measurements

The data will be transferred from field notebooks to a computer database and output in a spreadsheet format for use in the project reports.

11.4 Annual Evaluation

The quarterly results of the sampling and analysis program will be computed annually. The analytical results will be averaged and the data evaluated to examine spatial and temporal trends. This analysis will also include monthly piezometer readings. The types of spatial and temporal trends conducted will be evaluated after each year of sampling. Initially, the analysis will include:

- Plots of mean concentration versus time for each parameter analyzed for the individual wells

- Plots of concentration versus time of moving averages for each parameter analyzed for individual wells

- Regression analyses on plots of moving average concentration versus time to determine direction of trends

The entire monitoring program also will be reevaluated annually. Specific adjustments to the program that may be necessary include:

- Analyte List—Do analytes need to be added or deleted?

- Sampling Frequencies—Is quarterly sampling (and monthly piezometer readings) adequate or excessive?

- Monitoring Well Network—Is the monitoring well network adequate? Does any well need to be replaced? Should additional wells be installed? Can some of the monitoring wells be deleted from the sampling program?

- Sampling Program—Do the analytical data indicate that the overall concentrations are decreasing? Should the monitoring program continue?

At the end of the fifth year, all sampling results will be compiled. These analytical results will be averaged and evaluated for temporal trends. Compound concentrations will be compared with groundwater standards and evaluated.

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Section 12
Performance and System Audits

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the groundwater monitoring plan and QAPjP. The audits of field and laboratory activities include two separate independent parts: Internal and External audits.

12.1 External Audits

12.1.1 CLP Laboratories

The Contract Laboratory Program (CLP) Special Analytical Service (SAS) laboratories are audited on a regular basis by the U.S. EPA. The U.S. EPA EMSL-Las Vegas conducts the system audits of the CLP laboratories on an annual basis, and conducts performance audits on a quarterly basis.

The system audits, which will include examination laboratory documentation on sample receiving, sample log-in, sample storage chain of custody procedure, sample preparation and analysis, instrument operating records, etc. The performance audits will consist of sending performance evaluation (PE) samples to CLP laboratories for on-going assessment of laboratory precision and accuracy. The analytical results of the analysis of PE samples are evaluated by EPA to ensure the laboratory maintain a good performance.

Additional audits of the SAS laboratories may be conducted by the U.S. EPA Region 5 CRL.

12.1.2 Field Audits

All field activities conducted by CH2M HILL may be subject to onsite audit by the U.S. EPA Region 5 Central District office and/or CRL. Audits will be arranged with the U.S. EPA Remedial Project Manager.

12.2 Internal Audits

12.2.1 Field Audits

Field performance audits are conducted to evaluate the execution of sample identification and control, chain of custody procedures, field documentation, training, and sampling operation. Audits evaluate compliance with the procedures outlined in the QAPjP and GWMP. Field audits will be initiated by the site manager.

The site manager will perform the audit during a sampling event and will keep a written record of the evaluation.

The site manager will evaluate the following items:

- Responsibilities and Organization—To determine if the planned organization is operational and if necessary details of site activities are being communicated to project participants.
- Sample Collection—To ensure that written procedures outlined in the FSP are being implemented.
- Documentation—To ensure that all forms, identification tags, and field notebooks are being prepared and maintained.
- Quality Assurance Checks—To determine that quality control and assurance checks are being performed as specified in the QAPjP and GWMP.
- Field Equipment—To ensure that calibration and maintenance are being done and recorded.
- Training—To ensure that the sampling team members are adequately trained in field sampling and documentation procedures.
- Chain of Custody Procedures—To determine if custody documentation is being completed and maintained and samples are kept in custody at all times.

Following the audit, the auditor will review the preliminary results of his evaluation with the field team leader. The auditor will prepare an audit report containing the results of the evaluation and recommendations for any corrective action. The audit report will be reviewed by the project manager. The site manager shall implement any agreed upon corrective action. The site manager will also be responsible for verifying the implementation of the corrective action. Any noncompliance with standard procedures shall be identified and corrected.

GLT272/042.51

Section 13
Preventive Maintenance

13.1 Laboratory Instruments

The laboratories participating in the CLP SAS program will follow the CLP RAS SOPs for preventive maintenance for each measurement system and required support activity. All instrument maintenance activities will be documented in instrument log books to provide a history of maintenance records. If the CRL is used, lab instruments will be maintained according to CRL SOP.

13.2 Field Instruments

Preventative maintenance for field instruments is found with the field testing procedures in Appendix C.

Section 14

Specific Routine Procedures to Assess Data Precision, Accuracy and Completeness

14.1 Field Measurements

Field data will be assessed by the field leader. The field leader will review the field results for compliance with the established QC criteria that are specified in the QAPjP and GWMP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of reproducibility by multiple reading of a single sample. Upon completion of the field measurements the field data precision will be calculated using Equation 14-2. Data completeness will be calculated using Equation 14-1.

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \qquad \text{Eq. 14-1}$$

14.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, and completeness as follows:

14.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) and field and laboratory duplicate analyses. The relative percent difference (%RPD) will be calculated for each pair of duplicate analysis using the Equation 14-2.

$$\%\ \text{RPD} = \frac{S - D}{(S + D)/2} \times 100 \qquad \text{Eq. 14-2}$$

Where: S = First sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

14.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section III of the QAPjP using the analytical results of method blanks, reagent/preparation blanks, MS and MS/MSD samples, field blanks, and trip blanks. The percent recovery (%R) of spike samples will be calculated using Equation 14-3.

$$\%\ R = \frac{A - B}{C} \times 100 \qquad \text{Eq. 14-3}$$

Where: A = The analyte concentration determined experimentally from the spiked sample;

B = The background level determined by a separate analysis of the unspiked sample;

C = The amount of the spike added.

14.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated using Equation 14-1.

14.3 Project Assessment

Laboratory precision, accuracy and completeness will be calculated by the LSSS as part of data validation. Overall data assessment and data completeness assessment will be determined by CH2M HILL.

Section 15
Corrective Actions

Corrective actions may be required for two classes of problems: analytical and equipment problems, and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the appropriate personnel. If the problem is field realted, the field team leader is promptly notified. If the problem is analytical in nature, information on the problem will be promptly communicated to the U.S. EPA, Quality Assurance Section. The field team leader or Quality Assurance Section will then determine the corrective action. Implementation of corrective action will be confirmed in writing.

15.1 Sample Collection / Field Measurements

Field personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the SM or his designee. The SM will be responsible for assessing the suspected problems in consultation with the Project QAD. A decision will be made based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the manager.

The SM will be responsible for ensuring that corrective action for nonconformances are initiated by:

- Evaluating all reported nonconformances;
- Controlling additional work on nonconforming items;
- Determining disposition or action to be taken;

- Maintaining a written log of nonconformances in a field record book;
- Reviewing nonconformance reports and corrective actions taken;
- Ensuring nonconformance reports are included in the final site documentation in project files.

If appropriate, the SM will see that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- Repeat the measurement to check the error;
- Check for all proper adjustments for ambient conditions such as temperature;
- Check the batteries;
- Re-Calibration;
- Check the calibration;
- Replace the instrument or measurement devices;
- Stop work (if necessary).

The SM or his designee is responsible for all site activities. In this role, the SM, at times, is required to adjust the site programs to accommodate site specific needs. When it becomes necessary to modify a program, the responsible person notifies the RPM of the anticipated change and implements the necessary changes after obtaining their approval. The change in the program will be documented on the field record book. The entry will be signed by the initiators and the SM.

The SM for the Onalaska Landfill site is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties which include the U.S. EPA RPM.

15.2 Laboratory Analyses—Laboratory Corrective Actions

For the CLP Special Analytical Services (SASs), Corrective action is implemented at several different levels. The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures. If CRL is used, corrective actions by CRL will be implemented according to CRL Standard Operating Procedures (SOPs).

The Sample Management Office also may request corrective action for any contractual nonconformance identified by audits or data validation. The CRL may request corrective action by the laboratories for any nonconformances identified in the data validation process through the Sample Management Office or, for minor problems, the lab may be contacted directly. Corrective action may include:

- Re-analyzing the samples, if holding time criteria permits;
- Resampling and analyzing;
- Evaluating and amending sampling procedures and/or evaluating and amending analytical procedures;
- Accepting the data and acknowledging the level of uncertainty.

If resampling is deemed necessary due to laboratory problems, RPM must identify the necessary approach including cost recovery from the CLP for the additional sampling effort.

Section 16

Quality Assurance Reports to Management

The preparation of a separate QA report for this project is not anticipated. The annual reports for the groundwater monitoring plan will contain separate QA sections that summarize data quality information collected during the previous year. Changes in the Field Sampling Plan or QAPjP will be documented in the annual report. The quarterly reports will contain qualified data (if available) summarized in tables.

The contents of the QA section of the annual report will include but not be limited to the following elements:

- Project status
- Indication of whether the QA objectives are being met
- Performance and system audits conducted during the previous monitoring period
- Data validation narrative summary and data quality assessment
- QA problems and corrective actions
- Changes to the QAPjP or Groundwater Monitoring Plan
- Qualified Data summarized in tables

Onalaska Municipal Landfill
Appendix: A
Revision: 0
Date of Revision: 3/31/92

APPENDIX A
GROUNDWATER MONITORING PLAN

Appendix A—Groundwater Monitoring Plan

Provided under separate cover

APPENDIX B
SAS REQUEST FORMS

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of volatile organic compounds at part per trillion (ppt) levels in groundwater, surfacewater and sediment samples using purge and trap capillary column gas chromatography-mass spectrometry (GC/MS). Attachment 1 lists target compounds and detection limits.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

A method detection limit (MDL) study consisting of a minimum of seven replicate analyses and one verification spike sample must be performed.

For each sampling round analyze 17 low concentration groundwater samples. This number is inclusive of investigation and field QC samples (MS/MSD, field and trip blanks).

Annually two surfacewater and two sediment samples will be collected and analyzed for the select, low concentration, volatile organic compounds.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund Remedial

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipments by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required within 21 days of sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Method detection limit study: Federal Register V.49 No.209, (Appendix B of Part 136, 10-26) October 26, 1984, with special technical instructions as noted in section 8.

Analysis: EPA SW-846 Method 8260 (November 1990) with special technical instructions as noted in section 8.

Preparation: Sediment samples will be prepared according to EPA SW-846 Method 5030 (November 1990).

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

See attachment 2.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

See attachment 3.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact:

Dave Shekoski Phone: (414) 272-2426

12. DATA REQUIREMENTS

Parameter	Required Detection Limits	Precision Desired (+/- % or conc.)
See attachment 1	See attachment 1	+/- 20% of target detection limits listed in attachment 1

13. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits (+/- % or conc.)
Internal standards	Each sample, calibration standard, blank and matrix spike	See attachment 2 (item 2A)
Surrogate standards	Each Sample, calibration standard, blank, and matrix spike	See attachment 2 (item 2A)
Matrix spike/matrix spike duplicate	1 set per group of 20 investigative samples or less	As per CLP RAS organic SOW
Method blank	As per attachment 2 and CLP RAS organic SOW	As per attachment 2 and CLP RAS organic SOW
QC Reference Stds	Quarterly	± 25% of true value

14. Action Required if Limits are Exceeded:

See attachment 2 and CLP RAS organic SOW OLM01.0 for corrective actions. Contact the Sample Management Office for problems not outlined.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT 1

Organic Target Analytes	Aqueous Detection Limits (μg/L)	Sediment Detection Limits (μg/Kg)
Benzene	0.03	0.3
Toluene	0.08	0.8
Xylenes	0.02	0.2
Ethylbenzene	0.03	0.3
Trichloroethene	0.02	0.2
1,1-Dichloroethane	0.03	0.3
1,1,1-Trichloroethane	0.04	0.4
1,1-Dichloroethene	0.05	0.5
1,1,2,1-Tetrachloroethylene	0.20	2.0

Attachment 2

Special Technical Instructions

1. **Method Detection Limit (MDL) Study**

The MDL study shall be performed prior to receipt and analysis of field samples. The MDL study shall consist of a statistically determined MDL using the procedure described in the Federal Register (V.49, #209, Appendix B to Part 136, 10-26) and verified through a spike at the computed MDL.

A. Computed MDL

Using the Federal Register procedure, prepare and analyze a minimum of seven replicates containing all target compounds and internal/ surrogate standards (Details of the analysis shall follow EPA SW-846 Method 8260 and instruction No. 2 below).

Use the SAS target detection limits (Attachment 1) as the estimate of the detection limit for the spike levels for the seven replicates. Compute the statistically determined MDL.

B. Verified MDL

Following 1A above, prepare and analyze reagent water spiked with all target compounds (at the computed MDL) and internal/surrogate standards (Details of the analysis shall follow EPA SW-846 Method 8260 and instruction No. 2 below). All qualitative criteria used for identification of target compounds (i.e., presence of ions/abundance, retention times must be met for all compounds (see EPA SW-846 Method 8260). If any compound does not meet all qualitative criteria at the computed MDL, reanalyze a reagent water blank with only the compound(s) which failed the criteria. Contact the SMO coordinator if this reanalysis does not still meet criteria.

C. Target Compounds, Internal Standards, and Surrogate Standards

(1) Target Compounds: See Attachment 1 (only compounds to be reported).

(2) Internal/Surrogate Standards: See instruction No. 2 below.

D. Data Deliverables

See Attachment 3.

2. Sample Analysis

Analysis of all samples shall follow EPA SW-846 Method 8260 (attached) with modifications/specifications outlined below. **Corrective actions and QC limits shall follow the CLP RAS Organic SOW OLM01.0 unless otherwise noted below.**

A. Internal Standards

The internal standard compounds shall be pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Spiked at a concentration level of 1.0 μg/L.

B. Surrogate Standards

Surrogate standards shall additionally be spiked into all samples, blanks, calibration standards, matrix spike/matrix spike duplicate samples, etc. The surrogate standard compound shall be toluene-d_5, 4-bromofluorobenzene, and dibromofluoromethane. The concentration of each surrogate shall be equivalent to the internal standards (1.0 μg/L) in each investigative and QC sample. Prepare according to EPA SW-846 Method 8260, Section 5.7 and 5.9 and spike when internal standards are introduced (Section 7.5.1). Recovery limits are: toluene-d_5 (88 to 110 percent), 4-bromofluorobenzene (86 to 115 percent), and dibromofluoromethane (86 to 118 percent).

C. Tuning Criteria

Bromofluorobenzene (BFB) tuning criteria (EPA SW-846 Method 8260) Table 4 and Section 7.4.1 must be every 12 hours and prior to analysis of any calibration standard, sample, blank, etc.

D. Calibration

(1) Initial Calibration

Initial calibration shall consist of five points as discussed in EPA SW-846 Method 8260, Section 5.12. These calibration points shall be prepared as described in Section 5.12 of the method. RFs of all other compounds must be $\geq$ 0.05. The percent relative standard deviation (percent RSD) for the RFs of all compounds must be $\leq$ 35 percent.

(2) Continuing Calibration

A continuing calibration standard shall be analyzed every 12 hours containing all compounds at a concentration near the mid point concentration for the working range of GC/MS. The continuing calibration response factors shall be used to quantitate all samples analyzed. Spiked at concentrations as described in the method? The RFs of the compounds must be ≥ 0.05. The percent difference (percent D) for the RFs of all compounds must be ≤ 25 percent.

E. Matrix Spike/Matrix Spike Duplicate

A matrix spike/matrix spike duplicate sample shall be selected by field samplers and shall be additionally analyzed as 1 set per group of 20 samples. The spike compounds will consist of all target compounds (benzene, toluene, xylenes, ethylbenzene, trichloroethene, 1,1-dichloroethane, 1,1,1-trichloroethane, 1,1-dichloroethene, and 1,1,2,1-tetrachloroethylene) at concentrations representative of what is expected to be found in the samples. Calculate spike recovery (percent R) and relative percent difference (RFD) as per the CLP RAS organic SOW OLM01.0.

F. Qualitative/Quantitative Analysis

(1) EPA SW-246 Method 8260, Table 5 defines the primary and secondary characteristic masses used for the SAS target compounds (Attachment 5).

G. Method Blanks

Method blanks shall be analyzed every 12 hours after initial and continuing calibration standards. An acceptable method blank must contain less than five times the verified MDL for methylene chloride, acetone, toluene, and 2-butanone and less than the MDL for the other target compounds.

H. Dilutions

If samples require dilutions in order to bring some compounds within the calibration range, the lab shall report both the undiluted and diluted result (including all CLP SOW deliverables) where compounds are quantitated. Results from samples requiring dilution will be qualified as diluted and flagged with a "D."

I. Preservation/Container Requirements

Samples will arrive preserved with HCL to a pH < 2. Three VOA vials (40 mL each) will be sent per sample.

3. **QC Reference Samples**

QC reference sample ampules will be provided to the laboratory. Separate sample preparation instructions will be provided for appropriate dilution. Target levels shall be approximately mid-calibration range.

A reference will be analyzed at least once by each laboratory performing sample analyses.

GLT301/001.51

Attachment 3
Analytical Results Required

The data deliverables as described in the current CLP RAS Organic SOW OLM01.0 shall be used. The method detection limit study raw data shall also be submitted.

The laboratory shall report its own verified detection limits on the Form 1s for all field and analytical samples.

GLT301/002.51

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

Definition

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 analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a signifi-

cantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 \Big/ n\right]$$

$$S = (S^2)^{1/2}$$

where:

X_i; i=1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i=1 to n.

6. (a) Compute the MDL as follows:

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

where:

MDL = the method detection limit

$t_{(n-1,1-\alpha\ =\ .99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL = 0.64 MDL

UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A/S^2_B<3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S^2_A + 6S^2_B}{12}\right]^{1/2}$$

if $S^2_A/S^2_B>3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the S_{pooled} as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681\ (S_{pooled})$$

where 2.681 is equal to $t_{(13,\ 1-\alpha\ =.99)}$.

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from precentiles of the chi squared over degrees of freedom distribution.

LCL=0.72 MDL

UCL=1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates	Degrees of freedom (n-1)	$t_{(n-1,\ .99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
00	00	2.326

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which

affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See Section 5.)

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. (See Section 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. 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 and as required.

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 instruction provided by the manufacturer of the particular instrument.

2. Summary of Method

2.1 The method describes a technique for the simultaneous or 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. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) 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 9.3), or the sum of the dissolved plus suspended concentrations. (Section 9.1 plus 9.2).

3.4 *Total recoverable*—The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (Section 9.4).

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. functional relationship between emission intensity and concentration.

3.7 *Instrument check standard*—A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1)

ATTACHMENT 5

METHOD 8260

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Method 8260 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 ground water, 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. The following compounds can be determined by this method:

Analyte	CAS No.[b]	Appropriate Technique	
		Purge-and-Trap	Direct Injection
Benzene	71-43-2	a	a
Bromobenzene	108-86-1	a	a
Bromochloromethane	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
n-Butylbenzene	104-51-8	a	a
sec-Butylbenzene	135-98-8	a	a
tert-Butylbenzene	98-06-6	a	a
Carbon tetrachloride	56-23-5	a	a
Chlorobenzene	108-90-7	a	a
Chloroethane	75-00-3	a	a
Chloroform	67-66-3	a	a
Chloromethane	74-87-3	a	a
2-Chlorotoluene	95-49-8	a	a
4-Chlorotoluene	106-43-4	a	a
Dibromochloromethane	124-48-1	a	a
1,2-Dibromo-3-chloropropane	96-12-8	pp	a
1,2-Dibromoethane	106-93-4	a	a
Dibromomethane	74-95-3	a	a
1,2-Dichlorobenzene	95-50-1	a	a
1,3-Dichlorobenzene	541-73-1	a	a
1,4-Dichlorobenzene	106-46-7	a	a
Dichlorodifluoromethane	75-71-8	a	a
1,1-Dichloroethane	75-34-3	a	a
1,2-Dichloroethane	107-06-2	a	a
1,1-Dichloroethene	75-35-4	a	a
cis-1,2-Dichloroethene	156-59-2	a	a
trans-1,2-Dichloroethene	156-60-5	a	a
1,2-Dichloropropane	78-87-5	a	a
1,3-Dichloropropane	142-28-9	a	a

Analyte	CAS No.[b]	Appropriate Technique: Purge-and-Trap	Appropriate Technique: Direct Injection
2,2-Dichloropropane	594-20-7	a	a
1,1-Dichloropropene	563-58-6	a	a
Ethylbenzene	100-41-4	a	a
Hexachlorobutadiene	87-68-3	a	a
Isopropylbenzene	98-82-8	a	a
p-Isopropyltoluene	99-87-6	a	a
Methylene chloride	75-09-2	a	a
Naphthalene	91-20-3	a	a
n-Propylbenzene	103-65-1	a	a
Styrene	100-42-5	a	a
1,1,1,2-Tetrachloroethane	630-20-6	a	a
1,1,2,2-Tetrachloroethane	79-34-5	a	a
Tetrachloroethene	127-18-4	a	a
Toluene	108-88-3	a	a
1,2,3-Trichlorobenzene	87-61-6	a	a
1,2,4-Trichlorobenzene	120-82-1	a	a
1,1,1-Trichloroethane	71-55-6	a	a
1,1,2-Trichloroethane	79-00-5	a	a
Trichloroethene	79-01-6	a	a
Trichlorofluoromethane	75-69-4	a	a
1,2,3-Trichloropropane	96-18-4	a	a
1,2,4-Trimethylbenzene	95-63-6	a	a
1,3,5-Trimethylbenzene	108-67-8	a	a
Vinyl chloride	75-01-4	a	a
o-Xylene	95-47-6	a	a
m-Xylene	108-38-3	a	a
p-Xylene	106-42-3	a	a

a Adequate response by this technique.
b Chemical Abstract Services Registry Number.
pp Poor purging efficiency resulting in high EQLs.
i Inappropriate technique for this analyte.
pc Poor chromatographic behavior.

1.2 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for lists of analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25 mL sample volumes are presented.

1.3 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is approximately 5 µg/Kg (wet weight) for soil/sediment samples, 0.5 mg/Kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.4 Method 8260 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 into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large bore capillary or cryofocussed on a capillary precolumn before being flash evaporated to a narrow bore capillary for analysis. The column is temperature programmed to separate the analytes which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph. Wide bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in solvent to dissolve the volatile organic constituents. A portion of the solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.3 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Each identified component is quantitated by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter (Figure 1). Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks

result in what the laboratory feels is a false positive for a sample, this should be fully explained in text accompanying the uncorrected data.

3.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing of the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning. Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

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

4.0 APPARATUS AND MATERIALS

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

4.1.1 The recommended purging chamber is designed to accept 5 mL (and 25 mL if the lowest detection limit is required) samples with a water column at least 3 cm deep. The gaseous headspace 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. Alternate sample purge devices (i.e. needle spargers), may be utilized, provided equivalent performance is demonstrated.

4.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figure 2). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples. Traps normally last 2-3 months when used daily. Some signs of a deteriorating trap are: uncharacteristic recoveries of surrogates, especially toluene-d_8; a loss of the response of the internal standards during a 12 hour shift; and/or a rise in the baseline in the early portion of the scan.

4.1.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The trap bake-out temperature should not exceed 220°C. The desorber design illustrated in Figure 2 meets these criteria.

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

4.1.5 Trap Packing Materials

4.1.5.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.1.5.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

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

4.1.5.4 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through a 26 mesh screen (or equivalent).

4.2 Heater or heated oil bath - Should be capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.

4.3 Gas chromatography/mass spectrometer/data system

4.3.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout

desorption and temperature program operation. For some column configuration, the column oven must be cooled to < 30°C, therefore, a subambient oven controller may be required. The capillary column should be directly coupled to the source.

4.3.1.1 Capillary precolumn interface when using cryogenic cooling - This device interfaces the purge and trap concentrator to the capillary gas chromatograph. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused silica capillary precolumn. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.3.1.1.1 During the cryofocussing step, the temperature of the fused silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.3.2 Gas chromatographic columns

4.3.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5 μm film thickness, or equivalent.

4.3.2.2 Column 2 - 30 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific) or VOCOL (Supelco), 3 μm film thickness, or equivalent.

4.3.2.3 Column 3 - 30 m x 0.32 mm ID capillary column coated with DB-5 (J&W Scientific) or SE-54 (Supelco), 1 μm film thickness, or equivalent.

4.3.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 50 ng of the GC/MS tuning standard (BFB) is injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

4.3.4 GC/MS interface - The GC is interfaced to the MS with an all glass enrichment device and an all glass transfer line, but any enrichment device or transfer line can be used if the performance specifications described in Section 8.2 can be achieved. Any GC-to-MS interface that gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria (see Table 4) may be used. GC-to-MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane. This interface is only needed for the wide bore columns (≥ 0.53 mm ID).

4.3.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified 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 abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.5 Microsyringes - 10, 25, 100, 250, 500, and 1,000 µL.

4.6 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.7 Syringes - 5, 10, or 25 mL, gas-tight with shutoff valve.

4.8 Balance - Analytical, 0.0001 g, and top-loading, 0.1 g.

4.9 Glass scintillation vials - 20 mL, with Teflon lined screw-caps or glass culture tubes with Teflon lined screw-caps.

4.10 Vials - 2 mL, for GC autosampler.

4.11 Disposable pipets - Pasteur.

4.12 Volumetric flasks, Class A - 10 mL and 100 mL, with ground-glass stoppers.

4.13 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Tetraglyme - Reagent tetraglyme is defined as tetraglyme in which interference is not observed at the method detection limit of compounds of interest.

CAUTION: Glycol ethers are suspected carcinogens. All solvent handling should be done in a hood while using proper protective equipment to minimize exposure to liquid and vapor.

5.4.1 Tetraglyme (tetraethylene glycol dimethyl ether, Aldrich #17, 240-5 or equivalent), $C_8H_{18}O_5$ - Purify by treatment at reduced pressure in a rotary evaporator. The tetraglyme should have a peroxide content of less than 5 ppm as indicated by EM Quant Test Strips (available from Scientific Products Co., Catalog No. P1126-8 or equivalent).

5.4.1.1 Peroxides may be removed by passing the tetraglyme through a column of activated alumina. The tetraglyme 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 a vacuum is maintained at < 10 mm Hg for at least two hours 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. Cool the tetraglyme to ambient temperature and add 100 mg/L of 2,6-di-tert-butyl-4-methyl-phenol to prevent peroxide formation. Store the tetraglyme in a tightly sealed screw-cap bottle in an area that is not contaminated by solvent vapors.

5.4.2 In order to demonstrate that all interfering volatiles have been removed from the tetraglyme, an organic-free reagent water/tetraglyme blank must be analyzed.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$ - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10 mL tared 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 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100 µL syringe, immediately add two or more 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.

5.7.2.2 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 the 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 will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) 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.

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

5.7.5 Prepare fresh standards for gases every two months or sooner if comparison with check standards indicates a problem. Reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently. All other standards must be replaced after six months, or sooner if comparison with check standards indicates a problem. Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to QC check standards. It may be necessary to replace the standards more frequently if either check exceeds a 25% difference.

5.8 Secondary dilution standards - Using stock standard solutions, prepare in methanol, secondary dilution standards containing the compounds of interest, either singly or mixed together. Secondary dilution standards must 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. Store in a vial with no headspace for one week only.

5.9 Surrogate standards - The surrogates recommended are toluene-d_8, 4-bromofluorobenzene, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described in Section 5.7, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis.

5.10 Internal standards - The recommended internal standards are chlorobenzene-d_5, 1,4-difluorobenzene, 1,4-dichlorobenzene-d_4, and pentafluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Sections 5.7 and 5.8. It is recommended that the secondary dilution standard should be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/μL of BFB in methanol should be prepared.

5.12 Calibration standards - Calibration standards at a minimum of five concentrations should be prepared from the secondary dilution of stock standards (see Sections 5.7 and 5.8). Prepare these solutions in organic-free reagent water. One of the concentrations should be at a concentration near, but above, the method detection limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table 1 may be included). Calibration standards must be prepared daily.

5.13 Matrix spiking standards - Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. It is desirable to perform a matrix spike using compounds found in samples. Some permits may require spiking specific compounds of interest, especially if they are polar and would not be represented by the above listed compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 μg/10.0 mL.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C to -20°C in amber bottles with Teflon lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Direct injection - In very limited applications (e.g. aqueous process wastes) direct injection of the sample into the GC/MS system with a 10 μL syringe may be appropriate. One such application is for verification of the alcohol content of an aqueous sample prior to determining if the sample is ignitable (Methods 1010 or 1020). In this case, it is suggested that direct injection be used. The detection limit is very high (approximately 10,000 μg/L). Therefore, it is only permitted when concentrations in excess of 10,000 μg/L are expected, or for water-soluble compounds that do not purge. The system must be calibrated by direct injection using the same solvent (e.g. water) for standards as the sample matrix (bypassing the purge-and-trap device).

7.2 Chromatographic conditions

7.2.1 General:

Injector temperature:	200-225°C
Transfer line temperature:	250-300°C

7.2.2 Column 1 (A sample chromatogram is presented in Figure 5)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 160°C
Final temperature:	160°C, hold until all expected compounds have eluted.

7.2.3 Column 2, Cryogenic cooling (A sample chromatogram is presented in Figure 6)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 160°C
Final temperature:	160°C, hold until all expected compounds have eluted.

7.2.4 Column 2, Non-cryogenic cooling (A sample chromatogram is presented in Figure 7)

Carrier gas flow rate: It is recommended that carrier gas flow and split and make-up gases be set using performance of standards as guidance. Set the carrier gas head pressure to ≈ 10 psi and the split to ≈ 30 mL/min. Optimize the make-up gas flow for the separator (approximately 30 mL/min) by injecting BFB, and determining the optimum response when varying the make-up gas. This will require several injections of BFB. Next, make several injections of the volatile working standard with all analytes of interest. Adjust the carrier and split to provide optimum chromatography and response. This is an especially critical adjustment for the volatile gas analytes. The head pressure should optimize between 8-12 psi and the split between 20-60 mL/min. The use of the splitter is important to minimize the effect of water on analyte response, to allow the use of a larger volume of helium during trap desorption, and to slow column flow.

Initial temperature:	45°C, hold for 2 minutes
Temperature program:	8°C/min to 200°C
Final temperature:	200°C, hold for 6 minutes.

A trap preheated to 150°C prior to trap desorption is required to provide adequate chromatography of the gas analytes.

7.2.5 Column 3 (A sample chromatogram is presented in Figure 8)

Carrier gas (He) flow rate:	4 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 70°C, then 15°C/min to 145°C
Final temperature:	145°C, hold until all expected compounds have eluted.

7.3 Initial calibration for purge-and-trap procedure

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 50 ng injection or purging of 4-bromofluorobenzene (2 μL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.2 Assemble a purge-and-trap device that meets the specification in Section 4.1. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 minutes while backflushing at 180°C with the column at 220°C.

7.3.3 Connect the purge-and-trap device to a gas chromatograph.

7.3.4 A set of at least five calibration standards containing the method analytes is needed. One calibration standard should contain each analyte at a concentration approaching but greater than the method detection limit (Table 1) for that compound; the other calibration standards should contain analytes at concentrations that define the range of the method. The purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed. To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 μL of internal standard. Then transfer the contents to a purging device.

7.3.5 Carry out the purge-and-trap analysis procedure as described in Section 7.5.1.

7.3.6 Tabulate the area response of the characteristic ions (see Table 5) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured (Section 7.6.2). The RF is calculated as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the compound being measured.

7.3.7 The average RF must be calculated and recorded for each compound. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.3.7.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

7.3.7.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.7.3 Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.8 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs). Record the %RSDs for all compounds. The percent RSD is calculated as follows:

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where:

RSD = Relative standard deviation.
$\bar{x}$ = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N - 1}}$$

The %RSD for each individual CCC must be less than 30 percent. This criterion must be met for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

7.4 Daily GC/MS calibration

7.4.1 Prior to the analysis of samples, inject or purge 50 ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift.

7.4.2 The initial calibration curve (Section 7.3) for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC (Section 7.4.3) and CCC (Section 7.4.4).

7.4.3 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.4.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed in Section 7.3.8 are used to check the validity of the initial calibration. Calculate the percent difference using the following equation:

$$\% \text{ Difference} = \frac{\overline{RF_i} - RF_c}{\overline{RF_i}} \times 100$$

where:

$\overline{RF_i}$ = Average response factor from initial calibration.
RF_c = Response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (> 25% difference), for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before quantitative sample analysis begins. If the CCCs are not required analytes

by the permit, then all required analytes must meet the 25% difference criterion.

7.4.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.5 GC/MS analysis

7.5.1 Water samples

7.5.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be used are the headspace sampler (Method 3810) using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector (HECD), and extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (Method 3820).

7.5.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.5.1.3 Set up the GC/MS system as outlined in Sections 4.3 and 7.2.

7.5.1.4 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 7.4) before analyzing samples.

7.5.1.5 Adjust the purge gas (helium) flow rate to 25-40 mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response (see Section 7.3.7).

7.5.1.6 Remove the plunger from a 5 mL syringe and attach a closed syringe valve. If lower detection limits are required, use a 25 mL syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, 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. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial,

the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20 mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.5.1.7.1 Dilutions may be made in volumetric flasks (10 to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.5.1.7.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.1.7.3 Inject the proper aliquot of sample from the syringe prepared in Section 7.5.1.6 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.1.7.4 Fill a 5 mL syringe with the diluted sample as in Section 7.5.1.6.

7.5.1.8 Compositing samples prior to GC/MS analysis

7.5.1.8.1 Add 5 mL or equal larger amounts of each sample (up to 5 samples are allowed) to a 25 mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

7.5.1.8.2 The samples must be cooled at 4°C during this step to minimize volatilization losses.

7.5.1.8.3 Mix well and draw out a 5 mL aliquot for analysis.

7.5.1.8.4 Follow sample introduction, purging, and desorption steps described in the method.

7.5.1.8.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used unless a 25 mL sample is to be purged.

7.5.1.9 Add 10.0 µL of surrogate spiking solution (Section 5.9) and 10 µL of internal standard spiking solution (Section 5.10) through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of sample is equivalent to a concentration of 50 µg/L of each surrogate standard.

7.5.1.10 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.

7.5.1.11 Close both valves and purge the sample for 11.0 ± 0.1 minutes at ambient temperature. Be sure the trap is cooler than 25°C.

7.5.1.12 Sample desorption - The mode of sample desorption is determined by the type of capillary column employed for the analysis. When using a wide bore capillary column, follow the desorption conditions of Section 7.5.1.13. The conditions for using narrow bore columns are described in Section 7.5.1.14.

7.5.1.13 Sample desorption for wide bore capillary column. Under most conditions, this type of column must be interfaced to the MS through an all glass jet separator.

7.5.1.13.1 After the 11 minute purge, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4) and initiate the temperature program sequence of the gas chromatograph and start data acquisition. Introduce the trapped materials to the GC column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas at 15 mL/min for 4 minutes. If the non-cryogenic cooling technique is followed, the trap must be preheated to 150°C just prior to trap desorption at 180°C. While the purged analytes are being introduced into the gas chromatograph, empty the purging device using the sample syringe and wash the chamber with two 5 mL or 25 mL portions of organic-free reagent water depending on the size of the purge device. After the purging device has been emptied, leave the syringe valve open to allow the purge gas to vent through the sample introduction needle.

7.5.1.13.2 Hold the column temperature at 10°C for 5 minutes, then program at 6°C/min to 160°C and hold until all analytes elute.

7.5.1.13.3 After desorbing the sample for 4 minutes, condition the trap by returning the purge-and-trap system to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately 7 minutes, turn off the trap heater and open the syringe valve

to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

7.5.1.14 Sample desorption for narrow bore capillary column. Under normal operating conditions, most narrow bore capillary columns can be interfaced directly to the MS without a jet separator.

7.5.1.14.1 After the 11 minute purge, attach the trap to the cryogenically cooled interface at -150°C and adjust the purge-and-trap system to the desorb mode (Figure 4). Introduce the trapped materials to the interface by rapidly heating the trap to 180°C while backflushing the trap with an inert gas at 4 mL/min for 5 minutes. While the extracted sample is being introduced into the interface, empty the purging device using the sample syringe and rinse the chamber with two 5 mL or 25 mL portions of organic-free reagent water depending on the size of the purging device. After the purging device has been emptied, leave the syringe valve open to allow the purge gas to vent through the sample introduction needle. After desorbing for 5 minutes, flash heat the interface to 250°C and quickly introduce the sample on the chromatographic column. Start the temperature program sequence, and initiate data acquisition.

7.5.1.14.2 Hold the column temperature at 10°C for 5 minutes, then program at 6°C/min to 70°C and then at 15°C/min to 145°C. After desorbing the sample for 5 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately 15 minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

7.5.1.15 If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.5.1.16 For matrix spike analysis, add 10 μL of the matrix spike solution (Section 5.13) to the 5 mL of sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50 μg/L of each matrix spike standard.

7.5.1.17 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the

linear range of the curve. Proceed to Sections 7.6.1 and 7.6.2 for qualitative and quantitative analysis.

7.5.2 Water-miscible liquids

7.5.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50 fold with organic-free reagent water.

7.5.2.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100 mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a 5 mL gas-tight syringe.

7.5.2.3 Alternatively, prepare dilutions directly in a 5 mL syringe filled with organic-free reagent water by adding at least 20 μL, but not more than 100 μL of liquid sample. The sample is ready for addition of internal and surrogate standards.

7.5.3 Sediment/soil and waste samples - It is highly recommended that all samples of this type be screened prior to the purge-and-trap GC/MS analysis. The headspace method (Method 3810) or the hexadecane extraction and screening method (Method 3820) may used for this purpose. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. Use the screening data to determine whether to use the low-concentration method (0.005-1 mg/Kg) or the high-concentration method (> 1 mg/Kg).

7.5.3.1 Low-concentration method - This is designed for samples containing individual purgeable compounds of < 1 mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. Analyze all blanks and standards under the same conditions as the samples. See Figure 9 for an illustration of a low soils impinger.

7.5.3.1.1 Use a 5 g sample if the expected concentration is < 0.1 mg/Kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/Kg.

7.5.3.1.2 The GC/MS system should be set up as in Sections 7.5.1.3-7.5.1.4. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

7.5.3.1.3 Remove the plunger from a 5 mL Luerlock type syringe equipped with a syringe valve and fill until

overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 µL each of surrogate spiking solution (Section 5.9) and internal standard solution (Section 5.10) to the syringe through the valve (surrogate spiking solution and internal standard solution may be mixed together). The addition of 10 µL of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 µg/Kg of each surrogate standard.

7.5.3.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Section 7.5.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.5.3.1.5 Determine the percent dry weight of the soil/sediment sample. This includes waste samples that are amenable to percent dry weight determination. Other wastes should be reported on a wet-weight basis.

7.5.3.1.5.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the % dry weight of the sample by drying overnight at 105°C. Allow to cool in a desiccator before re-weighing. Concentrations of individual analytes are reported relative to the dry weight of sample.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

7.5.3.1.6 Add the spiked organic-free reagent water to the purging device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

NOTE: Prior to the attachment of the purge device, the procedures in Sections 7.5.3.1.4 and 7.5.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.5.3.1.7 Heat the sample to 40°C ± 1°C and purge the sample for 11.0 ± 0.1 minutes. Be sure the trap is cooler than 25°C.

7.5.3.1.8 Proceed with the analysis as outlined in Sections 7.5.1.12-7.5.1.17. Use 5 mL of the same organic-free reagent water as in the blank. If saturated peaks occurred

or would occur if a 1 g sample were analyzed, the high-concentration method must be followed.

7.5.3.1.9 For low-concentration sediment/soils, add 10 µL of the matrix spike solution (Section 5.7) to the 5 mL of organic-free reagent water (Section 7.5.3.1.3). The concentration for a 5 g sample would be equivalent to 50 µg/Kg of each matrix spike standard.

7.5.3.2 High-concentration method - The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e. petroleum and coke wastes) that are insoluble in methanol are diluted with tetraglyme or possibly polyethylene glycol (PEG). An aliquot of the extract is added to organic-free reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/Kg should be analyzed by this method.

7.5.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol weigh 4 g (wet weight) of sample into a tared 20 mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent dry weight of the sample using the procedure in Section 7.5.3.1.5. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10 mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of solvent into the vial and mark the bottom of the meniscus. Discard this solvent.)

7.5.3.2.2 Quickly add 9.0 mL of appropriate solvent; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes.

NOTE: Sections 7.5.3.2.1 and 7.5.3.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.5.3.2.3 Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed. Transfer approximately 1 mL of appropriate solvent to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis. The addition of a 100 µL aliquot of each of these extracts in Section 7.5.3.2.6 will give a concentration equivalent to 6,200 µg/Kg of each surrogate standard.

7.5.3.2.4 The GC/MS system should be set up as in Sections 7.5.1.3-7.5.1.4. This should be done prior to the addition of the solvent extract to organic-free reagent water.

7.5.3.2.5 The information in Table 10 can be used to determine the volume of solvent extract to add to the 5 mL of organic-free reagent water for analysis. If a screening procedure was followed (Method 3810 or 3820), use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100 µL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.3.2.6 Remove the plunger from a 5.0 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 µL of internal standard solution. Also add the volume of solvent extract determined in Section 7.5.3.2.5 and a volume of extraction or dissolution solvent to total 100 µL (excluding solvent in standards).

7.5.3.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/solvent sample into the purging chamber.

7.5.3.2.8 Proceed with the analysis as outlined in Sections 7.5.1.12-7.5.1.17. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 µL of the dilution solvent to simulate the sample conditions.

7.5.3.2.9 For a matrix spike in the high-concentration sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution (Section 5.9), and 1.0 mL of matrix spike solution (Section 5.13) as in Section 7.5.3.2.2. This results in a 6,200 µg/Kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100 µL aliquot of this extract to 5 mL of organic-free reagent water for purging (as per Section 7.5.3.2.6).

7.6 Data interpretation

7.6.1 Qualitative analysis

7.6.1.1 An analyte (e.g. those listed in Table 1) is identified by comparison of the sample mass spectrum with the mass

spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

7.6.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.6.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100% must be present in the sample spectrum). (2) The relative intensities of ions specified in (1) must agree within ± 20% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.

7.6.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting

peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

7.6.2 Quantitative analysis

7.6.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (e.g. see Table 6).

7.6.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste:

$$\text{concentration } (\mu g/L) = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured (Section 7.3.6).
V_o = Volume of water purged (mL), taking into consideration any dilutions made.

Sediment/Soil, Sludge, and Waste:

High-concentration:

$$\text{concentration } (\mu g/Kg) = \frac{(A_x)(I_s)\ (V_t)}{(A_{is})(RF)(V_i)(W_s)}$$

Low-concentration:

$$\text{concentration } (\mu g/Kg) = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)}$$

where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste above.
V_t = Volume of total extract (μL) (use 10,000 μL or a factor of this when dilutions are made).
V_i = Volume of extract added (μL) for purging.
W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

7.6.2.3 Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The percent dry weight of the sample (as calculated in Section 7.5.3.1.5) should be reported along with the data in either instance.

7.6.2.4 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulae given above should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control procedures.

8.2 Required instrument QC is found in the following sections:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Section 7.3.1.

8.2.2 There must be an initial calibration of the GC/MS system as specified in Section 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria specified in Section 7.4.3 and the CCC criteria in Section 7.4.4, each 12 hours.

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

8.3.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 10 mg/L in methanol. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate must be made using stock standards prepared independently from those used for calibration.

8.3.2 Prepare a QC reference sample to contain 20 µg/L of each analyte by adding 200 µL of QC reference sample concentrate to 100 mL of organic-free reagent water.

8.3.3 Four 5 mL aliquots of the well mixed QC reference sample are analyzed according to the method beginning in Section 7.5.1.

8.3.4 Calculate the average recovery ($\bar{x}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each analyte using the four results.

8.3.5 Tables 7 and 8 provide single laboratory recovery and precision data obtained for the method analytes from water. Similar results from dosed water should be expected by any experienced laboratory. Compare s and $\bar{x}$ (Section 8.3.4) for each analyte to the single laboratory recovery and precision data. Results are comparable if the calculated standard deviation of the recovery does not exceed 2.6 times the single laboratory RSD or 20%, whichever is greater, and the mean recovery lies within the interval $\bar{x} \pm 3S$ or $\bar{x} \pm 30\%$, whichever is greater.

NOTE: The large number of analytes in Tables 7 and 8 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

8.3.6 When one or more of the analytes tested are not comparable to the data in Table 7 or 8, the analyst must proceed according to Section 8.3.6.1 or 8.3.6.2.

8.3.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Section 8.3.2.

8.3.6.2 Beginning with Section 8.3.2, repeat the test only for those analytes that are not comparable. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.3.2.

8.4 For aqueous and soil matrices, laboratory established surrogate control limits should be compared with the control limits listed in Table 9.

8.4.1 If recovery is not within limits, the following procedures are required.

8.4.1.1 Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

8.4.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

8.4.1.3 If no problem is found, re-extract and re-analyze the sample.

8.4.1.4 If, upon re-analysis, the recovery is again not within limits, flag the data as "estimated concentration".

8.4.2 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

9.0 METHOD PERFORMANCE

9.1 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. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 7. Calculated MDLs are presented in Table 1.

9.3 The method was tested using water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 8. MDL values were also calculated from these data and are presented in Table 2.

10.0 REFERENCES

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TABLE 1.
CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)
FOR VOLATILE ORGANIC COMPOUNDS ON WIDE BORE CAPILLARY COLUMNS

ANALYTE	RETENTION TIME (minutes)			MDL[d] (µg/L)
	Column 1[a]	Column 2[b]	Column 2'[c]	
Dichlorodifluoromethane	1.55	0.70	3.13	0.10
Chloromethane	1.63	0.73	3.40	0.13
Vinyl Chloride	1.71	0.79	3.93	0.17
Bromomethane	2.01	0.96	4.80	0.11
Chloroethane	2.09	1.02	--	0.10
Trichlorofluoromethane	2.27	1.19	6.20	0.08
1,1-Dichloroethene	2.89	1.57	7.83	0.12
Methylene chloride	3.60	2.06	9.27	0.03
trans-1,2-Dichloroethene	3.98	2.36	9.90	0.06
1,1-Dichloroethane	4.85	2.93	10.80	0.04
2,2-Dichloropropane	6.01	3.80	11.87	0.35
cis-1,2-Dichloroethene	6.19	3.90	11.93	0.12
Chloroform	6.40	4.80	12.60	0.03
Bromochloromethane	6.74	4.38	12.37	0.04
1,1,1-Trichloroethane	7.27	4.84	12.83	0.08
Carbon tetrachloride	7.61	5.26	13.17	0.21
1,1-Dichloropropene	7.68	5.29	13.10	0.10
Benzene	8.23	5.67	13.50	0.04
1,2-Dichloroethane	8.40	5.83	13.63	0.06
Trichloroethene	9.59	7.27	14.80	0.19
1,2-Dichloropropane	10.09	7.66	15.20	0.04
Bromodichloromethane	10.59	8.49	15.80	0.08
Dibromomethane	10.65	7.93	15.43	0.24
trans-1,3-Dichloropropene	--	--	16.70	--
Toluene	12.43	10.00	17.40	0.11
cis-1,3-Dichloropropene	--	--	17.90	--
1,1,2-Trichloroethane	13.41	11.05	18.30	0.10
Tetrachloroethene	13.74	11.15	18.60	0.14
1,3-Dichloropropane	14.04	11.31	18.70	0.04
Dibromochloromethane	14.39	11.85	19.20	0.05
1,2-Dibromoethane	14.73	11.83	19.40	0.06
1-Chlorohexane	15.46	13.29	--	0.05
Chlorobenzene	15.76	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	15.94	13.33	20.87	0.05
Ethylbenzene	15.99	13.39	21.00	0.06
p-Xylene	16.12	13.69	21.30	0.13
m-Xylene	16.17	13.68	21.37	0.05
o-Xylene	17.11	14.52	22.27	0.11
Styrene	17.31	14.60	22.40	0.04
Bromoform	17.93	14.88	22.77	0.12
Isopropylbenzene	18.06	15.46	23.30	0.15
1,1,2,2-Tetrachloroethane	18.72	16.35	24.07	0.04

TABLE 1.
(Continued)

ANALYTE	RETENTION TIME (minutes)			MDL[d] (µg/L)
	Column 1[a]	Column 2[b]	Column 2'[c]	
Bromobenzene	18.95	15.86	24.00	0.03
1,2,3-Trichloropropane	19.02	16.23	24.13	0.32
n-Propylbenzene	19.06	16.41	24.33	0.04
2-Chlorotoluene	19.34	16.42	24.53	0.04
1,3,5-Trimethylbenzene	19.47	16.90	24.83	0.05
4-Chlorotoluene	19.50	16.72	24.77	0.06
tert-Butylbenzene	20.28	17.57	26.60	0.14
1,2,4-Trimethylbenzene	20.34	17.70	31.50	0.13
sec-Butylbenzene	20.79	18.09	26.13	0.13
p-Isopropyltoluene	21.20	18.52	26.50	0.12
1,3-Dichlorobenzene	21.22	18.14	26.37	0.12
1,4-Dichlorobenzene	21.55	18.39	26.60	0.03
n-Butylbenzene	22.22	19.49	27.32	0.11
1,2-Dichlorobenzene	22.52	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	24.53	21.08	--	0.26
1,2,4-Trichlorobenzene	26.55	23.08	31.50	0.04
Hexachlorobutadiene	26.99	23.68	32.07	0.11
Naphthalene	27.17	23.52	32.20	0.04
1,2,3-Trichlorobenzene	27.78	24.18	32.97	0.03
INTERNAL STANDARDS/SURROGATES				
4-Bromofluorobenzene	18.63	15.71	23.63	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 5 minutes, then program to 160°C at 6°/min.

[b] Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°/min.

[c] Column 2' - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10°/min, program to 120°C at 5°/min, then program to 180°C at 8°/min.

[d] MDL based on a 25 mL sample volume.

TABLE 2.
CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)
FOR VOLATILE ORGANIC COMPOUNDS ON NARROW BORE CAPILLARY COLUMNS

ANALYTE	RETENTION TIME (minutes) Column 3[a]	MDL[b] (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.06
Chloroethane	1.45	0.02
Trichlorofluoromethane	1.77	0.07
1,1-Dichloroethene	2.33	0.05
Methylene chloride	2.66	0.09
trans-1,2-Dichloroethene	3.54	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10

TABLE 2.
(Continued)

ANALYTE	RETENTION TIME (minutes) Column 3[a]	MDL[b] (µg/L)
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

[a] Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

[b] MDL based on a 25 mL sample volume.

TABLE 3.
ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

	Estimated Quantitation Limits		
	Ground water µg/L		Low Soil/Sediment[b] µg/Kg
Volume of water purged	5 mL	25 mL	
All analytes in Table 1	5	1	5

[a] Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent EQLs.

[b] EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor[c]
Water miscible liquid waste	50
High-concentration soil and sludge	125
Non-water miscible waste	500

[c]EQL = [EQL for low soil sediment (Table 3)] X [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4.
BFB MASS - INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

TABLE 5.
CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223, 227
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methylene chloride	84	86, 49
Naphthalene	128	-
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119

TABLE 5.
(Continued)

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,1,1,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	113	
Toluene-d_8	98	
Pentafluorobenzene	168	
1,4-Difluorobenzene	114	
Chlorobenzene-d_5	117	
1,4-Dichlorobenzene-d_4	152	

TABLE 6.
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES
ASSIGNED FOR QUANTITATION

Pentafluorobenzene	1,4-Difluorobenzene
Acetone	Benzene
Acrolein	Bromodichloromethane
Acrylonitrile	Bromofluorobenzene (surrogate)
Bromochloromethane	Carbon tetrachloride
Bromomethane	2-Chloroethyl vinyl ether
2-Butanone	1,2-Dibromoethane
Carbon disulfide	Dibromomethane
Chloroethane	1,2-Dichloroethane
Chloroform	1,2-Dichloroethane-d_4 (surrogate)
Chloromethane	1,2-Dichloropropane
Dichlorodifluoromethane	1,1-Dichloropropene
1,1-Dichloroethane	cis-1,3-Dichloropropene
1,1-Dichloroethene	trans-1,3-Dichloropropene
cis-1,2-Dichloroethene	4-Methyl-2-pentanone
trans-1,2-Dichloroethene	Toluene
2,2-Dichloropropane	Toluene-d_8 (surrogate)
Iodomethane	1,1,2-Trichloroethane
Methylene chloride	Trichloroethene
1,1,1-Trichloroethane	
Trichlorofluoromethane	1,4-Dichlorobenzene-d_4
Vinyl acetate	
Vinyl chloride	Bromobenzene
	n-Butylbenzene
Chlorobenzene-d_5	sec-Butylbenzene
	tert-Butylbenzene
Bromoform	2-Chlorotoluene
Chlorodibromomethane	4-Chlorotoluene
Chlorobenzene	1,2-Dibromo-3-chloropropane
1,3-Dichloropropane	1,2-Dichlorobenzene
Ethylbenzene	1,3-Dichlorobenzene
2-Hexanone	1,4-Dichlorobenzene
Styrene	Hexachlorobutadiene
1,1,1,2-Tetrachloroethane	Isopropyl benzene
Tetrachloroethene	p-Isopropyltoluene
Xylene	Naphthalene
	n-Propylbenzene
	1,1,2,2-Tetrachloroethane
	1,2,3-Trichlorobenzene
	1,2,4-Trichlorobenzene
	1,2,3-Trichloropropane
	1,2,4-Trimethylbenzene
	1,3,5-Trimethylbenzene

TABLE 7.
SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE BORE CAPILLARY COLUMN

Analyte	Conc. Range, µg/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery[b]	Percent Rel.Std Dev.
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8

TABLE 7.
(Continued)

Analyte	Conc. Range, µg/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery[b]	Percent Rel. Std. Dev.
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

[b] Standard deviation was calculated by pooling data form three concentrations.

TABLE 8.
SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR
VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW BORE CAPILLARY COLUMN

Analyte	Conc. µg/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery	Percent Rel. Std. Dev.
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.7	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3
n-Propylbenzene	0.5	7	99	6.6	6.7

TABLE 8.
(Continued)

Analyte	Conc. µg/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery	Percent Rel. Std. Dev.
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 9.
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
4-Bromofluorobenzene[a]	86-115	74-121
Dibromofluoromethane[a]	86-118	80-120
Toluene-d_8[a]	88-110	81-117

[a] Single laboratory data for guidance only.

TABLE 10.

QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATION SAMPLES

Approximate Concentration Range	Volume of Extract[a]
500 - 10,000 µg/Kg	100 µL
1,000 - 20,000 µg/Kg	50 µL
5,000 - 100,000 µg/Kg	10 µL
25,000 - 500,000 µg/Kg	100 µL of 1/50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

[b] Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

FIGURE 1.
PURGING DEVICE

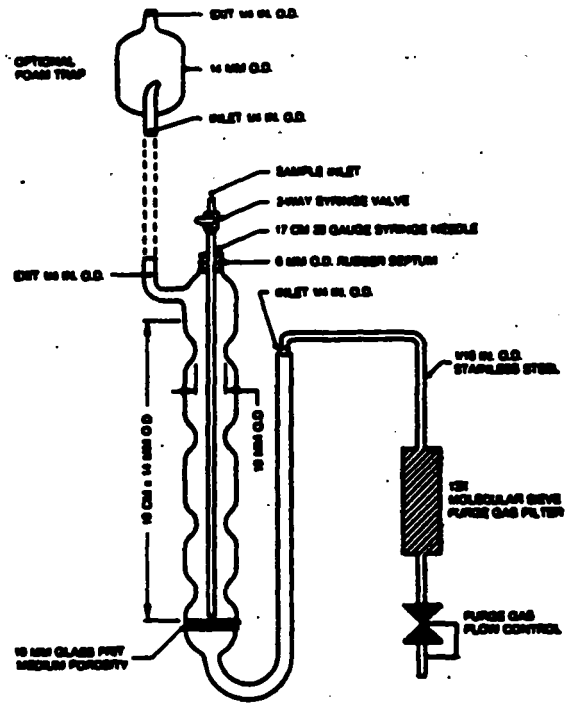


FIGURE 2.
TRAP PACKING AND CONSTRUCTION TO INCLUDE DESORB CAPABILITY

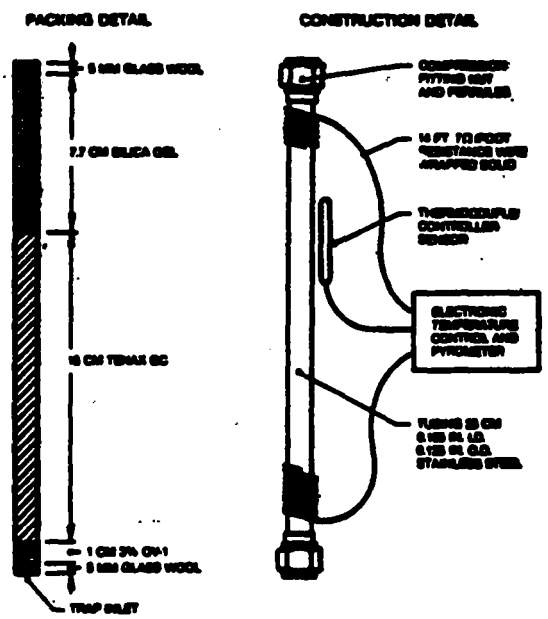


FIGURE 3.
SCHEMATIC OF PURGE-AND-TRAP DEVICE - PURGE MODE

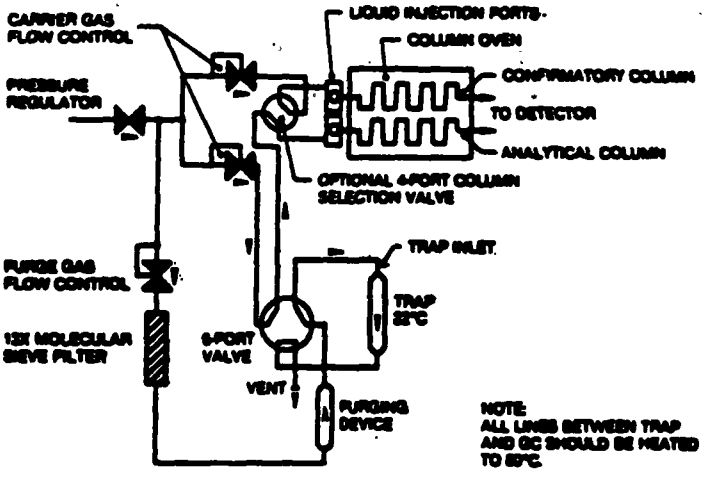


FIGURE 4.
SCHEMATIC OF PURGE-AND-TRAP DEVICE - DESORB MODE

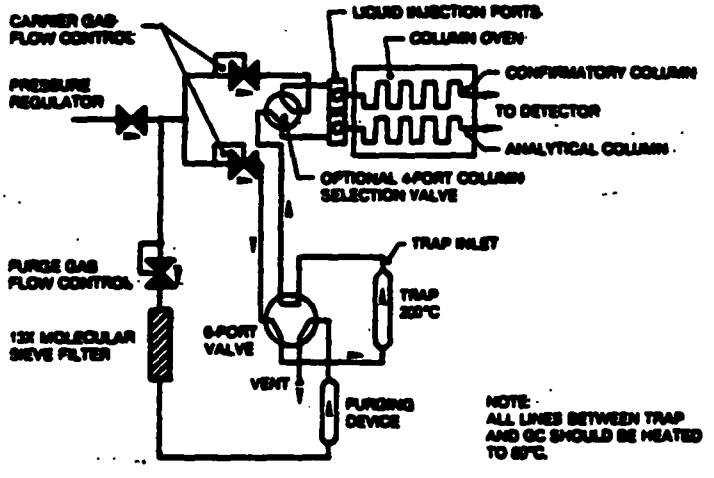


FIGURE 5.
GAS CHROMATOGRAM OF VOLATILE ORGANICS

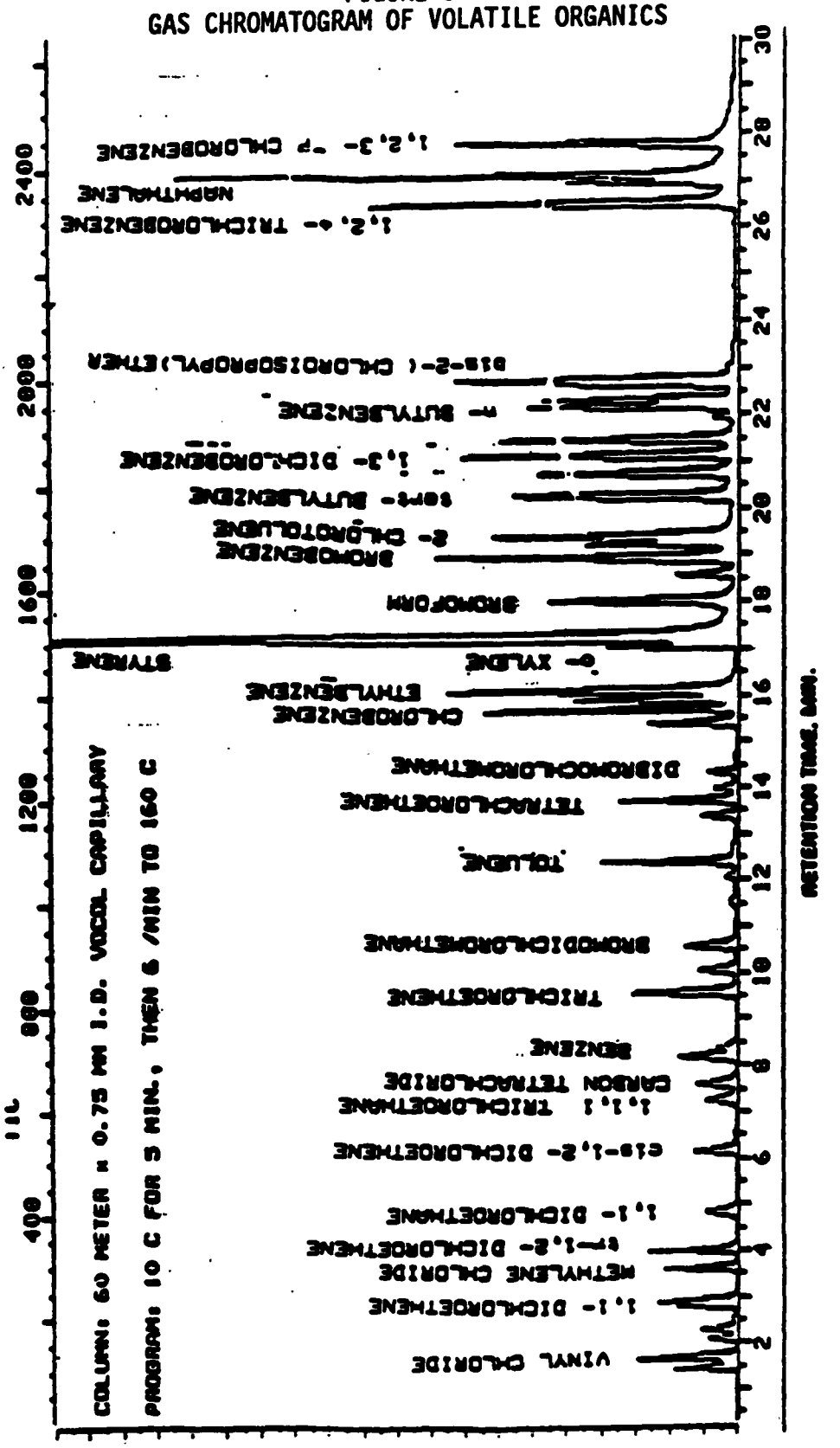


FIGURE 6.
GAS CHROMATOGRAM OF VOLATILE ORGANICS

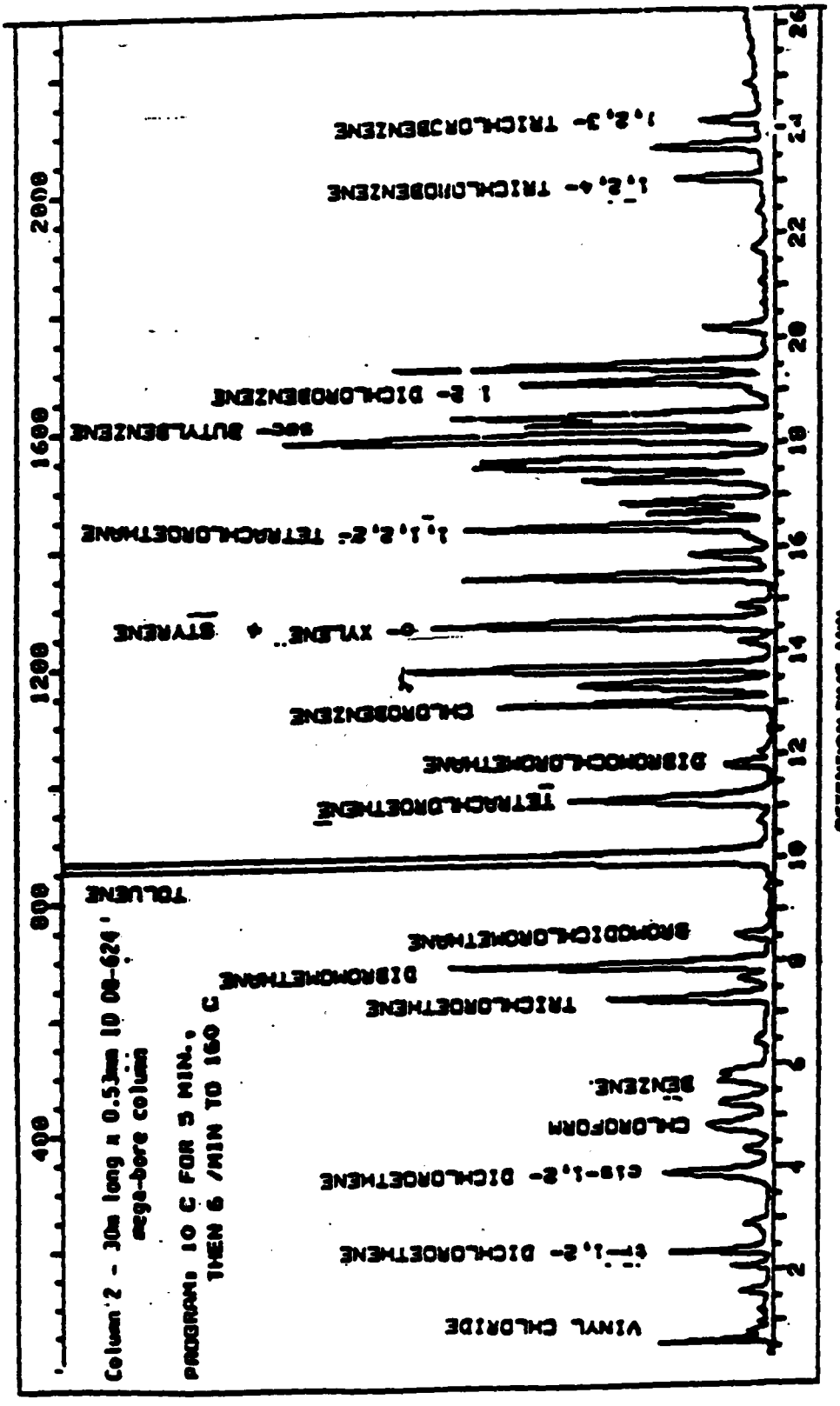


FIGURE 7.
GAS CHROMATOGRAM OF VOLATILE ORGANICS

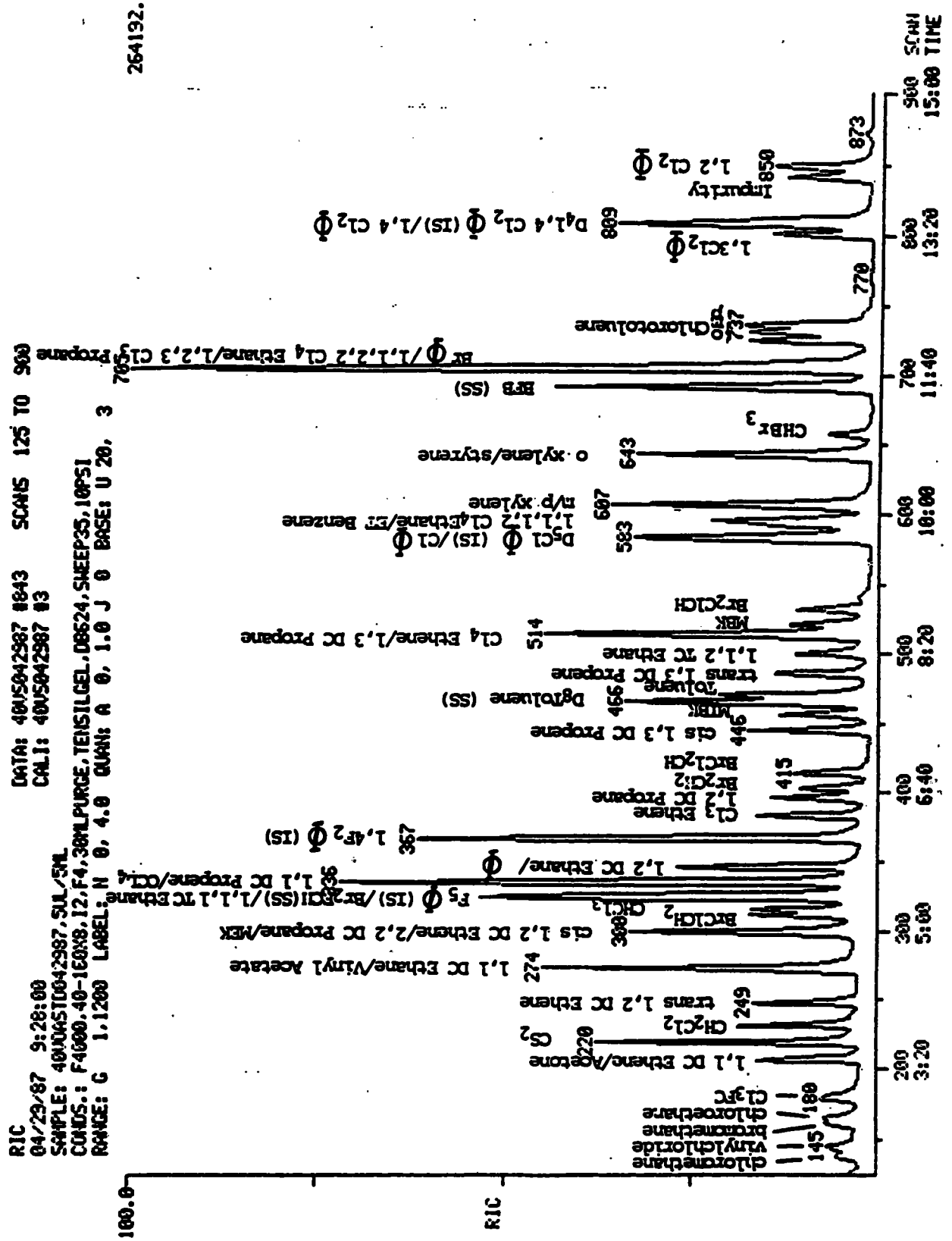


FIGURE 8.
GAS CHROMATOGRAM OF TEST MIXTURE

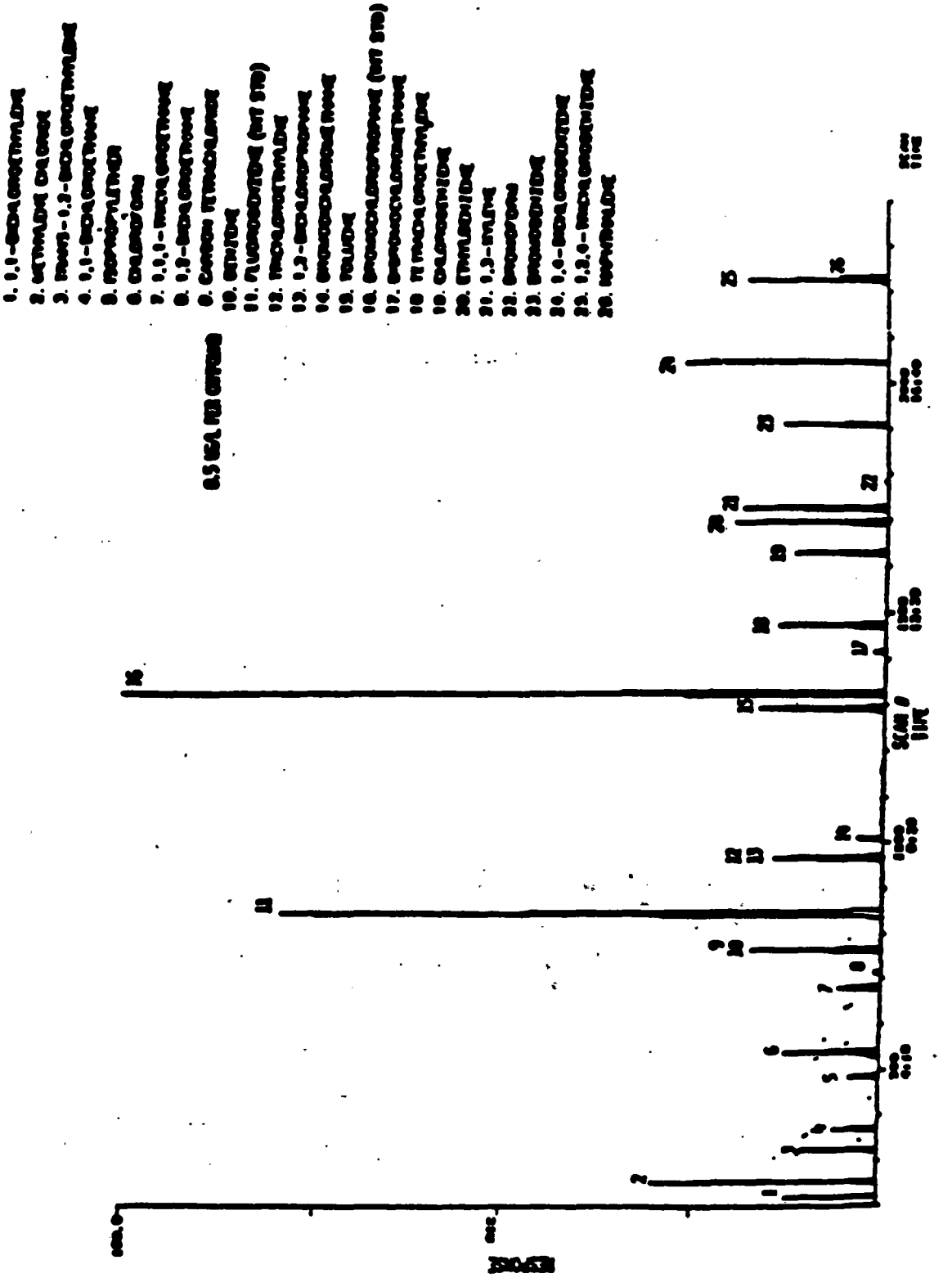
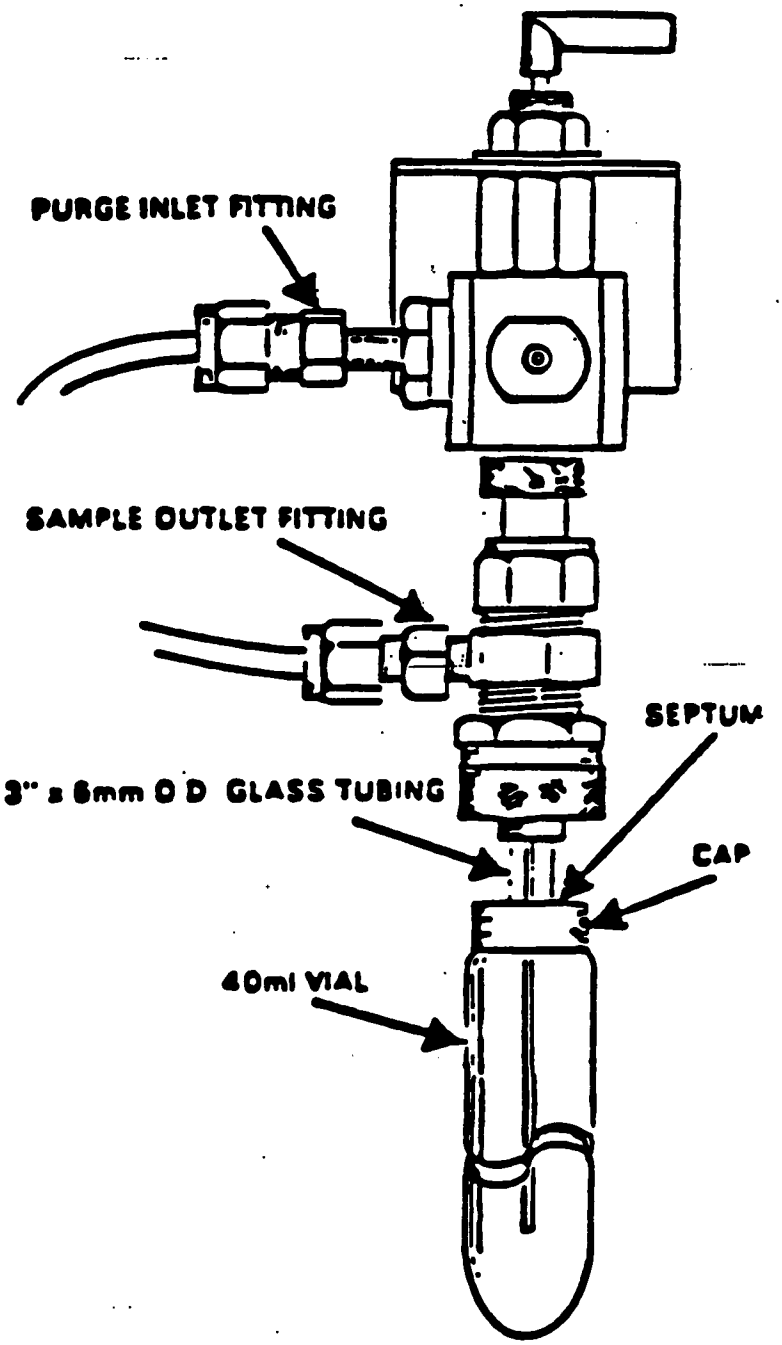
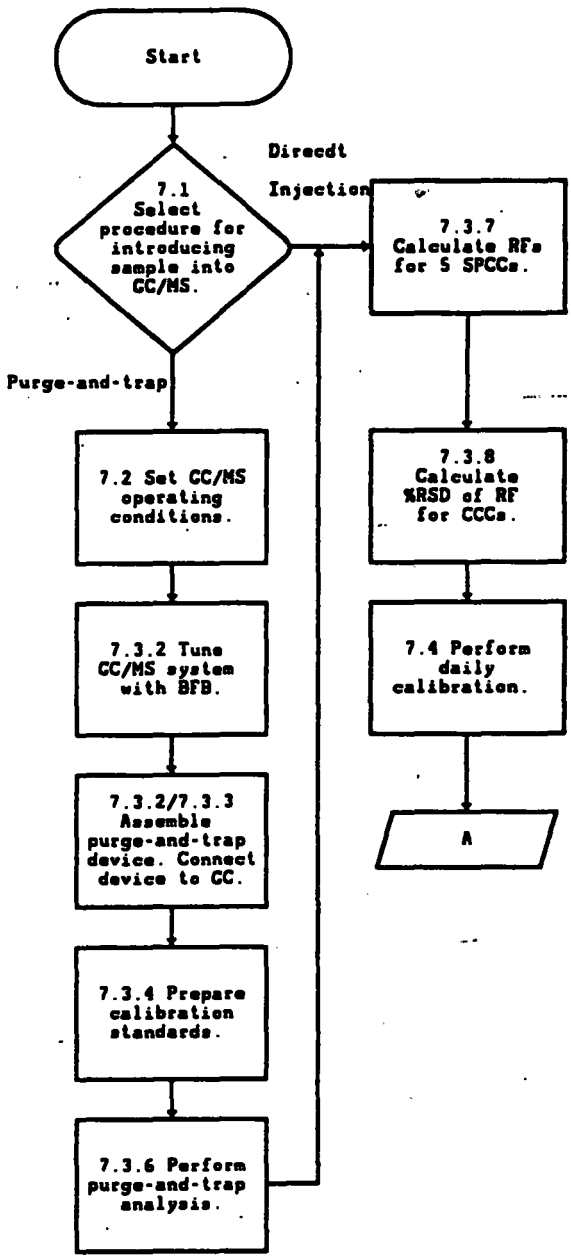
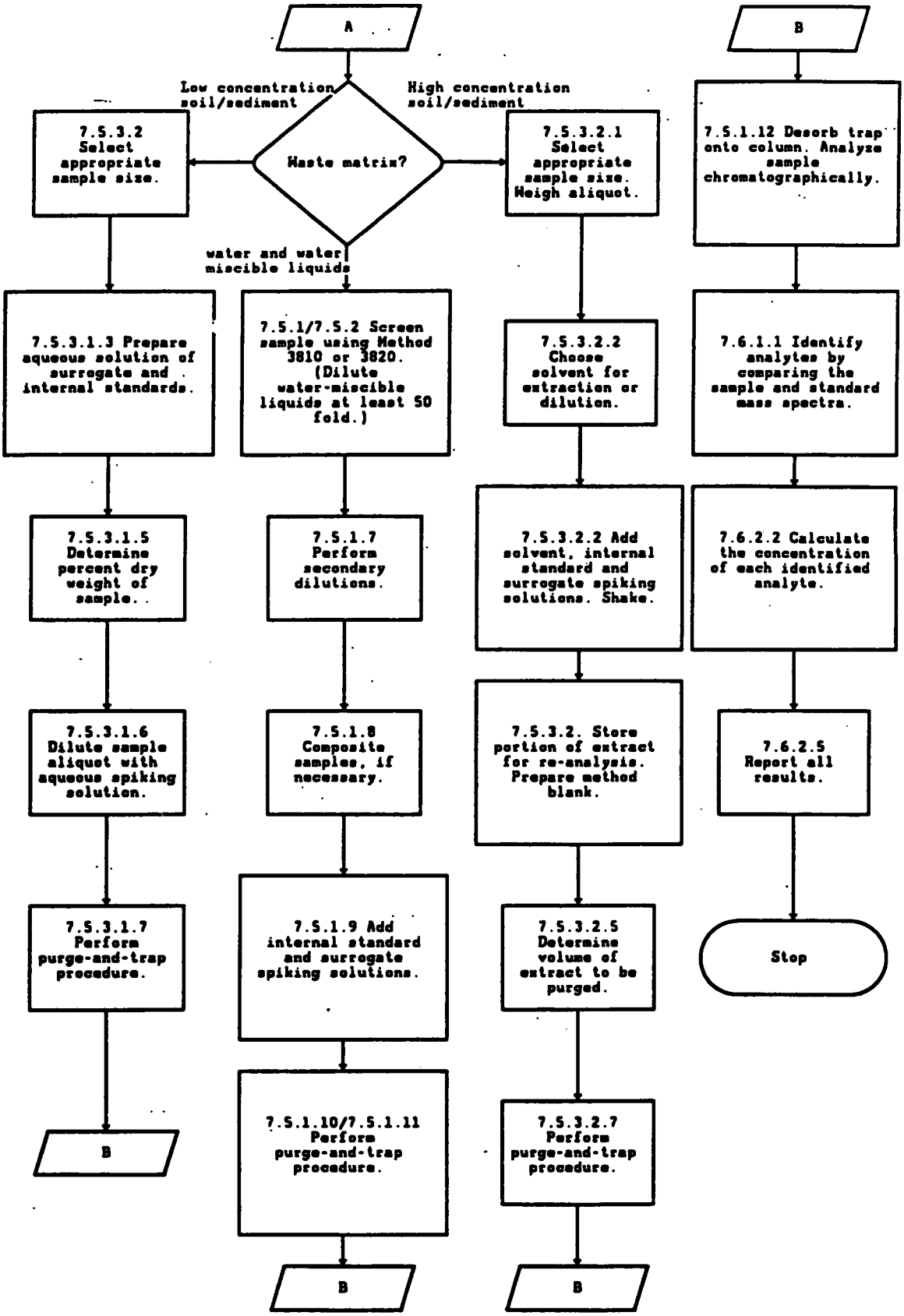


FIGURE 9.
LOW SOILS IMPINGER




Start
7.1 Select procedure for introducing sample into GC/MS.
Direcdt Injection
Purge-and-trap
7.3.7 Calculate RFs for 5 SPCCs.
7.2 Set GC/MS operating conditions.
7.3.8 Calculate %RSD of RF for CCCs.
7.3.2 Tune GC/MS system with BFB.
7.4 Perform daily calibration.
7.3.2/7.3.3 Assemble purge-and-trap device. Connect device to GC.
A
7.3.4 Prepare calibration standards.
7.3.6 Perform purge-and-trap analysis.


A
Low concentration soil/sediment
High concentration soil/sediment
Waste matrix?
7.5.3.2 Select appropriate sample size.
7.5.3.2.1 Select appropriate sample size. Weigh aliquot.
water and water miscible liquids
B
7.5.1.12 Desorb trap onto column. Analyze sample chromatographically.
7.5.3.1.3 Prepare aqueous solution of surrogate and internal standards.
7.5.1/7.5.2 Screen sample using Method 3810 or 3820. (Dilute water-miscible liquids at least 50 fold.)
7.5.3.2.2 Choose solvent for extraction or dilution.
7.6.1.1 Identify analytes by comparing the sample and standard mass spectra.
7.5.3.1.5 Determine percent dry weight of sample.
7.5.1.7 Perform secondary dilutions.
7.5.3.2.2 Add solvent, internal standard and surrogate spiking solutions. Shake.
7.6.2.2 Calculate the concentration of each identified analyte.
7.5.3.1.6 Dilute sample aliquot with aqueous spiking solution.
7.5.1.8 Composite samples, if necessary.
7.5.3.2. Store portion of extract for re-analysis. Prepare method blank.
7.6.2.5 Report all results.
7.5.3.1.7 Perform purge-and-trap procedure.
7.5.1.9 Add internal standard and surrogate spiking solutions.
7.5.3.2.5 Determine volume of extract to be purged.
Stop
B
7.5.1.10/7.5.1.11 Perform purge-and-trap procedure.
7.5.3.2.7 Perform purge-and-trap procedure.
B
B

METHOD 5030

PURGE-AND-TRAP

1.0 SCOPE AND APPLICATION

1.1 This method describes sample preparation and extraction for the analysis of volatile organics by a purge-and-trap procedure. The gas chromatographic determinative steps are found in Methods 8010, 8015, 8020, and 8030. Although applicable to Method 8240, the purge-and-trap procedure is already incorporated into Method 8240.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C (vapor pressure is approximately equal to mm Hg @ 25°C) and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system.

1.4 This method also describes the preparation of water-miscible liquids, solids, wastes, and soil/sediments for analysis by the purge-and-trap procedure.

2.0 SUMMARY OF METHOD

2.1 The purge-and-trap process: An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC following the normal water method.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from 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. The use of non-TFE plastic coating, 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 and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A field reagent blank prepared from reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-level and low-level samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes: 10-uL, 25-uL, 100-uL, 250-uL, 500-uL, and 1,000 uL: These syringes should be equipped with a 20-gauge (0.006-in I.D.) needle having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device. The needle length will depend upon the dimensions of the purging device employed.

4.2 Syringe valve: Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Syringe: 5-mL, gas-tight with shutoff valve.

4.4 Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g.

4.5 Glass scintillation vials: 20-mL, with screw-caps and Teflon liners or glass culture tubes with a screw-cap and Teflon liner.

4.6 Volumetric flasks: 10-mL and 100-mL, class A with ground-glass stoppers.

4.7 Vials: 2-mL, for GC autosampler.

4.8 Spatula: Stainless steel.

4.9 Disposable pipets: Pasteur.

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

4.10.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace 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. Alternate sample purge devices may be used, provided equivalent performance is demonstrated.

4.10.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figures 2 and 3). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.10.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figures 2 and 3 meet these criteria.

4.10.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as shown in Figures 4 and 5.

4.10.5 Trap Packing Materials

4.10.5.1 2,6-Diphenylene oxide polymer: 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.10.5.2 Methyl silicone packing: OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

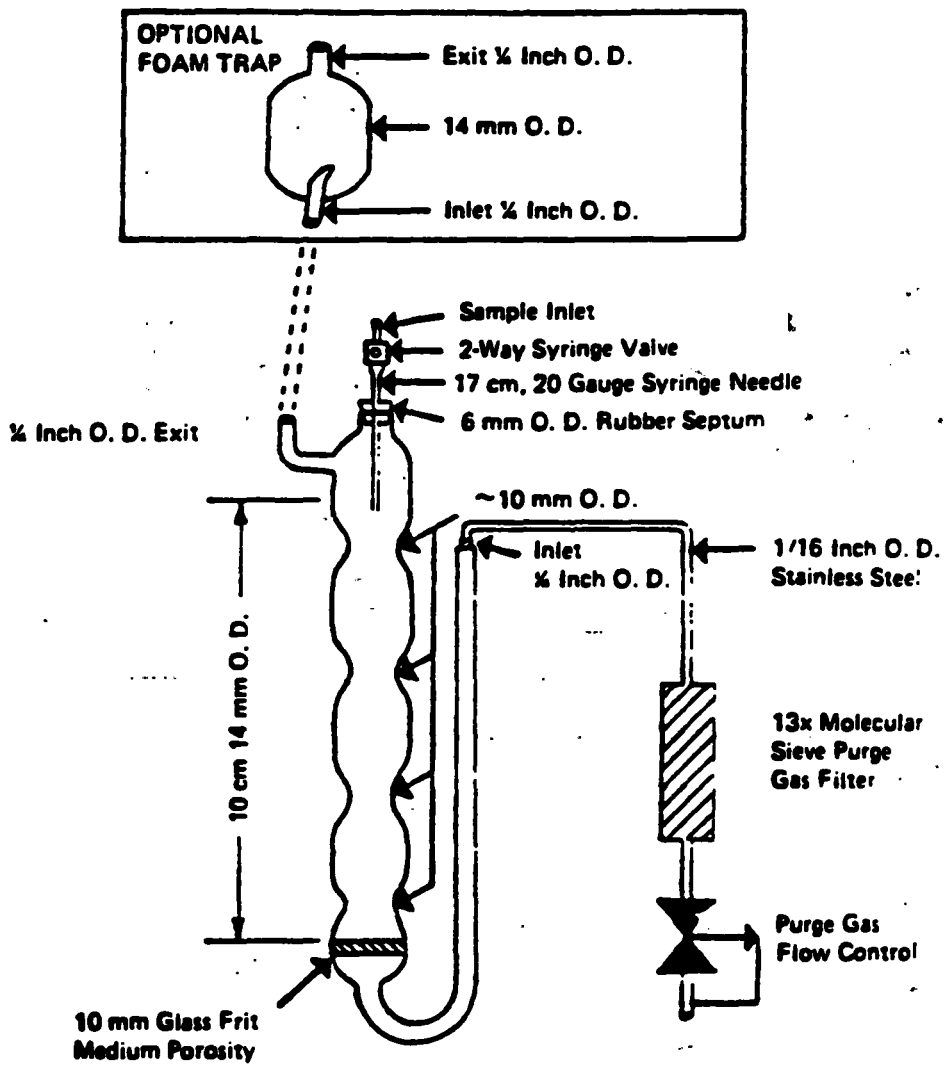


Figure 1. Purging chamber.

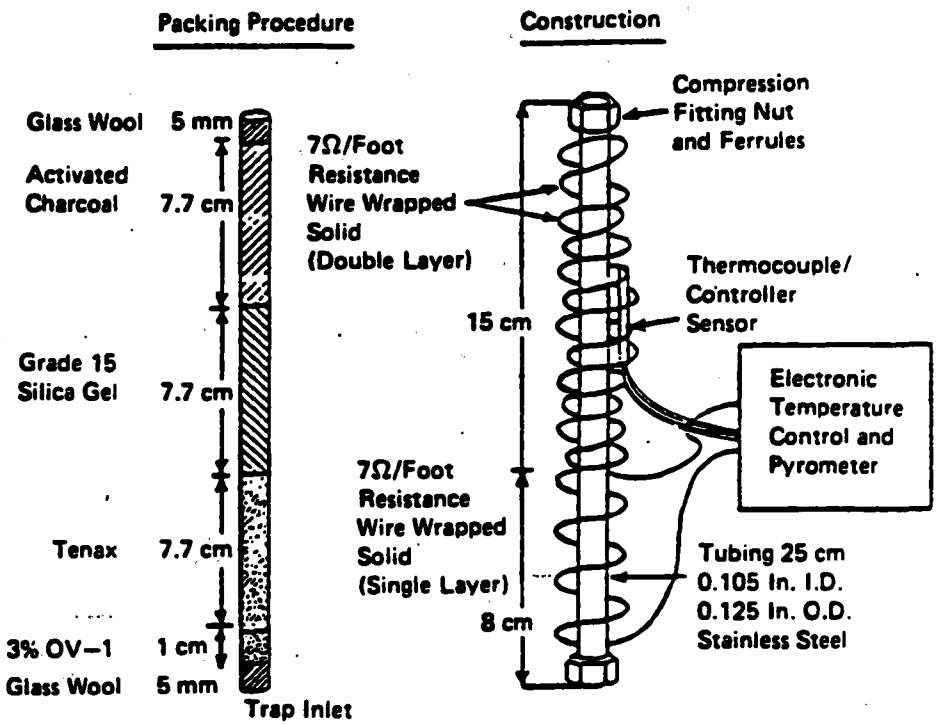


Figure 2. Trap packings and construction for Method 8010.

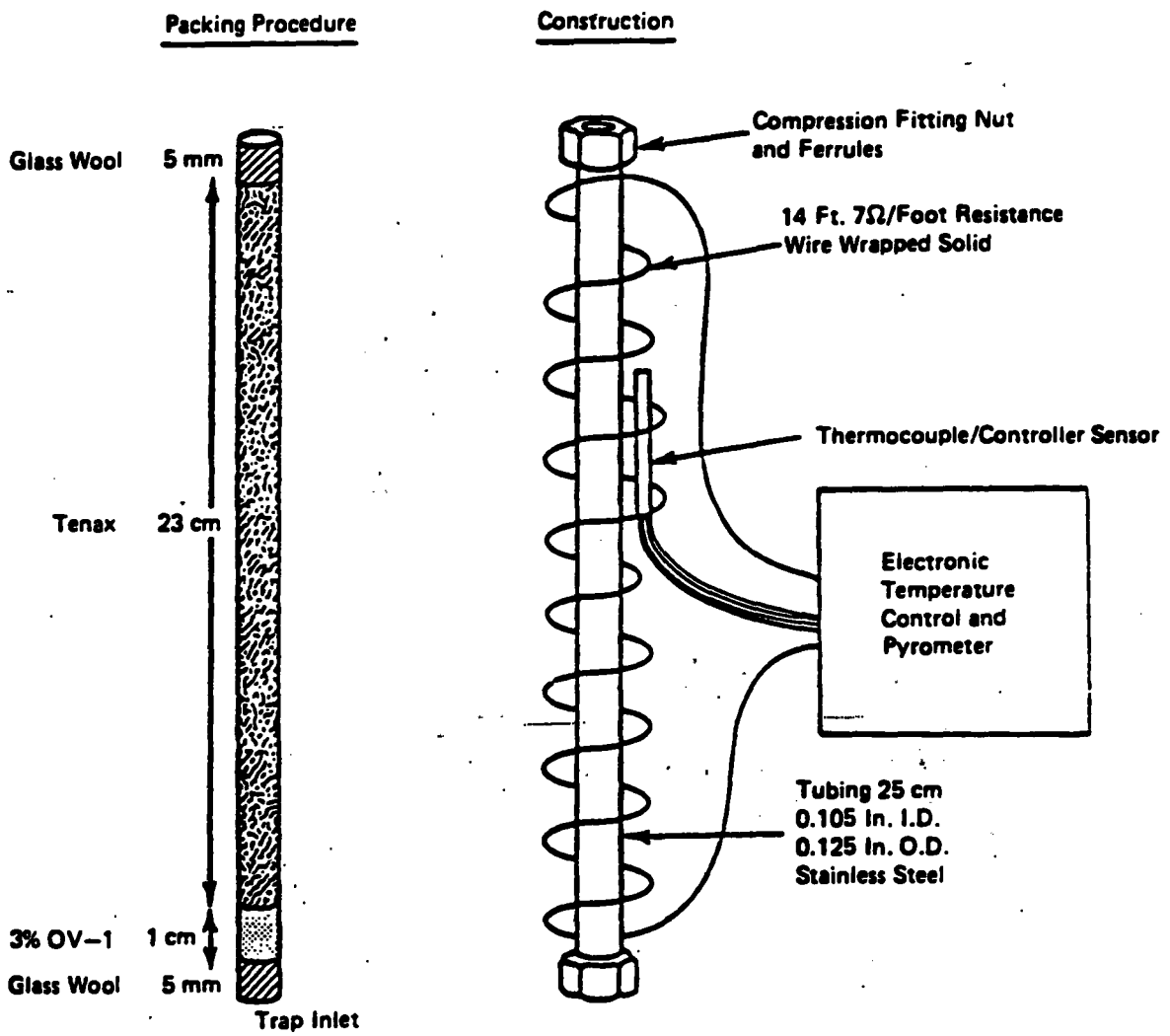


Figure 3. Trap packing and construction for Methods 8020 and 8030.

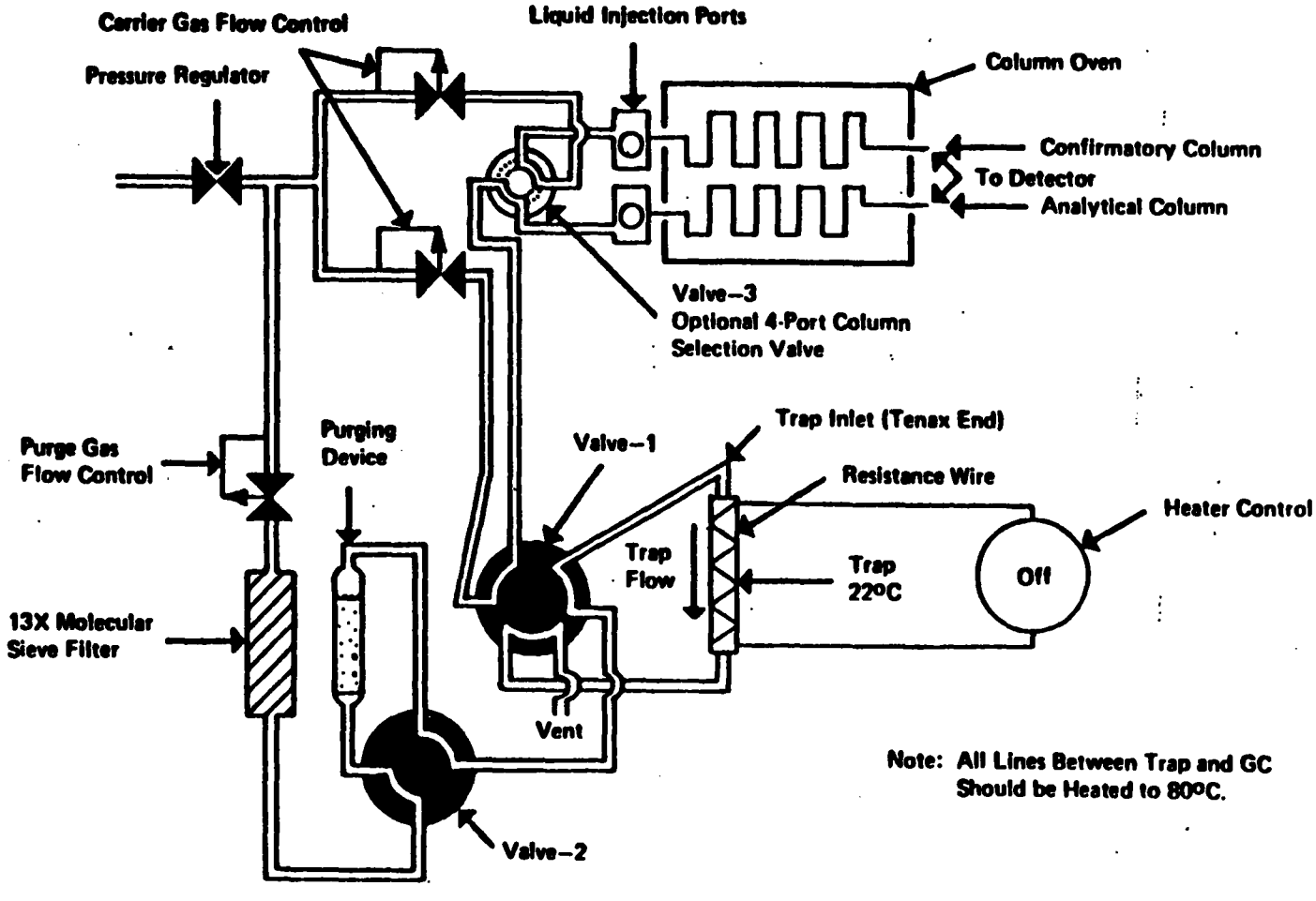


Figure 4. Purge-and-trap system, purge-sorb mode, for Methods 8010, 8020, and 8030.

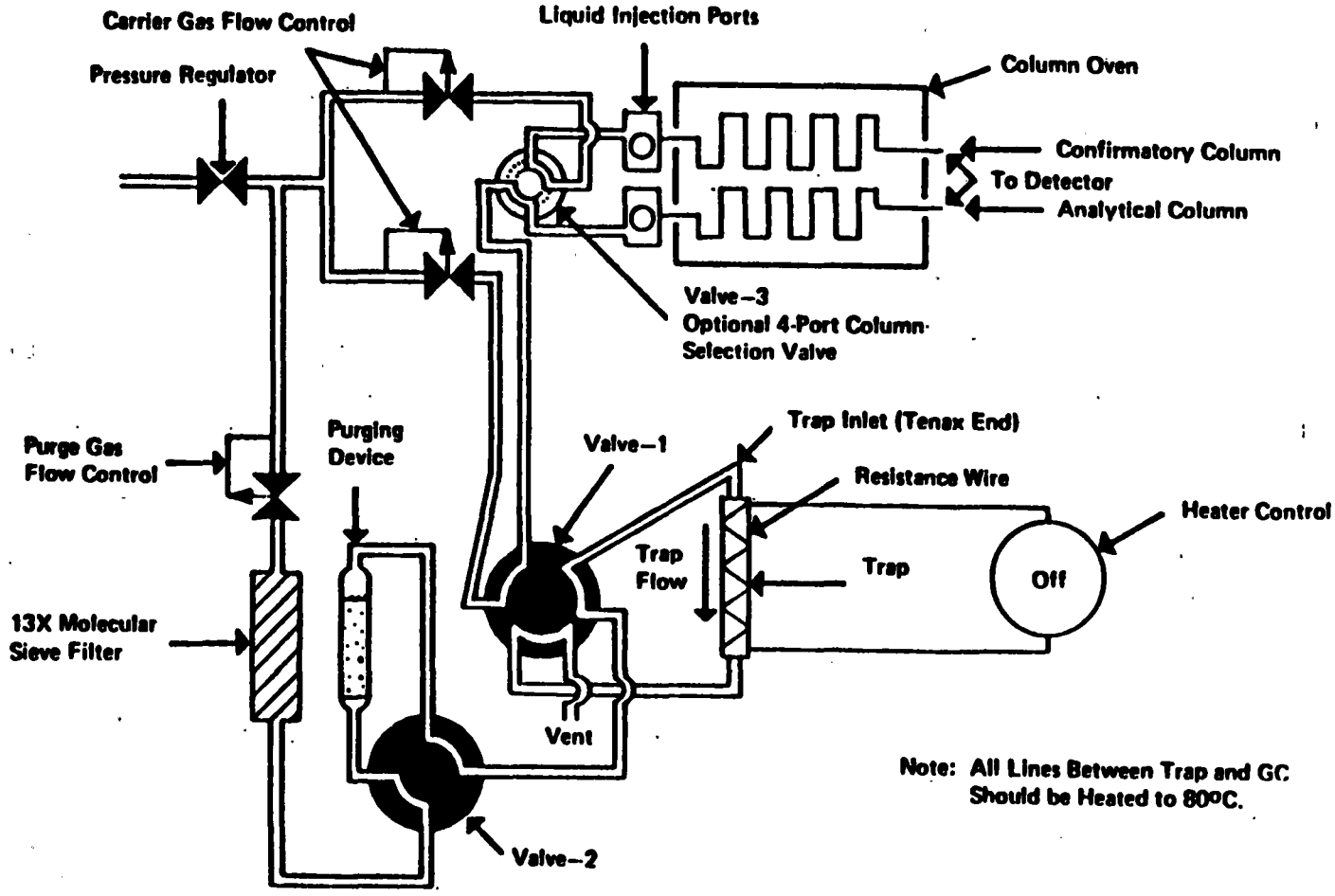


Figure 5. Purge-and-trap system, desorb mode, for Methods 8010, 8020, and 8030.

4.10.5.3 Silica gel: 35/60 mesh, Davison, grade 15 or equivalent.

4.10.5.4 Coconut charcoal: Prepare from Barnebey Cheney, CA-580-26 lot #M-2649, by crushing through 26 mesh screen.

4.11 Heater or heated oil bath: Should be capable of maintaining the purging chamber to within 1°C over a temperature range from ambient to 100°C.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as 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 trap 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 water 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.

5.2 Methanol: Pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Initial calibration: Prior to using this introduction technique for any GC method, the system must be calibrated. General calibration procedures are discussed in Method 8000, Section 7.4, while the specific determinative methods and Method 3500 give details on preparation of standards.

7.1.1 Assemble a purge-and-trap device that meets the specification in Section 4.10. Condition the trap overnight at 180°C in the purge mode 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 with the column at 220°C.

7.1.2 Connect the purge-and-trap device to a gas chromatograph.

7.1.3 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. Add 5.0 mL of reagent water to the purging device. The reagent water 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 the 20-gauge needle. Next, using a 10-uL or 25-uL microsyringe equipped with a long needle (Paragraph 4.1), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the microsyringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water. Similarly, add 10 uL of the internal standard solution. Close the 2-way syringe valve at the sample inlet.

7.1.4 Carry out the purge-and-trap analysis procedure using the specific conditions given in Table 1.

7.1.5 Calculate response factors or calibration factors for each analyte of interest using the procedure described in Method 8000, Section 7.4.

7.1.6 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. If the purge-and-trap procedure is used with Method 8010, the following five compounds are checked for a minimum average response factor: chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.1.6.1 Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.

7.1.6.2 Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.1.6.3 Tetrachloroethane and 1,1-dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2 On-going calibration: Refer to Method 8000, Sections 7.4.2.3 and 7.4.3.4 for details on continuing calibration.

TABLE 1. PURGE-AND-TRAP OPERATING PARAMETERS

	Analysis Method			
	8010	8015	8020	8030
Purge gas	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium
Purge gas flow rate (mL/min)	40	20	40	20
Purge time (min)	11.0 ± 0.1	15.0 ± 0.1	12.0 ± 0.1	15.0 ± 0.1
Purge temperature (°C)	Ambient	85 ± 2	Ambient	85 ± 2
Desorb temperature (°C)	180	180	180	180
Backflush inert gas flow (mL/min)	20-60	20-60	20-60	20-60
Desorb time (min)	4	1.5	4	1.5

7.3 Sample preparation:

7.3.1 Water samples:

7.3.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be utilized are: the use of an automated headspace sampler (modified Method 3810), interfaced to a gas chromatograph (GC), equipped with a photo ionization detector (PID), in series with an electrolytic conductivity detector (ECD); and extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC with a FID and/or an ECD.

7.3.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.3.1.3 Assemble the purge-and-trap device. The operating conditions for the GC are given in Section 7.0 of the specific determinative method to be employed.

7.3.1.4 Daily GC calibration criteria must be met (Method 8000, Section 7.4) before analyzing samples.

7.3.1.5 Adjust the purge gas flow rate (nitrogen or helium) to that shown in Table 1, on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

7.3.1.6 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, 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. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hr. Care must be taken to prevent air from leaking into the syringe.

7.3.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.3.1.7.1 Dilutions may be made in volumetric flasks (10-mL to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.3.1.7.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

7.3.1.7.3 Inject the proper aliquot of samples from the syringe prepared in Paragraph 7.3.1.5 into the flask. Aliquots of less than 1-mL are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions.

7.3.1.7.4 Fill a 5-mL syringe with the diluted sample as in Paragraph 7.3.1.5.

7.3.1.8 Add 10.0 uL of surrogate spiking solution (found in each determinative method, Section 5.0) and, if applicable, 10 uL of internal standard spiking solution through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. Matrix spiking solutions, if indicated, should be added (10 uL) to the sample at this time.

7.3.1.9 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.

7.3.1.10 Close both valves and purge the sample for the time and at the temperature specified in Table 1.

7.3.1.11 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with inert gas between 20 and 60 mL/min for the time specified in Table 1.

7.3.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5-mL flushes of reagent water (or methanol followed by reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

7.3.1.13 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 sec; 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 for Methods 8010 and 8020, and 210°C for Methods 8015 and 8030. Trap temperatures up to 220°C may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 min, 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.

7.3.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated response from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.3.1.15 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Method 8000 and the specific determinative method for details on calculating analyte response.

7.3.2 Water-miscible liquids:

7.3.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

7.3.2.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

7.3.2.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with reagent water by adding at least 20 uL, but not more than 100-uL of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

7.3.3 Sediment/soil and waste samples: It is highly recommended that all samples of this type be screened prior to the purge-and-trap GC analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. See Paragraph 7.3.1.1 for recommended screening techniques. Use the screening data to determine whether to use the low-level method (0.005-1 mg/kg) or the high-level method (>1 mg/kg).

7.3.3.1 Low-level method: This is designed for samples containing individual purgeable compounds of <1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on

purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples.

7.3.3.1.1 Use a 5-g sample if the expected concentration is <0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.

7.3.3.1.2 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature for Methods 8010 and 8020.

7.3.3.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution and internal standard solution to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together.) Matrix spiking solutions, if indicated, should be added (10 uL) to the sample at this time.

7.3.3.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Paragraph 7.3.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.3.3.1.5 In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination. Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

7.3.3.1.6 Add the spiked reagent water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

NOTE: Prior to the attachment of the purge device, steps 7.3.3.1.4 and 7.3.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.3.3.1.7 Heat the sample to 40°C ± 1°C (Methods 8010 and 8020) or to 85°C ± 2°C (Methods 8015 and 8030) and purge the sample for the time shown in Table 1.

7.3.3.1.8 Proceed with the analysis as outlined in Paragraphs 7.3.1.11-7.3.1.15. Use 5 mL of the same reagent water as in the reagent blank. If saturated peaks occurred or would occur if a 1-g sample were analyzed, the high-level method must be followed.

7.3.3.2 High-level method: The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to reagent water containing surrogate and, if applicable, internal and matrix spiking standards. This is purged at the temperatures indicated in Table 1. All samples with an expected concentration of >1.0 mg/kg should be analyzed by this method.

7.3.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 4 g (wet weight) of sample into a tared 20-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Paragraph 7.3.3.1.5. For waste that is soluble in methanol, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of methanol into the vial and mark the bottom of the meniscus. Discard this solvent.)

7.3.3.2.2 Quickly add 9.0 mL of methanol; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 min.

NOTE: Steps 7.3.3.2.1 and 7.3.3.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.3.2.3 Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of reagent methanol to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis.

7.3.3.2.4 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the addition of the methanol extract to reagent water.

7.3.3.2.5 Table 2 can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a high-level sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.3.3.2.6 Remove the plunger from a 5.0-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 uL of internal standard solution. Also add the volume of methanol extract determined in Paragraph 7.3.3.2.5 and a volume of methanol solvent to total 100 uL (excluding methanol in standards).

7.3.3.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

7.3.3.2.8 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 uL of methanol to simulate the sample conditions.

7.3.3.2.9 For a matrix spike in the high-level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution and 1.0 mL of matrix spike solution. Add a 100-uL aliquot of this extract to 5 mL of water for purging (as per Paragraph 7.3.3.2.6).

TABLE 2. QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-LEVEL SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000 ug/kg	100 uL
1,000-20,000 ug/kg	50 uL
5,000-100,000 ug/kg	10 uL
25,000-500,000 ug/kg	100 uL of 1/50 dilution [b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a]The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

[b]Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

7.4 Sample analysis:

7.4.1 The samples prepared by this method may be analyzed by Methods 8010, 8015, 8020, 8030, and 8240. Refer to these methods for appropriate analysis conditions.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3500 for sample preparation procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of a reagent 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.

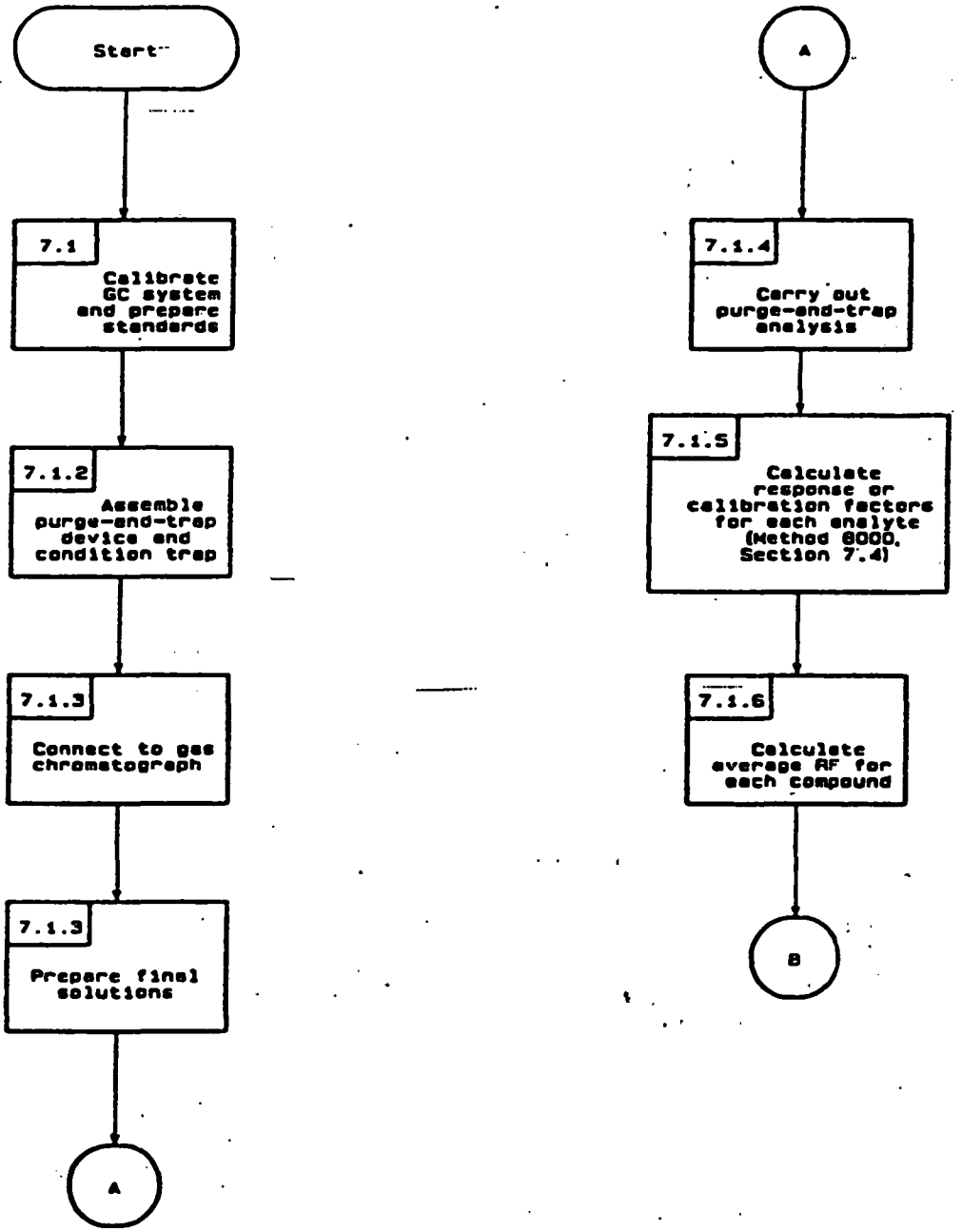
8.3 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages 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 <1 ug/g of the analytes in the sample, then the sensitivity of the instrument should be increased, or the sample should be subjected to additional cleanup.

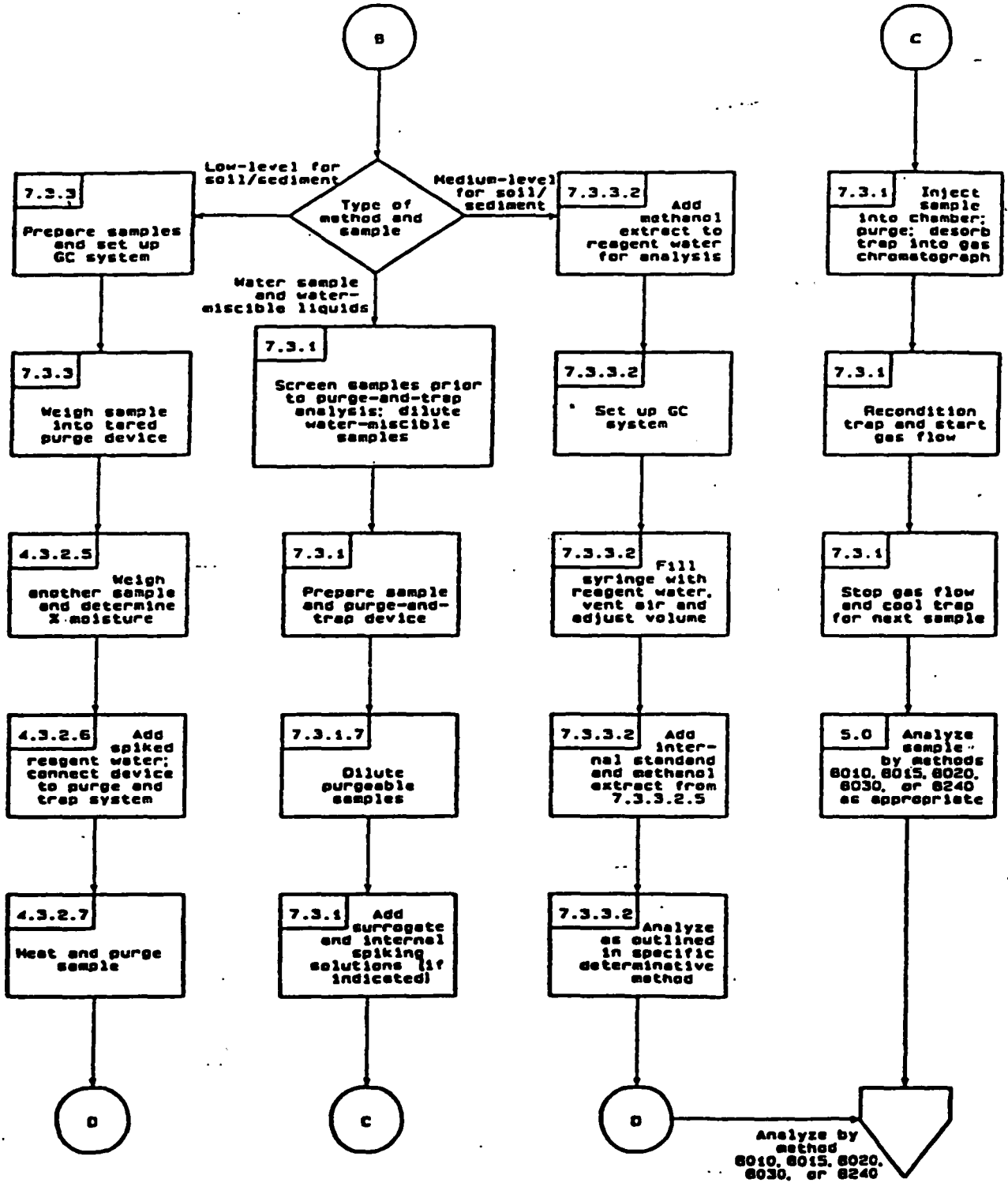
9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.


Start
7.1
Calibrate GC system and prepare standards
7.1.2
Assemble purge-and-trap device and condition trap
7.1.3
Connect to gas chromatograph
7.1.3
Prepare final solutions
A
A
7.1.4
Carry out purge-and-trap analysis
7.1.5
Calculate response or calibration factors for each analyte (Method 8000, Section 7.4)
7.1.6
Calculate average RF for each compound
B


B
C
Type of method and sample
Low-level for soil/sediment
Medium-level for soil/ sediment
Water sample and water-miscible liquids
7.3.3 Prepare samples and set up GC system
7.3.3.2 Add methanol extract to reagent water for analysis
7.3.1 Inject sample into chamber; purge; desorb trap into gas chromatograph
7.3.3 Weigh sample into tared purge device
7.3.1 Screen samples prior to purge-and-trap analysis; dilute water-miscible samples
7.3.3.2 Set up GC system
7.3.1 Recondition trap and start gas flow
4.3.2.5 Weigh another sample and determine % moisture
7.3.1 Prepare sample and purge-and-trap device
7.3.3.2 Fill syringe with reagent water, vent air and adjust volume
7.3.1 Stop gas flow and cool trap for next sample
4.3.2.6 Add spiked reagent water; connect device to purge and trap system
7.3.1.7 Dilute purgeable samples
7.3.3.2 Add internal standard and methanol extract from 7.3.3.2.5
5.0 Analyze sample by methods 8010, 8015, 8020, 8030, or 8240 as appropriate
4.3.2.7 Heat and purge sample
7.3.1 Add surrogate and internal spiking solutions (if indicated)
7.3.3.2 Analyze as outlined in specific determinative method
D
C
D
Analyze by method 8010, 8015, 8020, 8030, or 8240

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of monitoring well, surface water and sediment samples for select metals with detection limits lower than those provided by the ILM01.0 SOW.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen monitoring well samples will be collected during each sampling event. The aqueous samples will be analyzed for medium to low concentrations of filterable metals. The projected number is inclusive of QA samples.

Two surface water and two sediment samples will be collected and analyzed annually for select, low concentration metals. The surface waters will be field filtered.

3. Purpose of analysis (specify whether Superfund (Remedial or

Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

The following metals, with the listed detection limits, will be analyzed by graphite furnace atomic absorption (EPA SW-846 7000 series methods);

Analyte	Aqueous MDLs (µg/L)	Sediment MDLs (µg/Kg)
Arsenic	5	500
Barium	100	10000
Lead	2	200
Iron	50	5000
Manganese	5	500

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

One liter of the aqueous sample will be collected and preserved with 5 ml of HNO_3 to a pH <2. Samples should be stored at 4°C until the time of analysis.

100 grams of sediment sample will be collected and stored at 4°C until the time of analysis. Sediment samples will be prepared in accordance with EPA SW-846 Method 3050.

Any sample remaining after digestion should be stored at 4°C until the validation and the acceptance of the sample result.

Sample holding time is 6 months from date of receipt.

Zeeman, Smith/Hieftje background correction or equivalent (not D_2) is required for arsenic.

Matrix modifiers are required for the analysis of arsenic.

The IDL must be shown to have been met prior to the analysis of any samples. The lab can accomplish this by submitting their most recent form XI with each case.

Each calibration blank and QC audit solution must contain the same nitric acid concentration as the samples, or diluted samples.

The sample solutions analyzed must have their matrix concentration fully documented in the raw data.

Each analytical determination must have the resulting absorbance clearly recorded and documented in their order of determination.

The calibration range of the GFAA analyses can not be exceeded. Dilute any sample that does exceed the calibration range.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The deliverables included in the SOW ILM01.0 are required.

Current quarterly form XI, XII, and XIII for each element.

Form VIII must be modified to include the slope of each addition as well as the correlation coefficient.

Correct forms V, VI, and VII to reflect the SAS contract limits and IDL where appropriate.

10. Other (use additional sheets or attach supplementary information, as needed):

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11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Preparation Blank	At least 1 per group of 10 or fewer samples	≤ IDL
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 25% or RPD is ≤ SAS IDL.
Calibration Blank	At least 1 per group of 10 or fewer samples	≤ IDL
ICVs and CCVs	as per SOW ILM01.0	as per SOW ILM01.0
Matrix Spike	At least 1 per group of 10 or fewer samples	85-115% for aqueous samples and 75-125% for sediment samples
Lab Control Spikes	1 per group of 10 or fewer samples	90-110% for aqueous samples and 75-125% for sediment samples

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis for odor in monitoring well samples. Results will be reported as threshold odor numbers (TON).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain little odor.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 140.1, odor. This is a consistent series procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

In order to prevent the picking up of extraneous odors, samples must be collected in glass bottles, filled to the top and tightly capped.

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 24 hours from date of receipt.

Glassware must be cleaned shortly before use, with non-odorous soap, an acid cleaning solution, followed with rinsing with odor free water.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

In order to be able to reproduce the calculated odor results, bench records that clearly and legibly show the odor responses, the number of people used to make the determination, and sample volumes used will be provided.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Odor	0 TONs	Difference in duplicate results should not exceed 2 TONs.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	0 TONs
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 2 TONs

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

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C. Telephone Number: (312) 886-7078

D. Date of Request:

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1. General description of analytical service requested:

Analysis for chemical oxygen demand (COD) in monitoring well samples. Results will be reported in mg/L.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain low concentrations of COD.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 410.2, COD. This is the low-level titrimetric procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples will be preserved with sulfuric acid to a pH <2.

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 28 days from date of collection.

Extreme care should be exercised to avoid inclusion of organic materials in the glassware or distilled water used for reagent preparation or sample dilution.

If COD values greater than 50 mg/L are found, the normality of the potassium dichromate and ferrous ammonium sulfate solution should be increased by a factor of 10 and the samples reanalyzed.

A lab blank sill be run with each set of sample analyzed.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records that clearly and legibly show the order and titrant volumes of the; ferrous ammonium sulfate standardization, lab blanks, samples, lab control standards, and lab duplicates. These bench records should also show sample volumes and the volume and normality of the titrant standard in order to be able to be able to reproduce the calculated COD results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
COD	5 mg/l	Difference in duplicate results should not exceed ± 10 % for concentrations > 50 mg/l or 5 mg/l for concentrations less than 50 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 3.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

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CLP Sample Management Office
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SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Determination of Oil and Grease (O & G) in monitoring well samples. Results will be reported as mg/l.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium concentration of O & G expressed mg/L.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund Remedial

4. Estimated date(s) of collection: _____.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipments by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 413.2. This is a spectrophotometric method.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples will be in 1 quart or 1 liter glass bottles and preserved with 2 ml H_2SO_4 to pH <2.

Sample holding time is 28 days from the date of sample Receipt.

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample volume is best calculated by weighing the sample bottle full and empty to nearest 5 grams.

A solvent blank is necessary for each solvent lot, and will be free of interferences.

Prepare a 5 point calibration curve containing a zero concentration standard for each cell between 0 and 0.8 absorbance.

Matrix spikes and laboratory blanks will be prepared from tapwater, H_2SO_4, and #2 fuel oil.

Dilute samples or select shorter cell path if samples absorbance exceeds that of the highest standard or exceeds 0.8 absorbance.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records and IR spectra of solvent blanks, samples, lab blanks, matrix spikes, standards, etc., will be provided along with copies of work-sheets used to calculate results.

The order of instrumental measurements and cell path lengths must be identified.

In case narrative and on bench records identify any problem samples as to emulsions, interferences, etc.

All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA audit results.

10. Other (use additional sheets or attach supplementary information, as needed):

---

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Oil and Grease	5 mg/l	Any designated field duplicate values should not exceed ± 25% or 4

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Solvent Blank (90ml of Freon)	1 per solvent lot and sample set	< 5 mg/L
Lab Blank (1 liter of tapwater at pH < 2)	At least 1 per group of 10 or fewer samples	< 5 mg/L
Matrix spike (1 liter of tapwater at pH < 2 plus 15 to 20 mg/l of #2 fuel oil)	At least 1 per group of 20 or fewer samples	80-120% Recovery

* See Section III

III. Action required if Limits are Exceeded:

Take corrective action and retest samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

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CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
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SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Analysis for total organic carbon (TOC) in monitoring well samples. Most samples will be unfiltered, although certain aliquots can be filtered and preserved at the time of collection. Results are reported as mg/l C.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

   Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium concentration of TOC expressed as mg/L C.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

   Superfund Remedial

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipments by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 415.1, the combustion method.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples will be preserved with 1 ml/l H_2SO_4 to pH< 2.

Samples should be stored at 4oC until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

The holding time is not to exceed 28 days from sample collection.

The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures and specific instrument conditions should be clearly identified. Bench records tabulating order of calibration standards, lab blanks, samples, lab control standards, spikes, duplicates, etc., will be provided along with copies of work-sheets used to calculate results. Specify the organic compound used to prepare standards and

spikes. A photocopy of the instrument readout, (i.e. stripcharts, printer tapes, etc.) must be included. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA audit results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Total Organic Carbon (TOC)	2 mg/l	Difference in duplicate results should not exceed ± 10 % for concentrations > 20 mg.l or 2 mg/l for concentrations less than 20 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Matrix Spike**	At least 1 per group of 10 or fewer samples	85-115% Recovery
Lab Blank (1 liter of tapwater at pH < 2)	At least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

** Matrix spike concentrations will be greater than 30% of the sample concentration, but spiked sample shall not exceed

the working range of the standard curve or titration.

III. Action required if Limits are Exceeded:

Take corrective action and retest samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
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SAS Number
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SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis for color in monitoring well samples. Results will be reported in color units.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain low color.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 110.2, Color. This is a platinum-cobalt colorimetric procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 48 hours from date of receipt.

Turbid samples should be clarified by centrifugation.

If the sample color exceeds 70 units, dilute the sample with distilled water to a point where the color is less than 70 units.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

In order to be able to reproduce the calculated color results, bench records that clearly and legibly show the estimated color and sample dilutions will be provided.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Color	1 color unit	Difference in duplicate results should not exceed ± 10 % for concentrations > 10 color units or 1 color units for color readings less than 10 color units

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

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SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of total dissolved solids (TDS) in monitoring well water samples. Results are reported as mg/l dissolved solids.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium to high concentration of TDS.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund Remedial

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipments by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 160.1, Filterable Residue.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples will be stored at 4oC until sample analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Holding time is 7 days from date of sample receipt.

Use standard aliquots of 100 ml; however, do not use aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller aliquot.

Residue will be weighted to constant weight pursuant to Section 7.6 of Method 160.1 (weight loss is less than 0.5 mg or less than 4% weight loss from previous loss). Constant weights will also be obtainable on a single weight basis if the sample is dried for a minimum of 12 hours. The final weight is to be used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, and duplicate samples will be provided with copies of work sheets used to calculate results. Dates and times

of when the following tasks are preformed will be recorded as part of the bench record:

* determination of tare weights,

* sample filtration,

* determination of constant weights.

* determination of residue weights.

10. Other (use additional sheets or attach supplementary information, as needed):

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11. Name of sampling/shipping contact: David Shekoski
Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TDS	10 mg/l	Difference in duplicate sample aliquots shall not exceed 2 mg for residues. Duplicate differences shall not exceed 10% for sample values greater than 200 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
2. Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg residue
3. Lab Blanks (100 ml of filtered reagent water)	At least 1 per group of 10 or fewer samples	- 20 mg/l to + 20 mg/l

* See Section III

III. Action required if Limits are Exceeded:

Take corrective action and retest samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

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SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

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1. General description of analytical service requested:

Analysis of chloride in monitoring well water samples. Results are reported in mg/l chloride.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium concentration of chloride expressed mg/L.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund Remedial

4. Estimated date(s) of collection: _____.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipments by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 325.1 (Colorimetric, Automated Ferricyanide, AA-I) 1983 ed., or

2. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA-II) 1983 ed.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be stored at 4oC until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 28 days from date of collection.

Use a standard curve between 0 and 200 mg/l or less.

The calibration curve should include 5 points or more (including a zero concentration standard).

Samples with absorbance or peak heights greater than the highest standard must be diluted and reanalyzed.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The test procedure used will be clearly identified. Bench records tabulating order of calibration standards, verification and control standards, samples, matrix spikes,

titrant blanks, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of work sheets used to calculate results. A photocopy of instrument readouts, (i.e., strip charts, printer tapes, etc.) must be included for all analysis. All records of analysis and calculation must be legible and sufficient to recalculate all sample concentrations and QA audit results.

10. Other (use additional sheets or attach supplementary information, as needed):

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11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (± % or conc.)
Chloride	2 mg/l	Difference in duplicate sample results are to be < 2 mg/l for concentrations <50 mg.l and are to be <10% for concentrations exceeding 50 mg/l. The significant figures to report depends on sensitivity of colorimetric curve or the number of significant figures in titrant volume.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (± % or conc.)
Matrix Spike**	1 per group of 10 or fewer samples	85-115% Recovery
Lab Duplicate	1 per group of 10 or fewer samples	± (10% or 5 mg/l)
Lab Blank	1 per group of 10 or fewer samples	< 5 mg/l
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110% Recovery

* See Section III

** Matrix spike concentrations will be greater than 30% of the sample concentration, but spiked sample shall not exceed the working range of the standard curve or titration.

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis for alkalinity in monitoring well samples. Results will be reported in mg/L $CaCO_3$.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium concentration of alkalinity expressed as $CaCO_3$.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 310.1, Alkalinity. This is a titrimetric procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 14 days from date of collection.

Sample volume or titrant normality should be adjusted so the titrant volume is greater than 10 mls, yet less than 40 mls.

Samples will be analyzed at 25 ± 2°C, unless the pH meter provides automatic temperature compensation.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records that clearly and legibly show the order and titrant volumes of the; titrant standardization, lab blanks, samples, lab control standards, and lab duplicates. These bench records should also show sample volumes and the volume and normality of the titrant standard in order to be able to be able to reproduce the calculated alkalinity results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Alkalinity	3 mg/l	Difference in duplicate results should not exceed ± 10 % for concentrations > 20 mg.l or 2 mg/l for concentrations less than 20 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 3.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

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[ ]

SPECIAL ANALYTICAL SERVICES
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A. EPA Region and Site Name: Region V, Onalaska Landfill

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D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis for turbidity in monitoring well samples. Results will be reported in nephelometric turbidity units (NTUs).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium amounts of turbidity.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 180.1, Turbidity. This is a nephelometric procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Samples should be analyzed within 14 days of sample receipt.

Glassware used for this procedure must be kept scrupulously clean and be free of scratches and etching.

Samples with turbidities exceeding 40 units should be diluted with turbidity free water to a concentration less than 40 turbidity units.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records that clearly and legibly show sample volumes, dilutions, and the calibration curve data will be provided in order to be able to reproduce the calculated turbidity results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Turbidity	1 NTUs	Difference in duplicate results should not exceed ± 10 % for concentrations > 10 NTUs or 0.02 NTUs for concentrations less than 10 NTUs

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Matrix Spike**	At least 1 per group of 10 or fewer samples	85-115% Recovery
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

** Matrix spike concentrations will be greater than 30% of the sample concentration, but spiked sample shall not exceed the working range of the standard curve or titration.

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: Region V, Onalaska Landfill

B. Regional Representative: Kevin Adler

C. Telephone Number: (312) 886-7078

D. Date of Request:

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis for hardness in monitoring well samples. Results will be reported in mg/L as $CaCO_3$.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Seventeen aqueous samples will be collected during each sampling event. The aqueous samples will contain medium concentrations of hardness, with the hardness being expressed as mg/L as $CaCO_3$.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund remedial.

4. Estimated date(s) of collection: _____.

5. Estimated date(s) and method of shipment:

Method of shipment will be daily shipment by overnight carrier.

6. Approximate number of days results required after lab receipt of samples:

Sample results will be required 21 days after sample receipt.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA method 130.1, Hardness, Total as $CaCo_3$. This is an EDTA colorimetric procedure.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be preserved with HNO_3 to a pH of <2 and stored at 4°C until the time of analysis. Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Sample holding time is 14 days from date of collection.

Use inhibitors as necessary.

In order to avoid large titration volumes, sample volumes should be adjusted as to not contain more than 25 mg $CaCO_3$.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records that clearly and legibly show the order of; EDTA titrant standardization, lab blanks, samples, lab control standards, and lab duplicates. In order to be able to reproduce the calculated hardness results, these bench records should also show sample volumes, titrant volumes, and the normality of the titrant.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Hardness	3 mg/l as $CaCo_3$	Difference in duplicate results should not exceed ± 10 % for concentrations > 10 mg.l or 2 mg/l for concentrations less than 10 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2.0 mg.l)
Calibration Verification Std.	1 per group of 10 or fewer samples	90-110%

* See Section III

III. Action Required if Limits are Exceeded:

Take corrective action and resample affected samples. Contact the Sample Management Office for problems that might result in the delay of reporting sample results.

Onalaska Municipal Landfill
Appendix: C
Revision: 0
Date of Revision: 3/31/92

APPENDIX C
FIELD TESTING PROCEDURES

Appendix C
Field Measurements and Monitoring

pH

Method

Electrometric

References

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Method 150.1, 1983.

Orion SA250 pH Meter Instruction Manual, 1987, Part No. 205376-001, Orion Research Incorporated, Boston, MA.

Orion Ross pH Electrode Instruction Manual, 1988, Part No. 502700-098, Orion Research Incorporated, Boston, MA.

Sensitivity

0.01 pH unit

Range

1 to 12 pH units

Sample Holding Time

Less than 6 hours

Reagents

- pH buffer solutions for pH 4, 7, and 10
- Deionized water in squirt bottle
- 3 M KCL internal filling solution

- Storage solutions

Apparatus

- pH meter
- Combination pH and reference electrode
- Beakers, plastic or glass
- Spare battery

Calibration (most pH Meters)

1. Select either pH 4 and 7, or pH 7 and 10 buffers, whichever will bracket the expected sample concentration. Note: Mix all buffer solutions before use.

2. Place electrode in pH 7 buffer solution. Wait for the pH to stabilize and adjust CAL until pH display reads 7.0.

3. Rinse electrodes and replace pH 7 buffer with either pH 4 or 10 buffer.

4. Wait for the pH value to stabilize. Adjust SLOPE until pH display reads 4.0 for the pH 4 buffer or 10.0 for the pH 10 buffer. Note: Slope values in the 92 to 102 percent range are acceptable.

5. Rinse electrodes and replace pH 4/10 buffer with pH 7 buffer.

6. If display reading is not 7.0, repeat steps 2 through 4.

Autocalibration (Orion SA 250)

1. Select either pH 4 and 7, or pH 7 and 10 buffers, whichever will bracket the expected sample concentration. Note: Shake all buffer solutions before use.

2. Select pH mode and resolution (pH 0.1).

3. Press ISO and verify that the isopotential point is 7.0.

4. Place electrode and Automatic Temperature Compensation (ATC) probe in pH 7 buffer.

5. Press CAL. The display will alternate between 0.1 and the pH value of the buffer. Wait for the pH value to stabilize. Press ENTER. After a short pause the display will advance to 0.2.

6. Rinse electrodes and ATC probe and replace pH 7 buffer with either pH 4 or 10 buffer.

7. Wait for pH value to stabilize. Press ENTER. The letters pH will be displayed. The pH meter is calibrated and ready for use. Note: Slope values in the 92 to 102 percent range are acceptable.

8. Rinse electrode and ATC probe and place into sample. Read the pH directly.

Calibration Frequency

Daily, at the beginning and end of the day, recheck calibration with pH 7 buffer once every 10 samples and after maintenance.

Operating Procedure

1. Check all connections for tight fit.

2. Inspect electrodes (and ATC probe).

3. Check battery charge.

4. Perform calibration, at the beginning and end of the day.

5. Rinse the electrode with distilled water and then with the sample to be measured.

6. Place electrode (and ATC probe) in previously mixed sample. Immerse electrode such that junction is covered by sample.

7. When the display is stable, record sample pH.

8. Recheck calibration with pH 7 buffer solution once every ten samples.

9. After use store electrode. For short-term storage (up to 1 week) soak electrode in manufacturer's recommended storage solution. For long-term storage, the reference chamber should be filled and the filling hole securely covered.

10. Cover the sensing element and/or reference junction with its protective cap and a few drops of the manufacturer's recommended storage solution.

When calibrating the meter, use pH 4 and 7 buffers for sample with pH <7, and pH 7 and 10 buffers for samples with pH >7. Measurement of pH is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C. This is not applicable for meters equipped with an automatic temperature compensation probe.

Weak organic salts, inorganic salts, and oil and grease interfere with pH measurements. If oil and grease are visible, note on data sheet. Clean electrode as described in manufacturer's instrument manual.

Avoid rubbing or wiping electrode bulb to reduce chance of error from polorization. To ensure a quick response and free-flowing liquid junction, the sensing element and reference junction must not be allowed to dry out.

Quality Control Requirements

Accuracy will be assessed by performing two measurements on two standard buffer solutions that bracket the pH range of the samples. Recheck calibration with ph 7 buffer solution once every ten samples. Each measurement will be within ±0.05 standard unit of pH selection. Precision will be assessed by duplicate measurements and must be less than or equal to 0.1 standard unit. Duplicates will be run at the rate of one every ten samples.

Preventive Maintenance (Frequency)

1. Check batteries (daily).

2. Perform a two-point calibration (daily and after maintenance).

3. Inspect the electrode for scratches, cracks, salt crystal buildup, or membrane/ junction deposits. Rinse off any salt buildup with deionized water and remove membrane/junction deposits as described in the manufacturer's operators manual (as needed).

4. Clean electrode by soaking in 0.1M HCL or $HN0_3$ for 30 minutes, followed by soaking in storage solution for at least 1 hour (as needed or when slow response is observed).

5. Drain the reference chamber and flush it with the manufacturer's filling solution (weekly).

Specific Conductivity and Temperature

References

Methods for Chemical Analysis of Water and Wastes, U.S. EPA Method 120.1, 1983.

YSI Models 33 and 33M S-C-T Meters, Instructions, November 1987, Item 021470, Yellow Springs Instrument Co., Yellow Springs, Ohio.

Sensitivity

1 μmho/cm @ 25°C.

Range

0.1 to 100,000 μmho/cm.

Sample Holding Time

Determine onsite or within 24 hours.

Reagents

Distilled water in squirt bottle and standard potassium chloride solution.

Reagent Preparation

1. Stock Potassium Chloride (KCl) Solution (1.00 N): Dissolve 74.555 g KCl in distilled water and dilute to 1,000 mL in a volumetric flask.

2. Standard Potassium Chloride Solution (0.01 N): Dilute 10.0 mL of stock 1.00 N KCl solution to 1,000 mL with distilled water using a volumetric pipet and flask.

Apparatus

Conductivity meter and electrodes. Beakers or jars, plastic, or glass; spare batteries, size D alkaline.

Calibration Procedure

1. Switch mode to OFF and unplug the probe, correct meter zero (if necessary) by turning the adjustment screw so that the meter needle coincides with the zero on the conductivity scale.

2. Switch mode to REDLINE, correct meter redline (if necessary) by turning the adjustment screw so that the meter needle coincides with the redline on the meter face. If this cannot be accomplished, replace the batteries.

3. Plug the probe into the probe jack.

4. Place the probe in the 0.01 N standard potassium chloride solution. Record temperature (°C) and conductance (micromho/cm).

5. Correct conductivity reading for temperature. This value must correspond (±10 percent) to the expected value in Table 1. If the calibration fails, then appropriate corrective action must be performed and the instrument recalibrated.

Note: The temperature probe should be calibrated against a NBS, an ATSM standard or equivalent thermometer before each sampling event.

Operation Procedure

1. Perform calibration at end and beginning of the day.

2. Switch mode to TEMPERATURE. Allow time for the probe temperature to come to equilibrium with that of the water before reading. Read the temperature on the bottom scale of the meter in degrees Celsius.

3. Switch mode to X100. If the reading is below 50 on the 0 to 500 range (5.0 on the 0 to 50 mS/m range), switch to X10. If the reading is still below 50 (5.0 mS/m), switch to the X1 scale. Read the meter scale and multiply the reading by the mode factor. The answer is expressed in microohms/cm. Measurements are not temperature compensated.

4. When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2 percent; if greater, the probe is fouled and the measurement is in error. Clean the probe and remeasure.

Operating Suggestions

- Obstructions near the probe can disturb readings.

- When the calibration test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oil, and organic matter are the most likely contaminants.

- Caution: Do not touch the electrodes inside the probe. The plating material is soft and can be scraped off.

- If cleaning does not restore the probe performance, replatinizing may be required. Always rinse the probe thoroughly in tap water, then in distilled or deionized water after cleaning and before storage. It is best (not required) to store conductivity cells in deionized water.

- Most problems in obtaining good records with monitoring equipment are related to electrode fouling and to inadequate sample circulation.

Calibration Frequency

At the beginning and end of the day or after maintenance, recharge battery after each use. Factory checkout and calibration shall be yearly or when malfunctioning.

Calculations

Calculate conductivity using the formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

where:

G_{25} = conductivity at 25°C, μmho/cm
T = temperature of sample, °C
G_T = conductivity of sample at temperature T, μmho/cm

Table 1
Conductivity Meter Calibration Table

Temperature (°C)	Conductivity (μmho/cm)
15	1,141.5
16	1,167.5
17	1,193.6
18	1,219.9
19	1,246.4
20	1,273.0
21	1,299.7
22	1,326.6
23	1,353.6
24	1,380.8

25	1,408.1
26	1,436.5
27	1,463.2
28	1,490.9
29	1,518.7
30	1,546.7

Quality Control Requirements

The accuracy of conductivity measurements will be assessed by measurement with a 0.01 N standard KCl solution before sample analysis and at the end of the day. Accuracy of measurements will be ±5 percent of the standard. Precision will be assessed by analysis of duplicate measurements which will have a relative percent difference of ≤15 percent. Duplicates will be run at the rate of one every ten samples. The thermometer on the conductivity meter will be checked before each sampling event for accuracy against an ASTM, NBS standard or equivalent thermometer. Accuracy of the measurement shall be ±1°C.

Preventive Maintenance

- The only maintenance required in battery replacement (every 200 hours or as needed).
- Recalibration (if necessary) should be done at the factory.

Field Filtering

Reference

EPA 1979, Metals 5.

Sample Handling

Filter as soon as possible after sample collection.

Reagents and Apparatus

1. 10 percent HNO_3 solution in a squirt bottle and in a liter plastic bottle
2. DI water
3. Plastic forceps
4. Filtration apparatus
5. 0.45 μm membrane filters (142 mm)
6. Glass fiber prefilters (142 mm)
7. Peristaltic pump

Reagent Preparation

1. 10 percent HNO_3 solution: Add about 900 mLs of DI water to a 1 liter Erlenmeyer flask. Using a graduated cylinder, add 100 mLs concentrated HNO_3 to the DI water while stirring.

Procedure—Filter Stand

1. Using plastic forceps, place a 0.45 μm filter on top of filter apparatus.
2. Place a prefilter on top of membrane filter.
3. Place top onto filter apparatus. Screw wing nut bolts down until even and snug. Finish tightening with plastic wrench.
4. Attach end of PVC hosing from pump to filter apparatus.

5. Run 50 to 100 mLs of HNO_3 through apparatus, rinse with 50 to 100 mLs DI water. Do not collect this filtrate.

6. Place sample bottle under outlet.

7. Turn pump on, run sample through filter, and collect filtered sample from bottom of apparatus.

8. Shut off pump.

9. Rinse twice with DI water, remove filter and dispose, proceed as above for next sample.

10. Run a DI water blank every 10 to 20 samples.

Notes

Samples with high sediment can be filtered through several membranes with increasing pore size and several prefilters. The 0.45 μm membrane filter should always be on the grid, and the coarsest filters on the top.

Procedure—Disposable Inline Filter

1. Attach tubing from pump outlet to filter inlet (note flow direction on the filter housing).

2. Place sample bottle under outlet.

3. Turn pump on, run sample through filter and collect filtered sample from outlet.

4. Shut off pump.

5. Remove filter and discard.

6. Run 50 to 100 mL 10 percent HNO_3 through pump tubing, discarding rinsate.

7. Run 50 to 100 mL DI water through pump tubing, discarding rinsate. Allow enough time to remove all water from the tubing before turning off the pump.

8. Run a DI water blank every 10 to 20 samples.

HNu Monitoring

References

HNu Model PI101 Portable Photoioniozation Analyzer Instruction Manual, December 1985, HNu Systems, Inc., Newton, MA.

HNu Model ISP1 101 Intrinsically Safe Portable Photoionization Analyzer Instruction Manual, January 1986, HNu Systems, Inc., Newton, MA.

Sensitivity

0 to 20 ppm at full-scale detection at span = 9.8 ppm; 10.2 ev Probe.

Range

0.1 to 2,000 ppm.

Calibration Gas

Isobutylene at 100 ppm.

Calibration

By analyzing a gas of known concentration, the HNu is easily calibrated. Isobutylene is typically used as the calibration gas with the instrument calibrated to benzene equivalents. When calibrating the HNu, always remember to deliver the calibration gas at ambient temperature and pressure, handle gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use, all gas cylinders must have proper regulators.

Calibration Procedure

1. Identify the probe by lamp label.

2. Attach the probe to the readout unit. Twist connector clockwise until locked.

3. Affirm the relative photoionization sensitivity (PS) calibration gas. [The required reading for isobutylene to read in benzene equivalents is equal to isobutylene ppm x PS (Isob.)/PS (benzene).]

4. Turn the function switch to battery check position. The indicator should read within the green arc. If indicator is below the green arc or if red L&D comes on, battery must be charged.

5. Zero the instrument by turning function switch to standby and rotate potentiometer until the meter reads zero.

6. Connect sampling hose to regulator outlet and the other end to sampling probe of HNu.

7. Crack regulator valve.

8. Adjust span potentiometer to obtain proper reading.

9. If calibration can not be achieved, clean the UV light source window using lens paper and HNu cleaning compound.

10. If still unable to calibrate, perform preventive maintenance. Return to factory if those procedures do not work.

Calibration Frequency

Daily or after maintenance. Recharge battery after each use. Factory check out and calibration shall be yearly or when malfunctioning.

Preventive Maintenance (Frequency)

Battery (Daily)

Check the battery charge during each period of operation. When the meter needle falls below the green zone or the low-battery indicator light illuminates recharge battery. Do not use the instrument when light is on. When not operating, leave the analyzer assembled and connected to the battery charger. In case of emergency, the analyzer may be used with a low-battery charge.

Gas Cylinders and Valves (After Installation)

All gas supply lines must be leak tested. Leakage can be determined by testing line connections and valve stems with a commercially available leak test solution. Leaks are generally stopped by tightening the fitted surfaces but may require new hardware.

Air Sampling Stream (Initially, then as Needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. A fan draws gas in through the probe and ion chamber. Small fluctuations in the flow rate will not affect the measurement. A major obstruction to the flow rate will prevent proper operation and lengthen response time. Refer to the manufacturer's instrument manual for specific procedures.

Quality Control Requirements

Precision of ±30 percent. Daily calibration.

OVA Monitoring

Reference

Model OVA 128 Century Organic Vapor Analyzer, Instruction, December 1985, Foxboro, New Haven, CT.

Sensitivity

0.1 ppm (methane).

Range

0 to 1,000 ppm.

Calibration Gas

Methane gas at 100 ppm.

Calibration

By analyzing a gas of known concentration, the OVA is easily calibrated. Methane in air at a concentration of 100 ppm is typically used as the calibration mixture, although the OVA can be calibrated to many other compounds. Primary calibration of an OVA is performed at the factory. When calibrating the OVA, always remember to deliver the calibration gas at ambient temperature and pressure, handle the gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use, and all gas cylinders must have proper regulators.

Calibration Procedure

1. Connect probe readout assembly to sidepack unit.

2. Check battery condition by moving INSTR Switch to BATT.

3. Turn INSTR to ON and allow 5 minutes to warm up.

4. Use calibration adjust knob to set needle to level desired for activating alarm. If alarm level is not zero, the calibration switch must be set to appropriate level.

5. Turn volume knob fully clockwise.

6. Turn the alarm level adjust knob until the audible alarm is activated.

7. Move calibration switch to 1X and adjust meter reading to zero using zero calibration adjustment.

8. Turn pump switch on.

9. Open hydrogen tank valve and hydrogen supply valve. Wait 1 minute.

10. Depress ignitor button until burner lights (not more than 6 seconds).

11. Set calibration switch to 10X.

12. Connect sampling hose to regulator outlet and the other end to sampling probe of OVA.

13. Crack the regulator valve.

14. Check to see if proper reading is achieved.

15. If reading is ±10 percent from expected value, return to factory for recalibration.

Calibration Frequency

Daily or after maintenance, recharge battery after each use. Factory check out and calibration shall be yearly or when malfunctioning.

Preventive Maintenance

Battery (Daily)

Check battery condition by moving the INSTR switch to the BATT position. Recharge the battery if a low charge is indicated. Do not use the instrument with a low battery charge. When not operating, leave the analyzer connected to a battery charger. Never recharge battery in a hazardous environment.

Particle Filter (As Needed)

Particle filters (primary and secondary) remove foreign matter (>10 microns) from the sample stream. These filters must be in the sample line whenever the instrument is operating. A decrease in flow rate may indicate a plugged filter.

Sampling Fixtures (As Needed)

Sampling fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particulate matter.

Hydrogen Fuel, Calibration Gas, and Valves (Initially, after Changes)

Use prepurified or zero-grade hydrogen (certified total hydrocarbons as methane <0.5 ppm recommended). All fuel and calibration gas supply lines should be leak tested. Leakage can be determined by testing line connections and valve stems with a commercially available leak test solution. Leaks are usually stopped by tightening the fitted surfaces but may require new washers or hardware.

Air Sampling Pump System (Initially, As Needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. The OVA is equipped with a flow gauge that provides a method to check for air leaks. Refer to the manufacturer's instrument manual for specific procedures.

Contaminated Control (As Needed)

Background readings may be relatively high under normal ambient conditions. The sources of high background are normal methane background, contaminated hydrogen supply gas, and contamination in the air sample line. Background readings less than 1 ppm are generally accepted since sample measurement is additive to that background. However, the low background values are more desirable. High background is commonly corrected by running the OVA for an extended time in a

clean area. Refer to the manufacturer's instrument manual for specific analysis and correction procedures.

Quality Control Requirements

Precision of ±30 percent. Daily calibration.

GLT316/001.51

APPENDIX D
SAMPLE DOCUMENTATION AND PACKING AND SHIPPING INSTRUCTIONS

Appendix D
Sample Documentation and Packing and Shipping Instructions

Sample Documentation Instructions

Sample Identification Matrix (Figure 1)

1. Enter site name.

2. Enter project number.

3. Enter the case number and/or SAS number.

4. Enter the CRL log number.

5. Specify the sample matrix using the two- or three-digit codes listed below:

   - SS—Surface Soil
   - SB—Subsurface Soil
   - SWO—Surface Water, Onsite
   - SWC—Surface Water, Creek
   - SDO—Sediment, Onsite
   - SDC—Sediment, Creek

6. Enter the sample number.

7. Enter the organic traffic label number or the SAS sample number.

8. Enter the inorganic traffic label number.

9. Enter the chain-of-custody number.

10. Indicate the laboratory to be doing the analysis (abbreviations may be used as they are shown on the current laboratory list).

11. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g., 081292).

12. Enter the shipping date.

13. Enter the airbill number of the shipment.

14. List sample tag numbers corresponding to sample containers shipped under the traffic label number listed in either box 7 or 8.

15. List the QC lot numbers of the containers matching the tag numbers listed in Item 14.

    Note: The date recorded on this form must be suitable for computer entry. Each entry must be flush left and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, chain-of-custody, and airbill numbers.

Sample Tag (Figure 2)

1. Enter date of sampling.

2. Enter time of sampling (military time only).

3. Specify "grab" or "composite" sample with an "X."

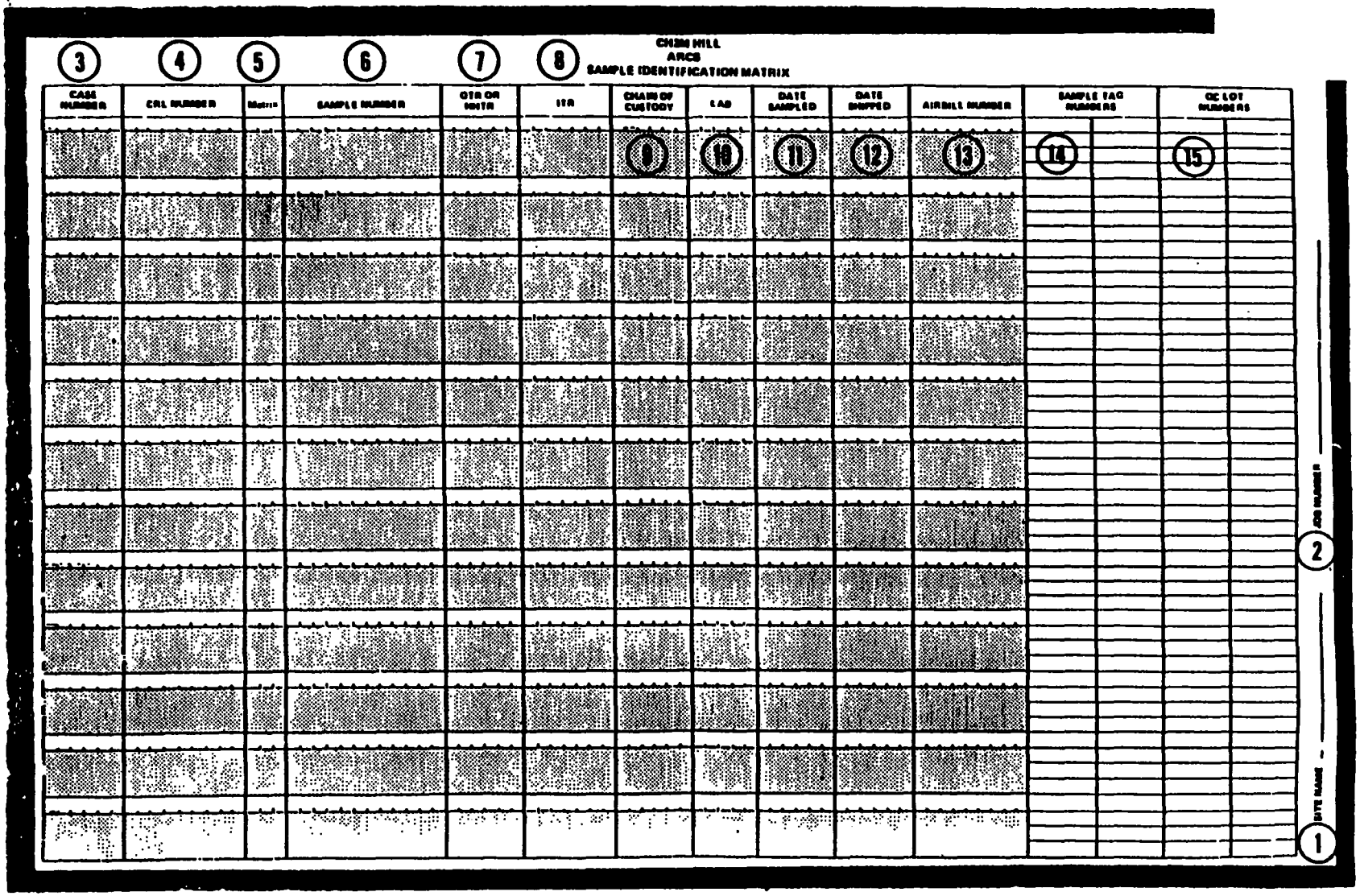
4. Enter CH2M HILL sample identification code.

5. Obtain signature of sample team leader.

6. Indicate preservative used (if any) with an "X."

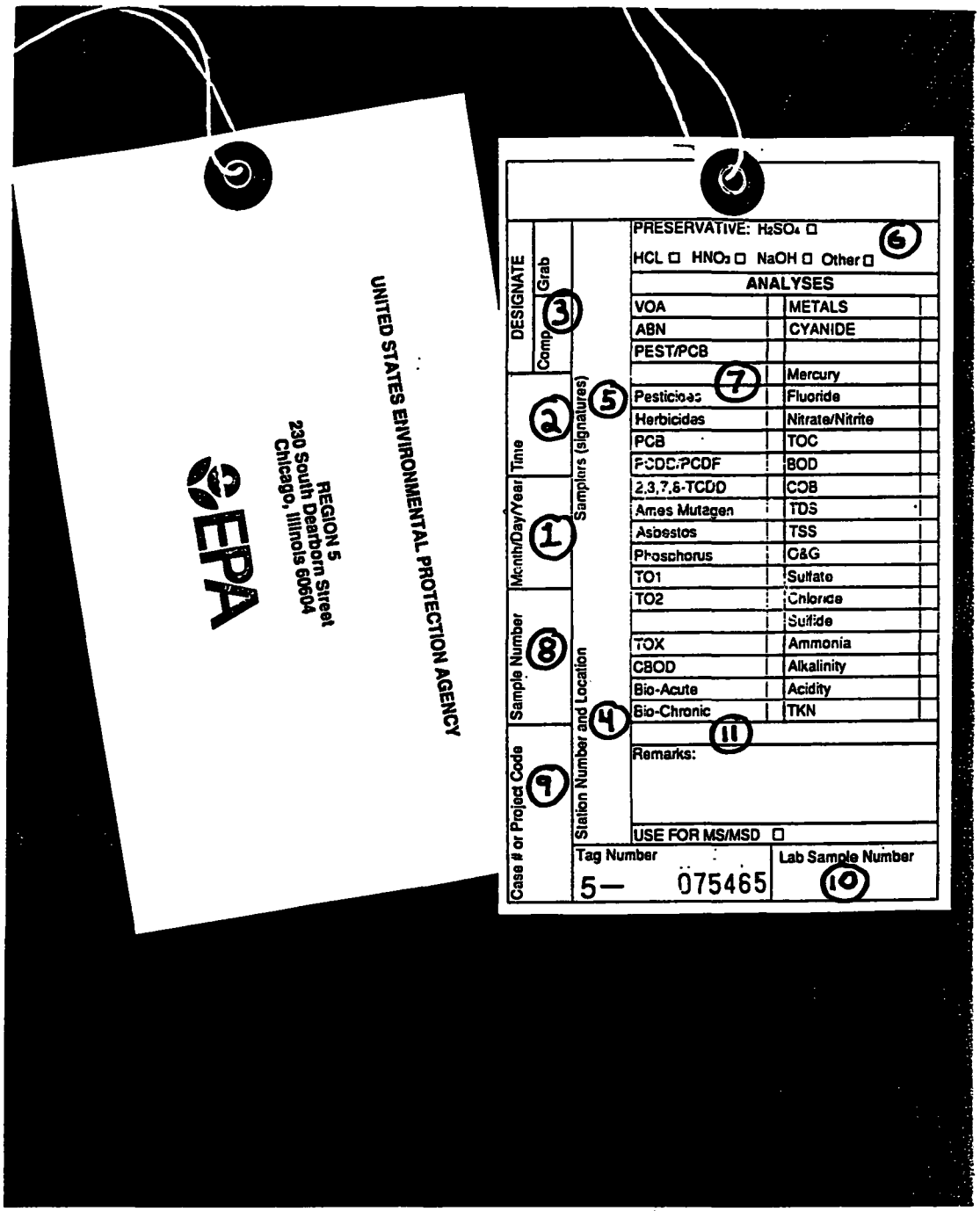
7. Specify all parameters for analysis by placing an "X" to the right of each one.

8. Indicate the sample number (for CLP Lab) or CRL log number (for CRL).



NOTE: For purposes of illustration this form has been reproduced at 50% of original size.

FIGURE 1



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 2

9. Indicate case number and/or SAS number (e.g., Case No. 1234 and/or SAS No. 5678E).

10. Leave BLANK (for laboratory use only).

11. Enter any desired analyses not listed on menu (e.g., PCBs, ammonia, sulfide, etc.) and mark box with an "X."

Inorganic Traffic Report (Figure 3)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

4b. Enter city and state of site.

4c. Enter spill ID No. (obtained from the PM or Data Coordinator).

5. Enter EPA region number (e.g., V).

6. Enter sample team leader's company/office.

7. Enter sample team leader's name.

8. Enter laboratory name and address, and laboratory contact.

9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the ITR Label Number.

13. Indicate sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from box 5 on ITR.

14. Specify sample concentration with an L, M, or H indicating contamination level.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, MS/MSD or field duplicate.

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete.**

Organic Traffic Report (Figure 4)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

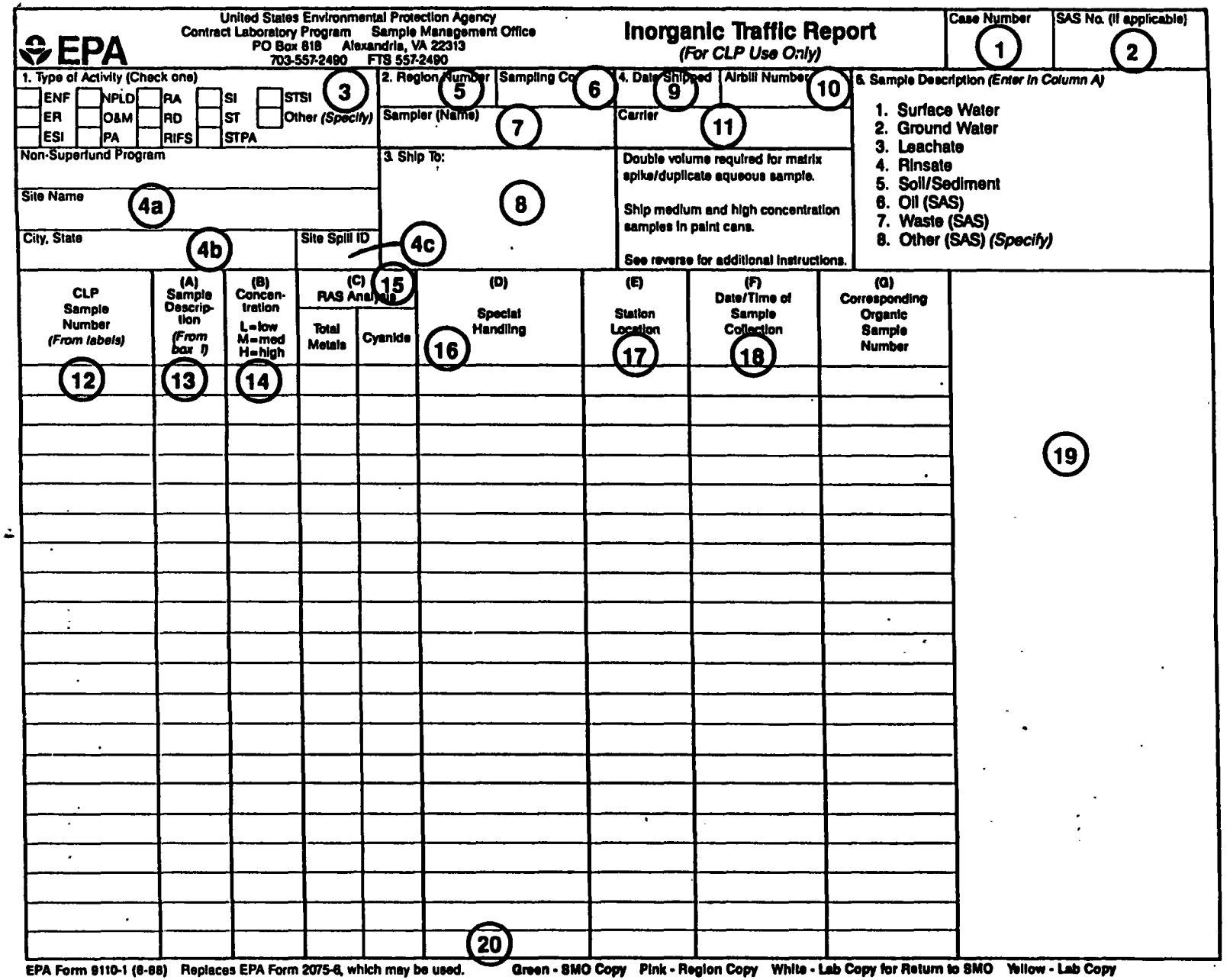
4b. Enter site city and state.

4c. Enter spill ID No. (obtained from the PM or Data Coordinator).

5. Enter EPA region number (e.g., V).

6. Enter sample team leader's company/office.

7. Enter sample team leader's name.



EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report

(For CLP Use Only)

Case Number (1) | SAS No. (if applicable) (2)

1. Type of Activity (Check one) (3)
ENF NPLD RA SI STSI
ER O&M RD ST Other *(Specify)*
ESI PA RIFS STPA

2. Region Number (5) | Sampling Co (6)

4. Date Shipped (9) | Airbill Number (10)

Sampler (Name) (7)

Carrier (11)

5. Sample Description *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

Non-Superfund Program

Site Name (4a)

City, State (4b)

Site Spill ID (4c)

3. Ship To: (8)

Double volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

CLP Sample Number *(From labels)* (12)	(A) Sample Description *(From box 1)* (13)	(B) Concentration L=low M=med H=high (14)	(C) RAS Analysis (15) Total Metals	(C) RAS Analysis (15) Cyanide	(D) Special Handling (16)	(E) Station Location (17)	(F) Date/Time of Sample Collection (18)	(G) Corresponding Organic Sample Number

(19)

(20)

EPA Form 9110-1 (8-88) Replaces EPA Form 2075-6, which may be used. Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

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FIGURE 3

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report
(For CLP Use Only)

Case Number (1) | SAS No. (if applicable) (2)

1. Type of Activity (Check one) (3)
☐ ENF ☐ NPLD ☐ RA ☐ SI ☐ STSI
☐ ER ☐ O&M ☐ RD ☐ ST ☐ Other *(Specify)*
☐ ESI ☐ PA ☐ RIFS ☐ STPA

2. Region Number (5) | Sampling Co. (6)

Sampler (Name) (7)

4. Date Shipped (9) | Airbill Number (10)

Carrier (11)

5. Sample Description *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

Non-Superfund Program

Site Name (4a)

City, State (4b) | Site Spill ID (4c)

3. Ship To: (8)

Triple volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

CLP Sample Number *(From labels)* (12)	(A) Sample Description *(From box 1)* (13)	(B) Concentration L=low M=med H=high (14)	(C) RAS Analysis (15) VOA	BNA	Pest/PCB	(D) Special Handling (16)	(E) Station Location (17)	(F) Date/Time of Sample Collection (18)	(G) Corresponding CLP Inorganic Sample Number	(19)
						(20)				

EPA Form 9110-2 (8-88) Replaces EPA Form 2075-7, which may be used. Blue - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

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FIGURE 4

8. Enter laboratory name and address and laboratory contact.

9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the OTR Label Number.

13. Specify sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from box 5 on OTR.

14. Specify the sample concentration with an L, M, or H, indicating contamination level.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, MS/MSD or field duplicate (replicate).

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete.**

SAS Packing List (Figure 5)

1. Enter assigned SAS case number.

2. Enter EPA region number (e.g., V).

3. Enter sample team leader's name.

4. Enter sample team leader's company/office and phone number.

5. Enter date sample was taken.

6. Enter date of shipment.

7. Enter site name.

8. Enter laboratory name and address.

9. Enter name of laboratory contact.

10. List SAS sample numbers, which should include the SAS number.

11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag No. 5-48246).

12. Leave BLANK (for laboratory use only).

Chain-of-Custody Record (Figure 6)

1. Enter first six digits of the CRL sample identification code.

2. Leave blank.

3. Obtain full signature of sample team leader and signed initials of active team members (including paperwork person).

4. Enter last three digits of the CRL sample identification code.

5. List sampling dates for all samples.

6. List sampling times for all samples (military time only).

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

8. List CH2M HILL sample numbers.

9. Enter number of containers per sample.

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number (1)

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office: (2)	Sampling Date(s): (5)	Ship To: (8)	For Lab Use Only
Sampling Contact: (3)	Date Shipped: (6)		Date Samples Rec'd:
(name) (4) (phone)	Site Name/Code: (7)	Attn: (9)	Received By:

	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

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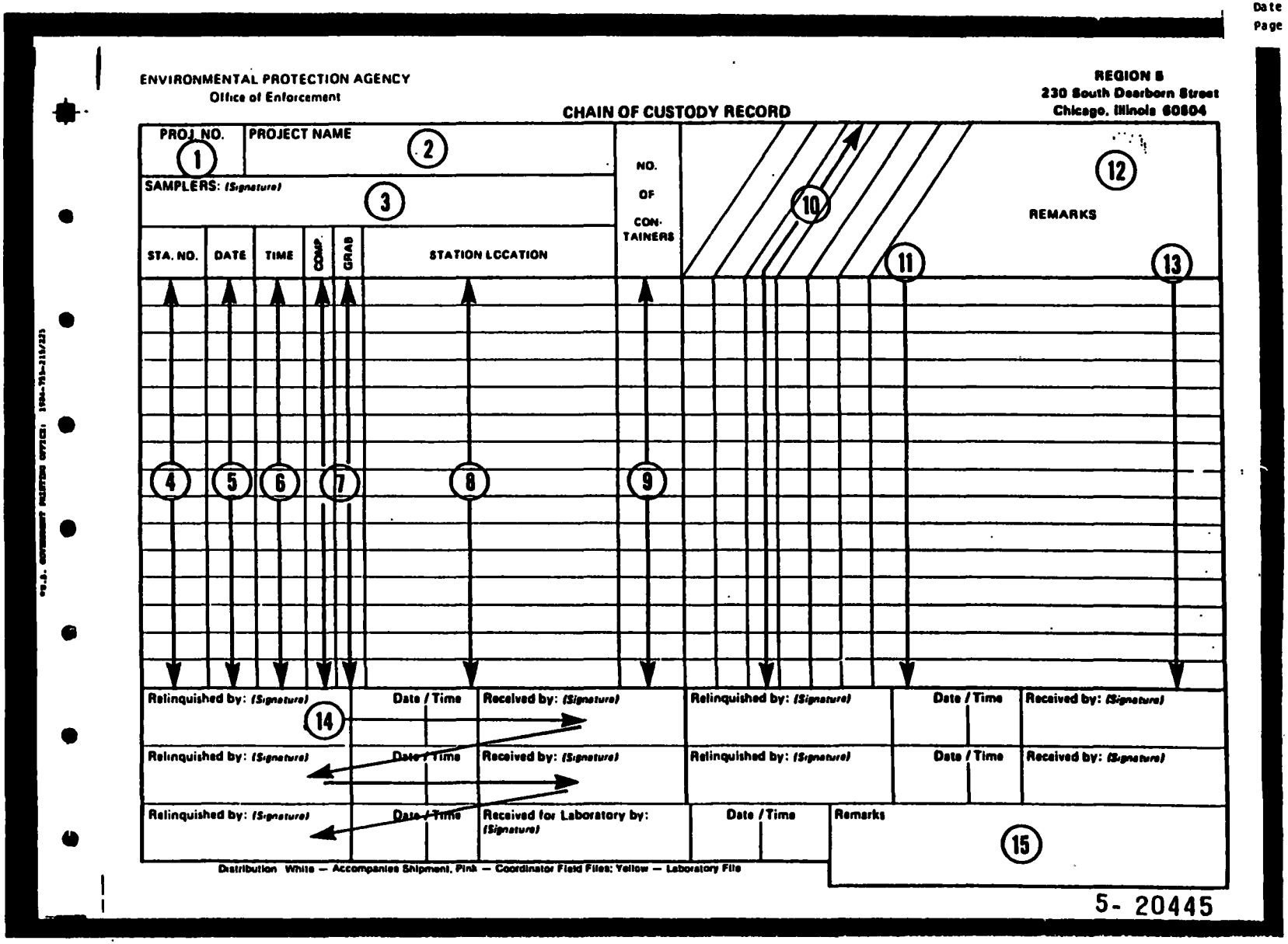
FIGURE 5



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 6

10. List analyses individually.

11. Enter column heading for traffic label number and list serial numbers for corresponding sample identification codes.

12. Write in the words "CASE No. :" or "SAS No. :" and enter the correct number.

13. Enter column heading for "tag number" and list tag numbers for each sample container.

14. Obtain signature of sample team leader and carry out chain-of-custody procedures.

15. State carrier service and airbill number, lab service, and custody seal numbers.

Combined Chain of Custody and Traffic Report Forms (Figure 7)

A. Project Code: Leave blank.

B. Account Code: Leave blank.

C. Regional Information: If sampling is in support of oversight activities, indicate here. If not—leave blank.

D. Non-Superfund Program: If sampling is not done under the Superfund program, enter the name of the program (e.g., RCRA).

E. Site Name, City, State: Complete as instructed.

F. Site Spill ID: Enter ID code provided by the office.

G. Region No.: Enter "Region 5."

H. Sampling Company: Enter "CH2M HILL."

I. Sampler Information: Complete as instructed.

J. Type of Activity:

SF—Superfund lead
PRP—PRP lead
ST—State lead
FED—Federal lead
PA—Preliminary assessment
SSI—Screening site investigation
LSI—Listing site investigation
RIFS—Remedial Investigation/Feasibility Study
RD—Remedial design
O&M—Operation & Maintenance
NPLD—National Priorities List delete
CLEM—Classic emergency
REMA—Removal assessment
REM—Removal
OIL—Oil response
UST—Underground storage tank response

K. Shipping Information: Complete as instructed.

L. Ship To: Enter lab name, address and sample recipient/custodian.

M. SAS/Case No.: Complete as instructed.

N. Sample Numbers: For routine organic/inorganic samples, enter the CLP numbers from the "stick-on" labels. For SAS samples, enter the SAS sample numbers (SAS number plus a unique sequential numeric suffix).

O. Sample Information: Complete as instructed.

P. Regional Specific Tracking Number or Tag Number: Enter sample tag number(s).

Q. Station Location Number: Enter sample identifier (as defined in the QAPP).

R. Time/Date: Complete as instructed. Use military time.

COMBINED CHAIN OF CUSTODY AND TRAFFIC REPORT FORMS

EPA — United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

SAS No. (If applicable) (M) | Case No. (M)

1. Project Code (A)	Account Code (B)	2. Region No. (G)	Sampling Co. (H)	4. Date Shipped (K)	Carrier (K)
Regional Information (C)		Sampler (Name) (I)		Airbill Number (K)	
Non-Superfund Program (D)		Sampler Signature (I)		5. Ship To (L)	
Site Name (E)		3. Type of Activity (J): SF, PRP, ST, FED; PA, SS, LSI; NPLD; CLEM, REMA, REM, OIL, UST			
City, State (F)	Site Spill ID (F)				

6. Preservative (Enter in Column D): 1. HCl 2. HNO_3 3. $NaHSO_4$ 4. H_2SO_4 5. Other (SAS) (Specify) 6. Ice only N. Not preserved

7. Sample Description (Enter in Column A): 1. Surface Water 2. Ground Water 3. Leachate 4. Rinsate 5. Soil/Sediment 6. Oil (SAS) 7. Waste (SAS) 8. Other (SAS) (Specify)

CLP Sample Numbers (from labels) (N)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp./ Grab	D Preservative from Box 6 (O)	E RAS Analysis: VOA	BNA	Pest/ PCB	High ARO/ TOX	F Regional Specific Tracking Number or Tag Numbers (P)	G Station Location Number (Q)	H Mo/Day/ Year/Time Sample Collection (R)	I Sampler Initials (S)	J Corresp. CLP Inorg. Samp. No. (T)	K Designated Field QC (U)

Shipment for Case complete? Y/N (V) | Page 1 of ___ (W) | Sample used for a spike and/or duplicate (X) | Additional Sampler Signatures (Y) | Chain of Custody Seal Number (Z)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature) (AA)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature) ☐ Declined (BB)

EPA Form 9110-2 (Rev. 5-91) Replaces EPA Form (2075-7), previous edition which may be used

DISTRIBUTION:
Blue - Region Copy Pink - SMO Copy White - Lab Copy Yellow - Lab Copy for Return to SMO

FIGURE 7

S. Sampler Initials: OPTIONAL.

T. Corresponding CLP Organic/Inorganic Sample Number: Enter CLP sample number (from "stick-on" labels) of corresponding sample from same location. Not applicable to SAS forms.

U. Designated Field QC: Indicate QC status when applicable (field blanks, trip blanks, duplicates, MS/MDS, etc.).

V. Sampling Status: Is the sampling for this Case/SAS complete? Circle one.

W. Page 1 of : Record number of documents enclosed in cooler.

X. MS/MSD and/or Duplicate: List samples.

Y. Additional Samplers Signatures: OPTIONAL.

Z. Chain of Custody Seal No.: Enter the numbers that appear on the custody seals to be used to seal the cooler (there should be two).

AA. "Relinquished by" and "Time/Date:" Complete as instructed. Use military time.

BB. Split Samples: PRP representative (PRP contractor) shall sign off here if work is oversight.

Distribution: The **Lab Copy** and **Lab Copy for Return to SMO** are included with the shipment. The **Region Copy** and **SMO Copy** are returned to the office.

Notice of Transmittal (Figure 8)

1. Enter name of team leader.

2. Enter team leader's firm name.

3. Enter CH2M HILL project number.

4. Enter case number.

5. Enter date.

6. Enter number of samples shipped.

7. Enter matrix of samples.

8. Enter the site name in words.

9. Enter the location of the site (city, state).

Central Regional Laboratory Sample Data Report (Figure 9)

The Central Regional Laboratory Sample Data Report is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number or SAS number.

2. Enter the site name.

3. Enter the laboratory name.

4. Enter the date shipped.

5. Enter the Superfund D.U. number.

6. Enter the EPA RPM.

7. Enter the CERCLIS number.

8. Enter the page numbers.

9. Enter the CRL numbers.

10. Enter the organic or inorganic traffic report number or the SAS packing list number.

11. Check the appropriate boxes for the analyses to be performed.

NOTICE OF TRANSMITTAL

DATE:

TO: CH2M HILL - REM/FIT Office, Reg. V-X (WI)
310 West Wisconsin Avenue, Suite 700
P.O. Box 2090
Milwaukee, Wisconsin 53201

Attn.: Shirley Stringer

FROM: ① (name) / ② (firm)

CH2M HILL PROJECT #: ③

Enclosed are appropriate copies of the sample documentation forms completed under Case # ④ for the ⑤, 19⑤ shipment of ⑥ (qty) ⑦ (matrix) samples from the ⑧ site located in ⑨, ⑨.

GLT85/15

FIGURE 8

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT
ORGANICS/INORGANICS

THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

CASE NUMBER/SAS No. (1) SITE NAME (2) LABORATORY (3) DATE SHIPPED (4)

SUPERFUND OU NUMBER (5) EPA RPM or OSC (S M S)/(CES) (6) CERCLIS NUMBER (7) PAGE (8) OF

ACTIVITY NUMBER

CRL LOG NUMBER	ORGANIC TRAFFIC REPORT NUMBER	INORGANIC TRAFFIC REPORT NUMBER or SAS Packing List No.	WATER OR LIQUIDS														SEDIMENTS or SOILS													
(9)	(10)	(10)					(11)																						

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 9

Packaging and Shipping Procedures

Low-Concentration Samples

1. Prepare coolers for shipment.
   - Tape drains shut.
   - Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
   - Place mailing label with laboratory address on top of coolers.
   - Fill bottom of coolers with about 3 inches of vermiculite or use performed poly-foam liner.
   - Place appropriate traffic reports, SAS packing lists, or regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.
2. Arrange decontaminated sample containers in groups by sample number.
3. Mark volume levels on bottles with a grease pencil.
4. Secure appropriate sample tags around lids of containers with string or wire.
5. Secure container lids with strapping tape.
6. Arrange containers in front of assigned coolers.
7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.
8. Seal each container within a separate plastic bag.
9. Arrange containers in coolers so that they do not touch.

10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags and placed on and around the containers (especially on VOA vials).

11. Fill remaining spaces with vermiculite (or place poly-foam liner cover on top of samples).

12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express.

13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.

14. Close lid and latch.

15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape (Figure 10).

16. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals (see Figure 10).

17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.

18. Telephone the SMO in Alexandria, Virginia.

    (Note: This step should be omitted for samples sent to the CRL).

    Ms. Leslie Braun (subject to change)
    703/557-2490

    Provide the following information:

    - Your name
    - Project name
    - Case number

- Number of samples sent to each laboratory for analysis
- Airbill numbers

This must be done IMMEDIATELY following sample shipment. If the SMO is closed at that time, call in the information first thing the next day.

Medium- and High-Concentration Samples

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required (Figures 10 and 11). Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed in the cooler, as indicated below.

1. Place approximately ½ inch of vermiculite in the bottom of the can.

2. Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.

3. Place the jar in the can and fill the remaining volume with vermiculite.

4. Close the can and secure the lid with metal clips.

5. Write the traffic report number on the lid.

6. Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.

7. Place the cans in the cooler.

Special Instructions for Shipping Samples by Federal Express (Figures 11 and 12)

1. Label cooler as hazardous shipment.

   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker saying "This Side Up" on two adjacent sides.
   - Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NANo. 9188.
   - Label cooler with "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.

2. Complete the special shipping bill for restricted articles (Figures 10 and 11).

   - Under Proper Shipping Name, write "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.
   - Under Class, write "ORM-E."
   - Under Identification No., write NA No. 9188.

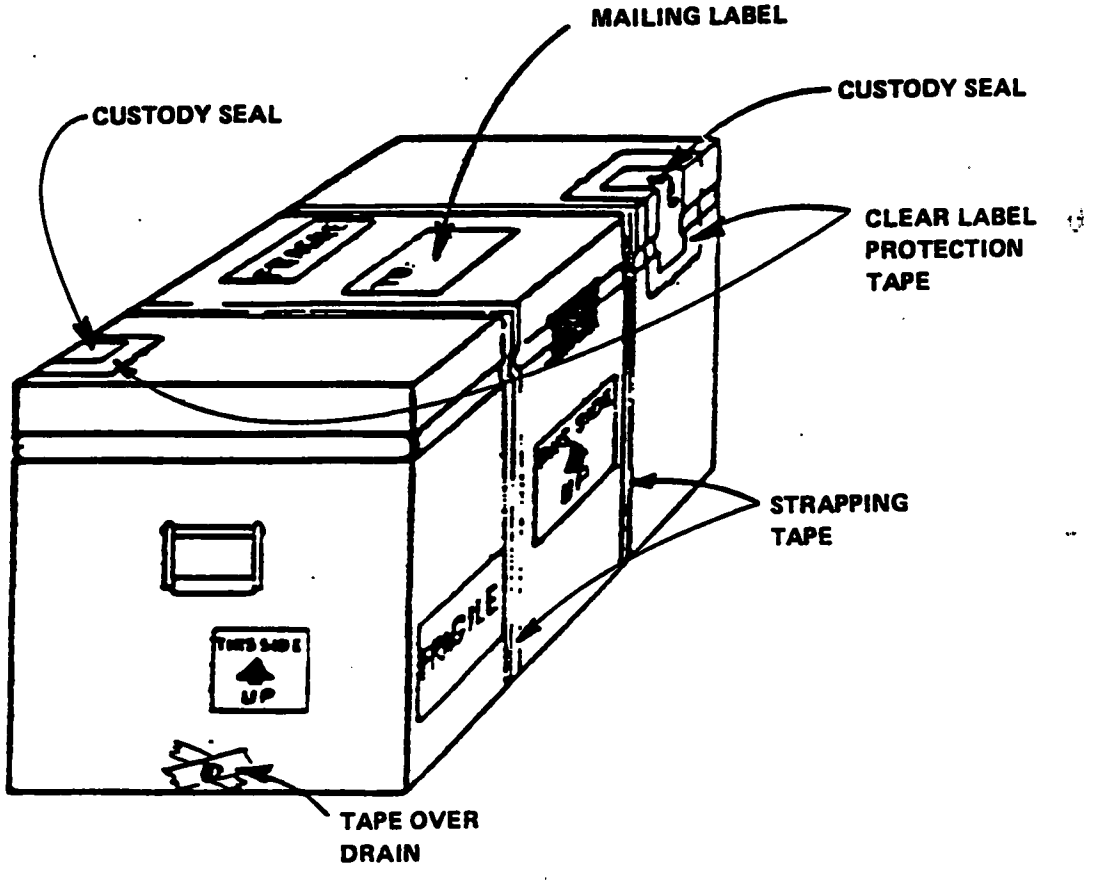

MAILING LABEL
CUSTODY SEAL
CUSTODY SEAL
CLEAR LABEL
PROTECTION
TAPE
STRAPPING
TAPE
FRAGILE
THIS SIDE
UP
TAPE OVER
DRAIN

FIGURE 10

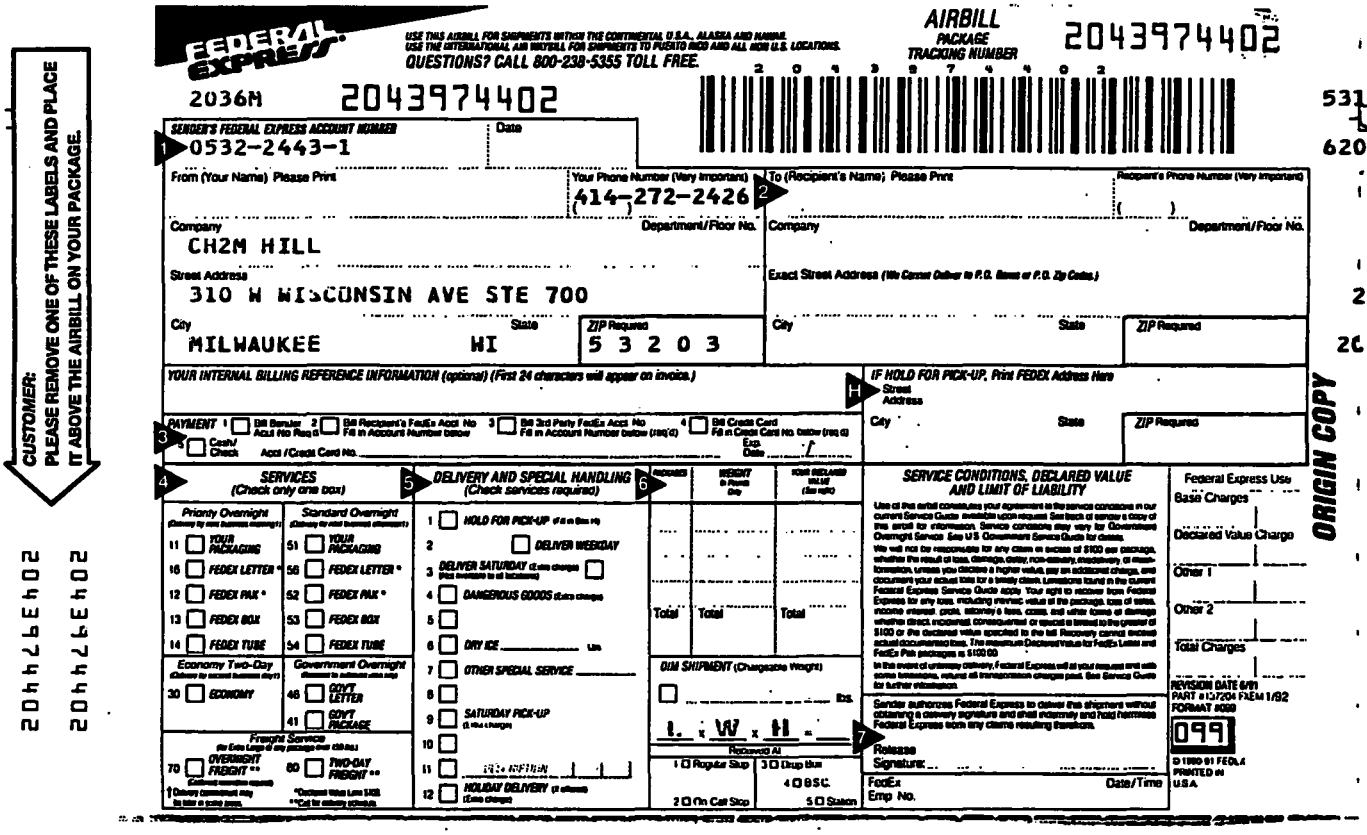


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FIGURE 11

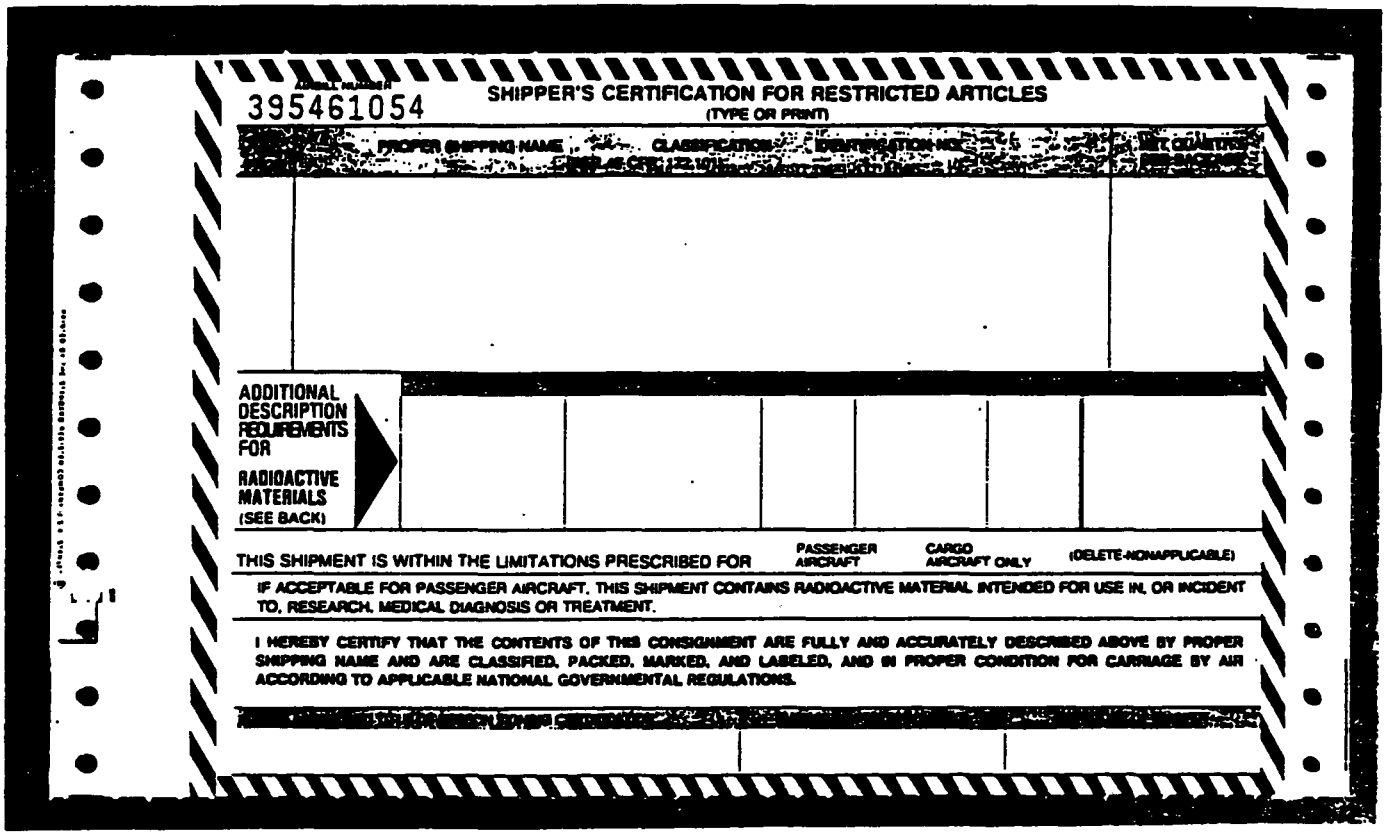

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FIGURE 12