QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

ONALASKA LANDFILL SITE
Onalaska, Wisconsin

WA 01-5LL5.0/Contract No. 68-W8-0040

January 12, 1989

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Engineers
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GLT824/30

Remedial Planning Activities
(ARCS V)
Contract Number 68-W8-0040
QUALITY ASSURANCE PROJECT PLAN (QAPP)

Project Title: Onalaska Municipal Landfill RI/FS

EPA WA No.: 01-5LL5.0

EPA Project Officer: Doug Ballotti

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Reviewed _____ Date: _____
EPA Director, Central Regional Laboratory

Approved _____ Date: _____
EPA QA Officer

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Section 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 determine that the precision, accuracy, completeness, and representativeness of its data are known and documented. To determine that the responsibility is met uniformly, each EPA contractor must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is contracted to perform.

This QAPP presents the organization, objectives, functional activities, and specific QA and quality control (QC) activities associated with the remedial investigation activities for the Onalaska Municipal Landfill site.

Section 2
PROJECT DESCRIPTION

SITE DESCRIPTION

The Onalaska Municipal Landfill is in LaCrosse County, Wisconsin, approximately 10 miles north of the City of LaCrosse near the confluence of the Mississippi River and within 400 feet of the Black River (Figure 2-1). Several homes are within 500 feet of the site, and a subdivision of about 50 homes is 1.25 miles southeast of the site. The area is generally rural, and residents use a local surficial sand and gravel aquifer for their water supply.

The 11-acre site was mined as a sand and gravel quarry in the early 1960s (see Figure 2-2). 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 with 2 feet of compacted clay. Two gates restrict but do not prevent vehicular access to the site. From 1980 to 1982, groundwater contamination was documented in an area 300 feet south of the landfill.

SITE GEOLOGY

The site geology consists of soil units and unconsolidated deposits overlying a sandstone bedrock. The soil units consist of a group of fine to loamy fine sands. The soil drains readily and is easily eroded by the wind. The wetlands adjacent to the site are underlain by poorly drained alluvial soils consisting of sandy and silty materials.

The unconsolidated deposits are about 135 feet thick and consist primarily of sand and gravel of glaciofluvial and alluvial origin. The site is located within an eroded bedrock valley that was filled with outwash transported by the Black and Mississippi Rivers near the end of Wisconsinan stage glaciation.

Two distinct subsoils have been observed at the site. The near-surface subsoil has a higher silt and clay content; a noncontinuous lense of silty clayey sand is present within the subsoil. The hydraulic conductivity of the near-surface subsoil was estimated to range from 1×10^{-3} to 1×10^{-6} cm/s. The other subsoil consists primarily of very

fine to coarse sands with trace amounts of gravel, silt, and clay. The hydraulic conductivity of the lower subsoil ranges from 1 x 10^{-2} to 1 x 10^{-3} cm/s (Warzyn 1978).

The bedrock near the Onalaska Landfill consists of undifferentiated Cambrian sandstone up to 1,200 feet thick (Young and Borman 1973) and includes the Jordan Sandstone, the St. Lawrence Formation, and the Franconia, Galesville, Eau Claire, and Mount Simon Sandstones. The sandstones are fine- to coarse-grained and contain small amounts of shale. Bedrock was not encountered in any of the borings drilled onsite, but was found at a depth of 134 feet while drilling a replacement well on the Miller property 300 feet south of the site.

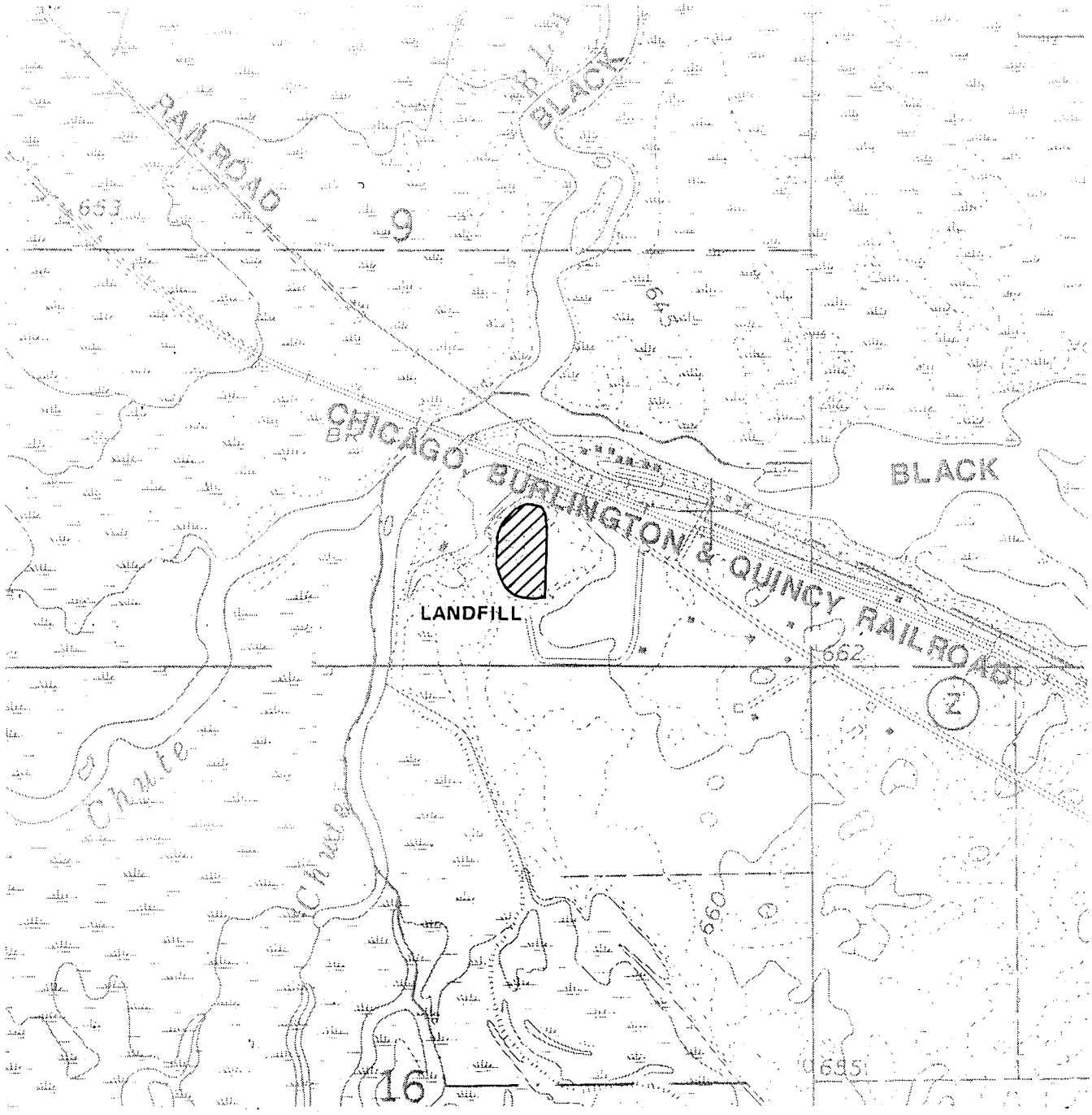
HYDROLOGY/HYDROGEOLOGY

The location of the Onalaska Landfill in relation to the Black and Mississippi Rivers is of critical importance in understanding the surface water and groundwater flow regime at the site. The Black River flows south-southwesterly within 400 feet of the site. As it flows past the site, the river channel branches into tributaries that flow into Lake Onalaska and the Mississippi River. Maximum, average, and minimum discharges of the Black River are measured 6 miles upstream at the Galesville gauging station and are 65,500, 1,635 and 180 cfs, respectively.

The main channel of the Mississippi River flows southeast within 1.5 miles of the site. The Mississippi River is dammed approximately 6 miles south of the site, forming Lake Onalaska and most of the wetlands adjacent to the site.

Groundwater flow directions were determined using readings from six monitoring wells, historic reference quarterly water level measurements, and water level measurements recorded June 1, 1988 (Figure 2-3). For most of the year, horizontal groundwater flow is south-southwesterly toward the wetlands bordering the Black River. However, during the spring runoff period the flow field is altered, and groundwater flows south-southeasterly.

The horizontal groundwater gradient ranges from 2.2 x 10^{-3} to 2.2 x 10^{-4} and averages 5.3 x 10^{-4}, remaining relatively flat throughout the year. The variation in horizontal groundwater gradient is due to seasonal variations associated with spring runoff. Vertical groundwater gradients measured at the monitoring well nest (B4S and B4D) indicate there is a slight downward gradient of 1 x 10^{-2}.



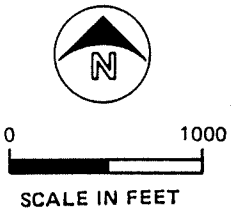


FIGURE 2-1
SITE LOCATION MAP
ONALASKA LANDFILL QAPP

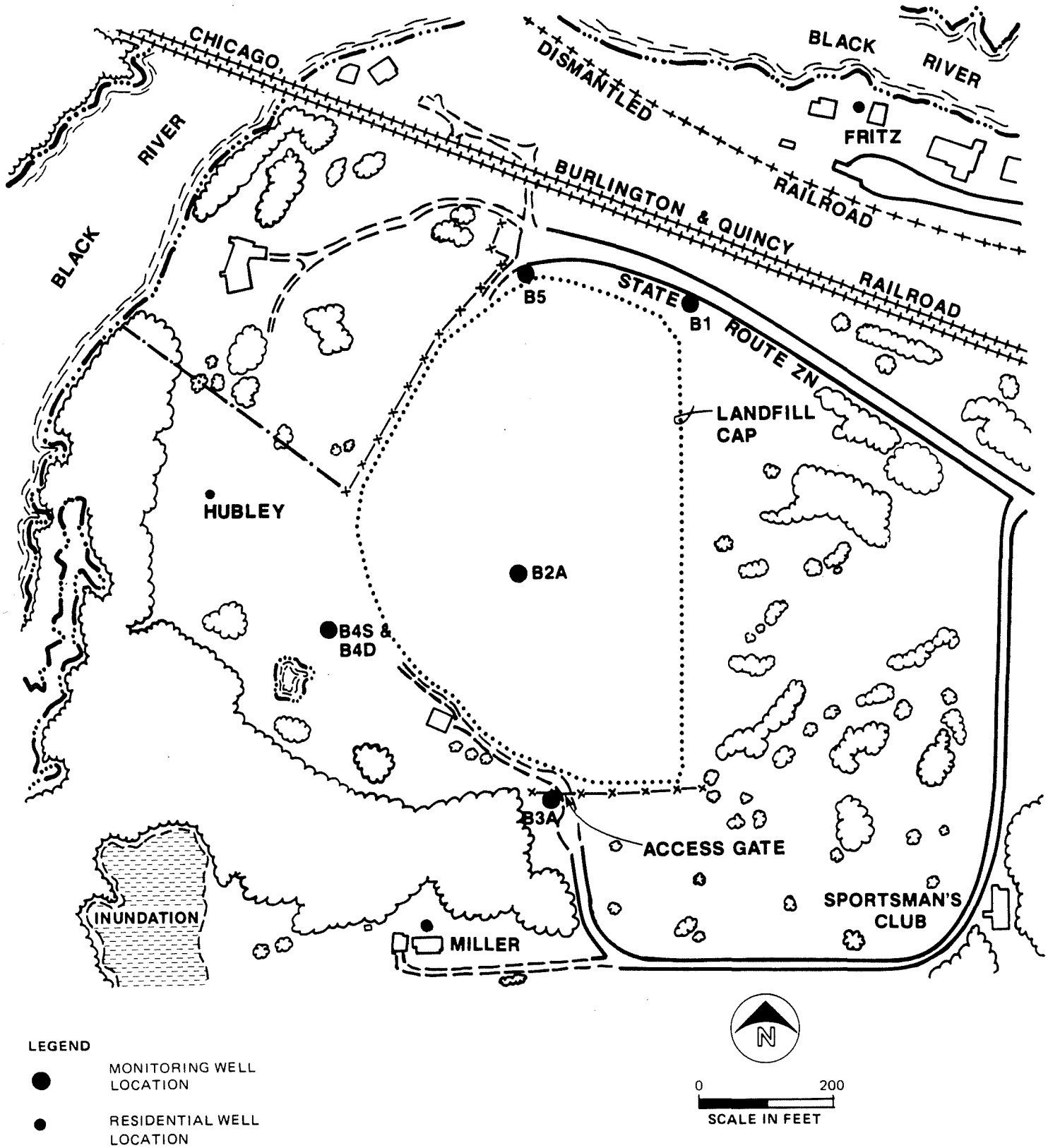
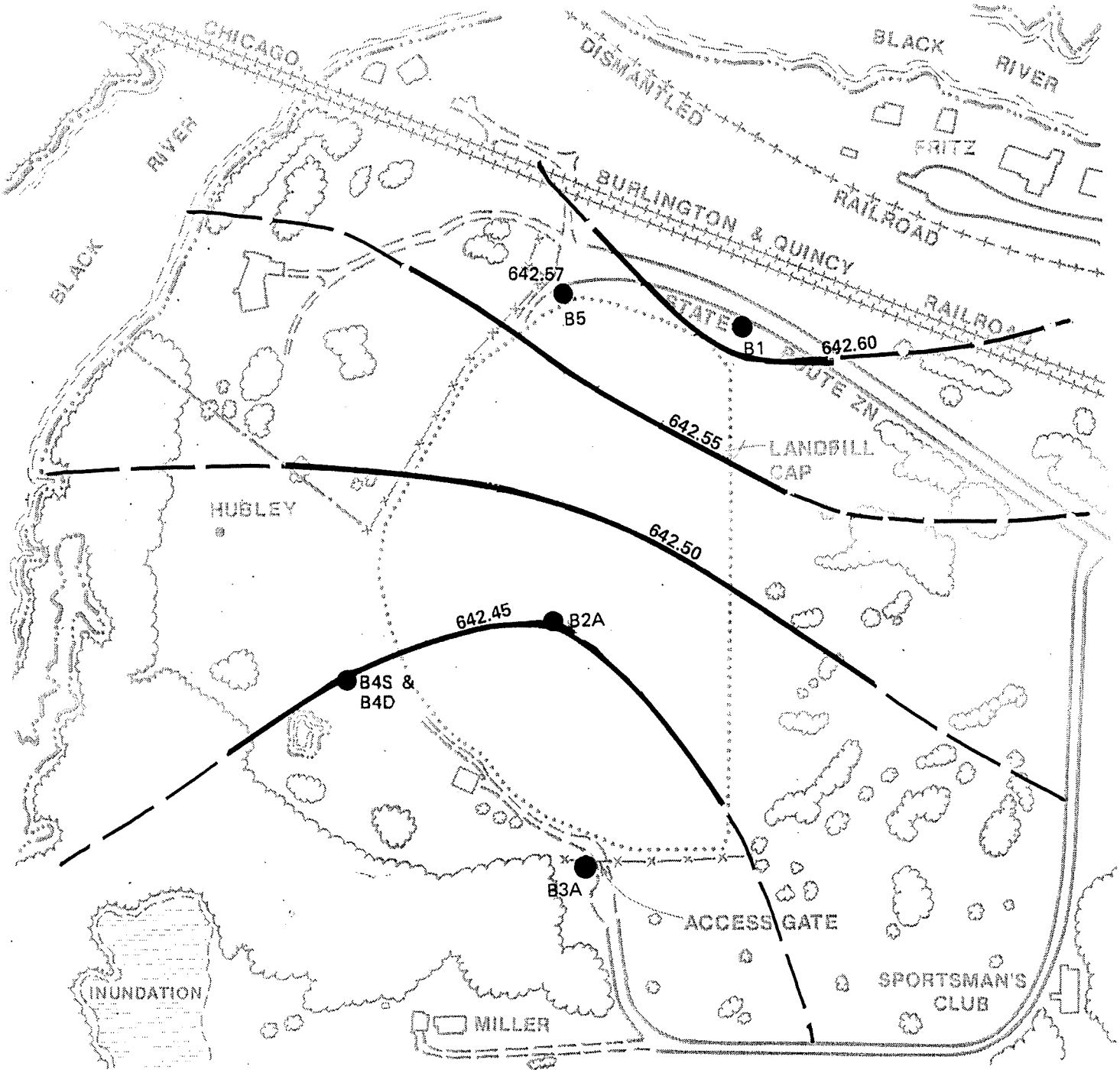


FIGURE 2-2
SITE MAP
ONALASKA LANDFILL QAPP



LEGEND

- MONITORING WELL LOCATION
- GROUNDWATER CONTOUR
- INFERRED CONTOURS

FIGURE 2-3
GROUNDWATER CONTOUR MAP, 6/1/88
ONALASKA LANDFILL QAPP

Careful review of the historic groundwater level measurements indicates that the direction of groundwater flow displays considerable variation. The groundwater flow regime at the site is affected by the seasonal surface water fluctuations in the Black and Mississippi Rivers. The fluctuations are directly related to the elevation changes of the Black River and Lake Onalaska, which either recharge the adjacent sand and gravel aquifer or receive groundwater discharge as the river and lake levels fluctuate. During most of the year, groundwater is discharging under the site in a south-southwesterly direction to the upper Mississippi River Wildlife and Fish Refuge bordering the Black River. However, during spring runoff when surface water levels are high, the Black River and Lake Onalaska recharge the sand and gravel aquifer. This modifies the direction of groundwater flow to the south-southeast away from the river. The seasonal changes in the groundwater flow regime correlate extremely well with seasonal changes in the Black River discharge volume (Borman and Young 1973). A conceptual cross section of the site showing the proximity of the Black River to the landfill is presented in Figure 2-4.

SITE HISTORY AND BACKGROUND

SITE HISTORY

The Town of Onalaska owned and was licensed to operate the Onalaska Landfill from 1969 until the Wisconsin Department of Natural Resources (DNR) ordered its closure in 1980. During the 11 years of operation, the Onalaska Landfill provided waste disposal for residential, commercial, and industrial generators located within the township and for nonresidents with a written permit. 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 lockable 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 dirty, black smoke resulting from the burning of naphtha, an oily industrial solvent waste. Consequently, the DNR required an area be designated specifically 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. A partial list of industrial contributors is shown in Tables 2-1 and 2-2.

Outers Laboratories and Metallics, Inc., contributed significant quantities of industrial wastes to the site. Daily landfill operation reports indicate Outers Laboratories and Metallics, at the time owned by the same individual, 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, Outers and Metallics hauled solvent wastes in 55-gallon barrels. Once a week, 20 to 25 barrels of industrial wastes from both 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, approximately 300 barrels were mass buried at the landfill.

On one occasion, the tank truck hauling the wastes was buried, presumably in the south section of the landfill, when the contents could not be drained because the discharge outlet was plugged with hardened paint resin and solvent. In August 1975, the DNR recommended that Outers find alternative methods to dispose of their "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 they were 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

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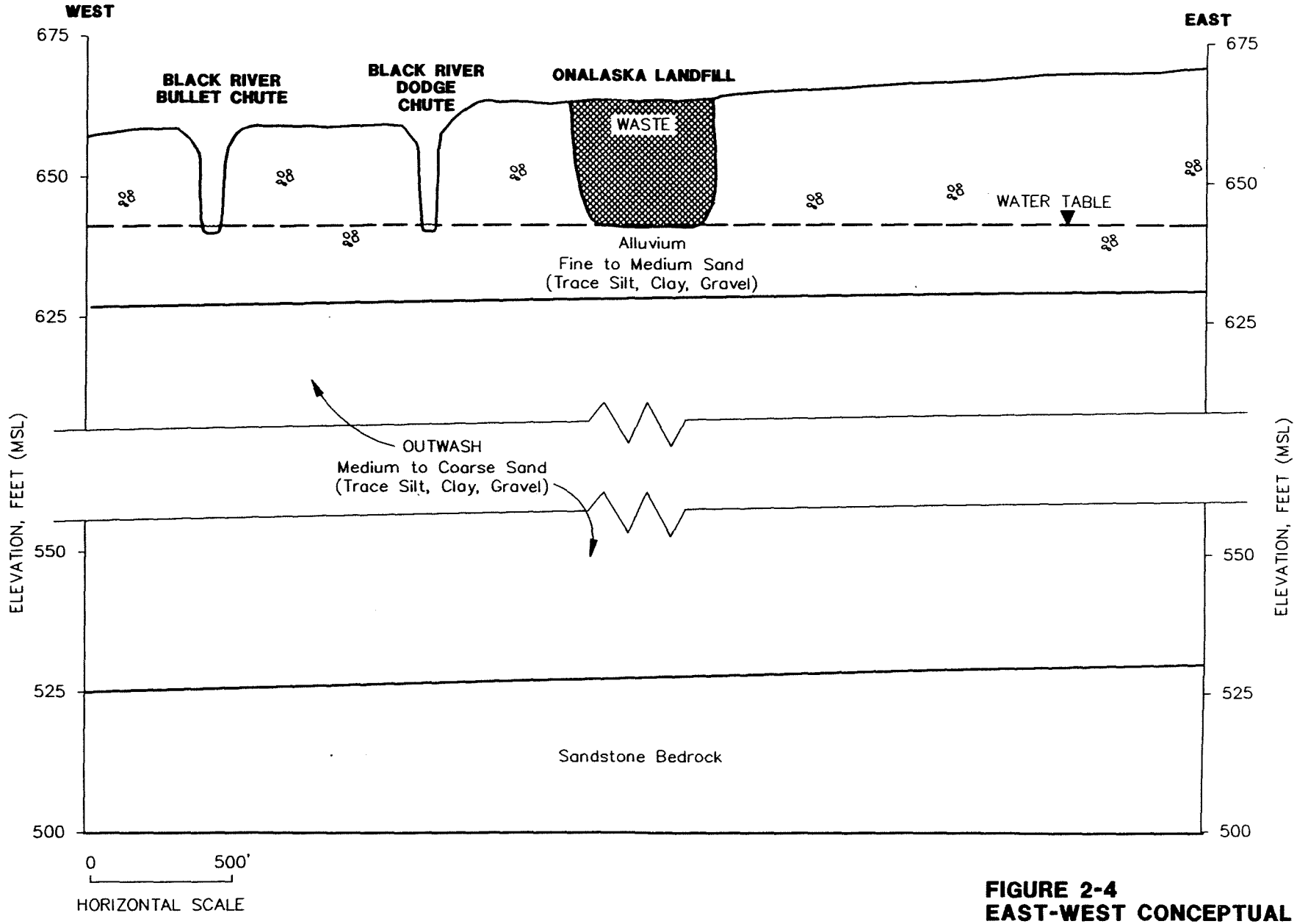

FIGURE 2-4
EAST-WEST CONCEPTUAL CROSS SECTION
ONALASKA LANDFILL QAPP

Table 2-1
ONALASKA LANDFILL USERS

Town of Onalaska

Town of Medary

Town of Campbell

City of Onalaska

City of French Island

City of West Salem

Outers Laboratories

Metallics, Inc.

Continental Can Company, Inc.

Heileman's Brewing Company

Bly Rendering Works

St. Francis Hospital

Trempealeau Electric Company

Modern Clean-Up Service (hauler)

Onalaska Rubbish Service (hauler)

Bill's Pumping Service (hauler)

Hilltopper Rubbish Service (hauler)

Midway Machine Products

Coulee Tool and Die

Empire Screen Printing, Inc.

L. B. White Company, Inc.

Pesticide firm from Waterloo, Iowa

Septic Tank Cleaner Firm

Unknown nearby school

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Table 2-2
PARTIAL LIST OF WASTES DEPOSITED AT
ONALASKA LANDFILL

	SOURCE
High Flash Naphtha (metal cleaning waste)	Outers/Metallics
Mineral Spirits	Outers/Metallics
Gun Oil	Outers
Gun Cleaning Solvents	Outers
Paint Residues	Outers/Metallics
Asphaltum	Outers/Metallics
Water Soluble Solvents (Okite Materials)	Outers/Metallics
Lubricating Oils	Outers/Metallics
Synthetic Lubricant (PTL-1009) (amine soap)	Continental Can
Cannery wash (99 percent water)	Continental Can
Septic Tank Sludges	Septic Tank Sludge Haulers
Animal Carcasses, Hides, Intestines	Bly Rendering Works
Animal Manure	Bly Rendering Works
Transformers	Trempealeau Electric
Entire Rendering Works Building (4 stories)	Bly Rendering Works
Insecticides (DDT, etc.)	Waterloo, Iowa
Beer Cooling Units	Heileman's Brewing
Beer Cans (partially full and empty)	Heileman's Brewing
Cardboard, Wood, Paper Waste	St. Francis Hospital, Outers/ Metallics
Plastic Waste	St. Francis Hospital
Empty Drums	Outers/Metallics
Full Drums (Naphtha and Paint Wastes)	Outers/Metallics
Tank Truck (paint wastes) (500 gal)	Outers/Metallics
Municipal Rubbish	Town or City of: Onalaska, Medary, Campbell, French Island, West Salem
Tires	Tire Haulers

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and submitted a report to the 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 at the site was 1 foot. Studies showed that the recurrent 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 the 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 monitoring wells and private wells for compliance with drinking water standards for organic and inorganic constituents. The investigations indicated groundwater contamination had occurred. The water in Cecil Miller's residential well south of the site exceeded the drinking water standards for barium 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.

HAZARDOUS MATERIALS CHARACTERIZATION

The Onalaska landfill used about 7 acres for open pit disposal. Records indicate that refuse was compacted and covered at the end of each collection day. There is little indication that the wastes were segregated, so industrial, commercial, and municipal wastes are considered mixed throughout the fill area. The industrial waste solvents from Outers Laboratories and Metallics, Inc., are an exception since a specific area was designated specifically 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 other commercial wastes were deposited simultaneously in the same area 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 bi-monthly. Open burning reportedly continued, even though banned, as late as 1979.

Source Description

Table 2-3 is a summary of the primary industrial and commercial waste contributors to the landfill. Outers Laboratories and Metallics, Inc., contributed the greatest quantities of liquid industrial wastes delivered to the landfill. Their liquid 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 (correspondence from Outers Laboratories). During the period the liquid solvent wastes were delivered to the site for open burning, no specific area was used for dumping and burning of the waste. Drums containing solvent or paint residue waste were also left to be burned or buried. Later, the wastes were poured directly into prepared pits from 55-gallon barrels and still later from a 500-gallon tank truck. Paint residues and solvents were also delivered to the landfill and deposited along with the other solvent wastes. In addition, they deposited smaller quantities of other wastes that included paint and ink components, cutting oils, lubricating oils, and asphaltum. Outers and Metallics delivered about 20 to 25 drums of solvent and paint residue per week from late 1969 to 1975 (correspondence from Outers to DNR, November 10, 1975) resulting in a total estimated volume of about 320,000 gallons.

Continental Can discharged large quantities of can manufacturing wastes. The waste was composed of mostly water and an amine soap and is believed to be biodegradable (correspondence from Town of Onalaska, July 21, 1977). Continental Can reportedly discharged 600 gallons per week of can wash waste between 1975 and 1978, resulting in a total estimated volume of 90,000 gallons.

There are no other known industrial liquid wastes at the site. The other industrial contributions listed in Table 2-3 consist primarily of solid wastes that include insecticides, paint cans, bottles, plastics, paper, and other commercial rubbish. Figure 2-5 shows the approximate boundary of the landfill disposal area and possible disposal locations for some specific wastes.

Table 2-3
MAJOR COMMERCIAL AND INDUSTRIAL WASTE CONTRIBUTIONS

Generator	Description of Waste Deposited	Manner of Disposal	Quantity	Time Frame
Outers Laboratories and Metallics, Inc.	Naphtha (VM&P, High-Flash, and Stoddard Solvent); Toluene; Solvosol	Open burning and occasional burial of drums throughout site	5,000 gal/mo	Late 1969-71 1971-1976
		Open pit dumping followed by cover and compacting	6-7 drums/mo	
		Barrels (intact)	300 barrels	1976
	Paint and ink residues	500-gallon tank truck, and 5-gallon pails		
	Degreasers (water soluble); cutting oils, lube oils, asphaltum			
	Gun oil and/or gun cleaning solvents	Small bottles	Truck load	
	Solid wastes (paper, plastics, packing material)	Open pit dumping	Two noncompacting trucks/week	1970-1978
Continental Can Co., Inc.	Can wash containing 99% water; synthetic lubricant PTL-1009	Bill's Pumping Service, land applied	600 gal/week	2 yrs, 10 mos. (1975-78)
St. Francis Hospital	Paper, plastics, miscellaneous	Direct dumping	20 yd^3 every 4 days	1978 (?)
Trempeauleau Electric	Transformers (transformer oil may have been used to burn off insulation to salvage copper)	Dumped near sign "Place Transformers Here"	12 each	1973 (?)
Heileman's Brewing Company	Shorts and rejects of empty cans; beer cooling units	Direct dumping	Unknown	1975 (?)
Bly Rendering Works	Stack of animal hides after fire; cattle intestines, manure	Pit dumping	3 dump trucks/wk	
	Entire building (four stories)	Buried in deep hole		
Unknown Firm from Waterloo, Iowa	Insecticides (DDT, etc.; in paper bags)	Buried in designated area (sign)	Unknown	1975 (?)
Unknown Septic Tank Cleaners	Septic waste	Land dumping	Unknown	1970 - (?)

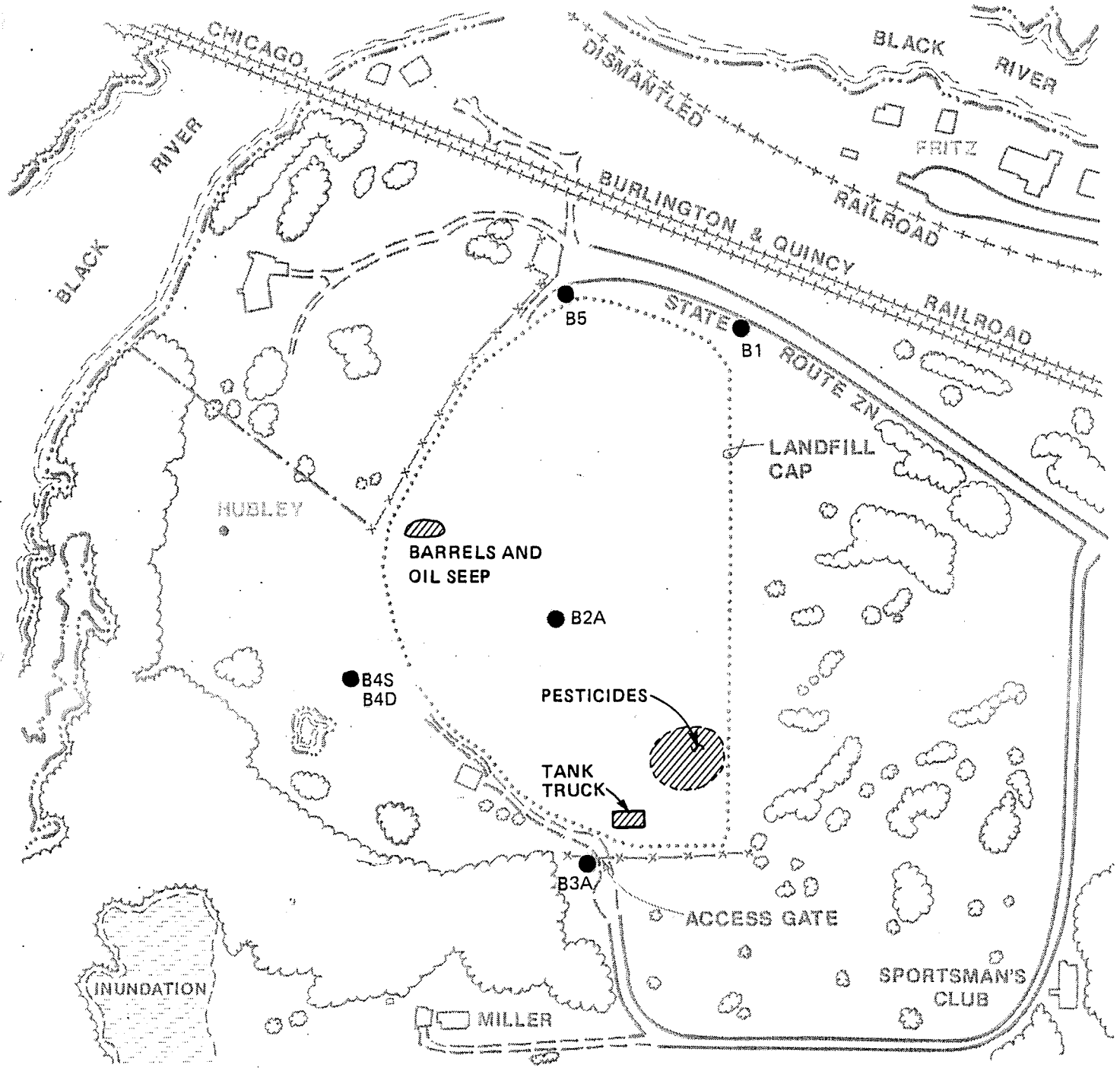


FIGURE 2-5
POTENTIAL WASTE BURIAL LOCATIONS
ONALASKA LANDFILL QAPP

Waste Description

Review of the existing records suggests Outers and Metallics may have delivered at least two kinds of naphtha to the site--high-flash naphtha and VM&P or Stoddard naphtha. The 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 are slightly more volatile and are derived from petroleum. 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 overall as general solvents. Both the petroleum and coal derived naphthas are less dense than water and would float on the water table if the waste reached the aquifer. Some of the organic compounds detected in the groundwater from past analyses may be derived from the naphtha wastes floating on the water table. The liquid naphtha waste could generate a complex mixture of dissolved organic compounds in groundwater over a period of time. The two 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 the degradation products consist of aliphatic and aromatic carboxylic acids, toluene, and other complex mixtures of aromatic and aliphatic hydrocarbons. Adding to the contaminants, the naphtha solvents will also contain constituents derived from the process for which they were used, including metal particles and paint and ink residues.

Barium has been detected in the groundwater at elevated concentrations. Sources other than naturally occurring barium are unknown.

SITE ENVIRONMENTAL QUALITY

Existing Groundwater Quality

Inorganic and organic analytical data are available for six monitoring wells installed on the site by the Town of Onalaska in 1978 and five residential wells near the site. The wells are all completed in the sand and gravel aquifer. The monitoring wells are screened near the water table and the residential wells are probably shallow sand points. Three of the residential wells and most of the monitoring wells have been sampled quarterly since 1978. Analysis was done for indicators of inorganic contamination and included

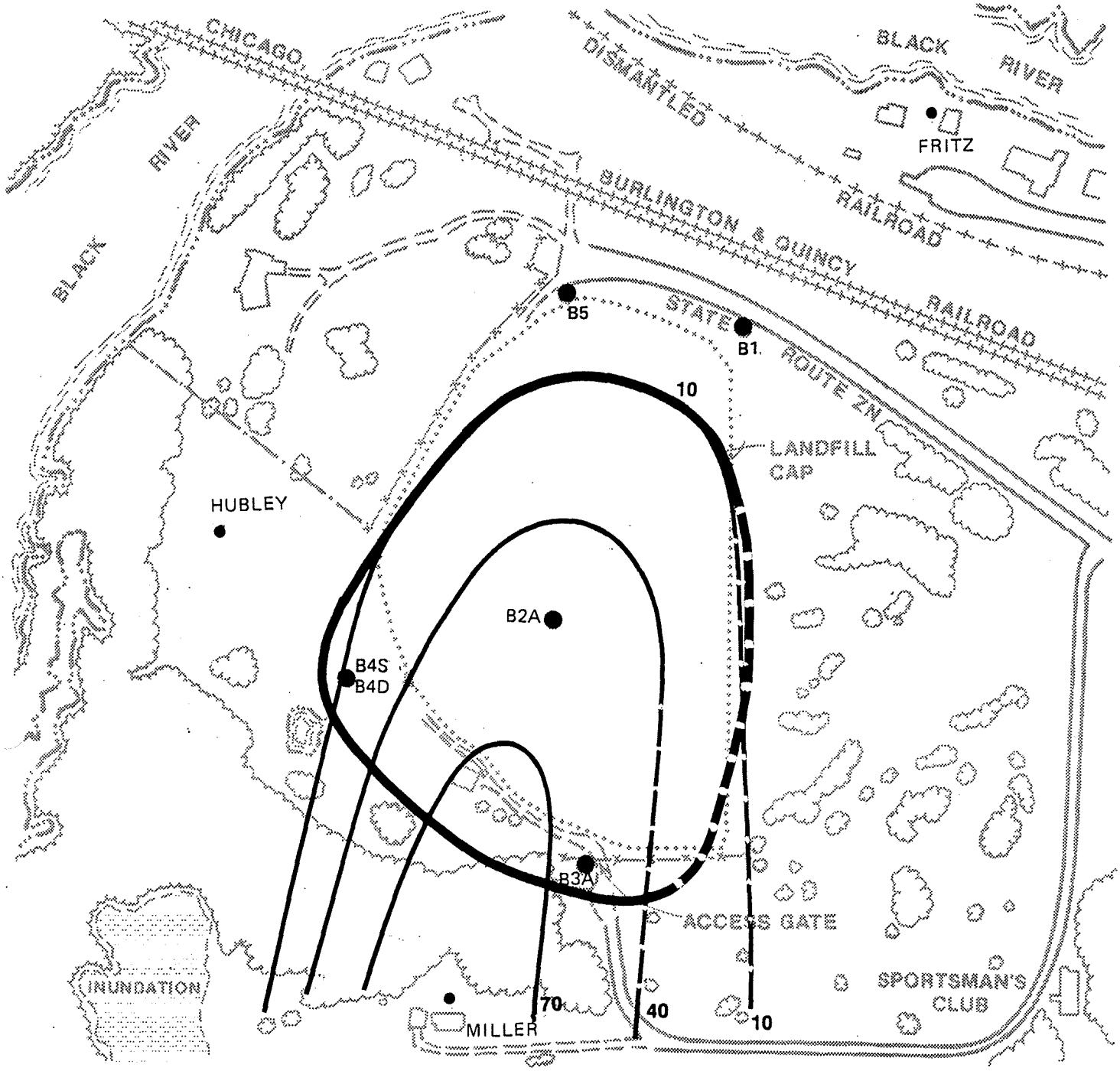
chloride, iron, manganese, alkalinity, hardness, and conductivity. Analysis for organic contamination included COD, 1,1-dichloroethene, 1,1-dichloroethane, 1,1,1-trichloroethane, trichloroethene, perchloroethene, toluene, ethylbenzene, and xylene.

The wells were sampled by DNR personnel, Waryzn Engineering, and Davey Labs. Analytical laboratories have included Davey Labs, Thompson Labs, and Wisconsin State Lab of Hygiene. Evaluation of the data is intended to provide an overview of existing groundwater quality and to help formulate a conceptual model of the sources and pathways of contaminant migration to aid in the further sampling locations.

Spatial and temporal trends in chloride concentrations provided the clearest indication of contaminant migration. Figures 2-6 through 2-8 present contours of chloride concentrations from 1978 to 1988 based on results of the analyses from the six monitoring wells and the Fritz, Hubley, and Miller wells. The contouring suggests that in 1978 the chloride concentration was only slightly elevated onsite relative to the general background level of about 5 mg/l (as found in the upgradient Fritz well). In 1980 the concentration of chloride is shown to have generally increased and a plume of elevated chloride is migrating offsite to the south. The 1982 and 1984 data show chloride levels diminishing onsite as the plume migrates to the south. The site was capped from 1980 to 1982 and is reflected by the diminishing chloride concentrations onsite in the groundwater. Figure 2-8 presents the average of the three most recent samples available, two samples in March and September of 1986 and one sample in March of 1988. The contours show continued decrease in chloride levels onsite and south of the site as the chloride disperses and migrates south. The calculated average groundwater velocity of 80 feet/year presented earlier corresponds well to the migration of chloride seen in these figures.

Additional discussion of inorganic and organic contamination is presented in Section 2 of the Work Plan. The existing groundwater quality data is presented in Appendix A of the Work Plan.

The following observations have been made on the basis of evaluation of the existing groundwater data.



LEGEND

- MONITORING WELL LOCATIONS
- RESIDENTIAL WELL LOCATION
- 1978
- 1980
- INFERRED CONTOURS

FIGURE 2-6
GROUNDWATER CHLORIDE CONTOURS (mg/l) 1978-1980
ONALASKA LANDFILL QAPP

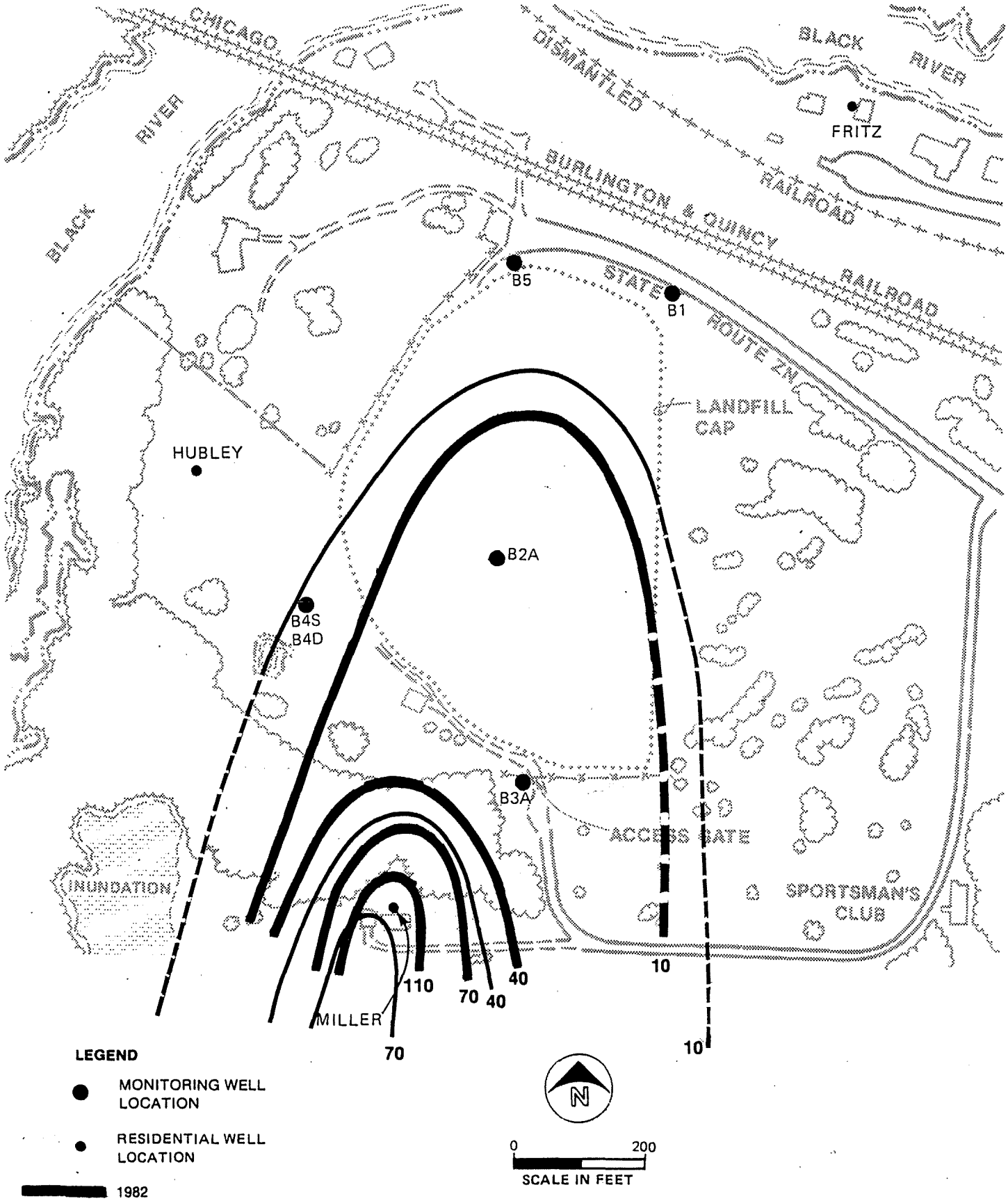


FIGURE 2-7
GROUNDWATER CHLORIDE CONTOURS (mg/l) 1982-1984
ONALASKA LANDFILL QAPP

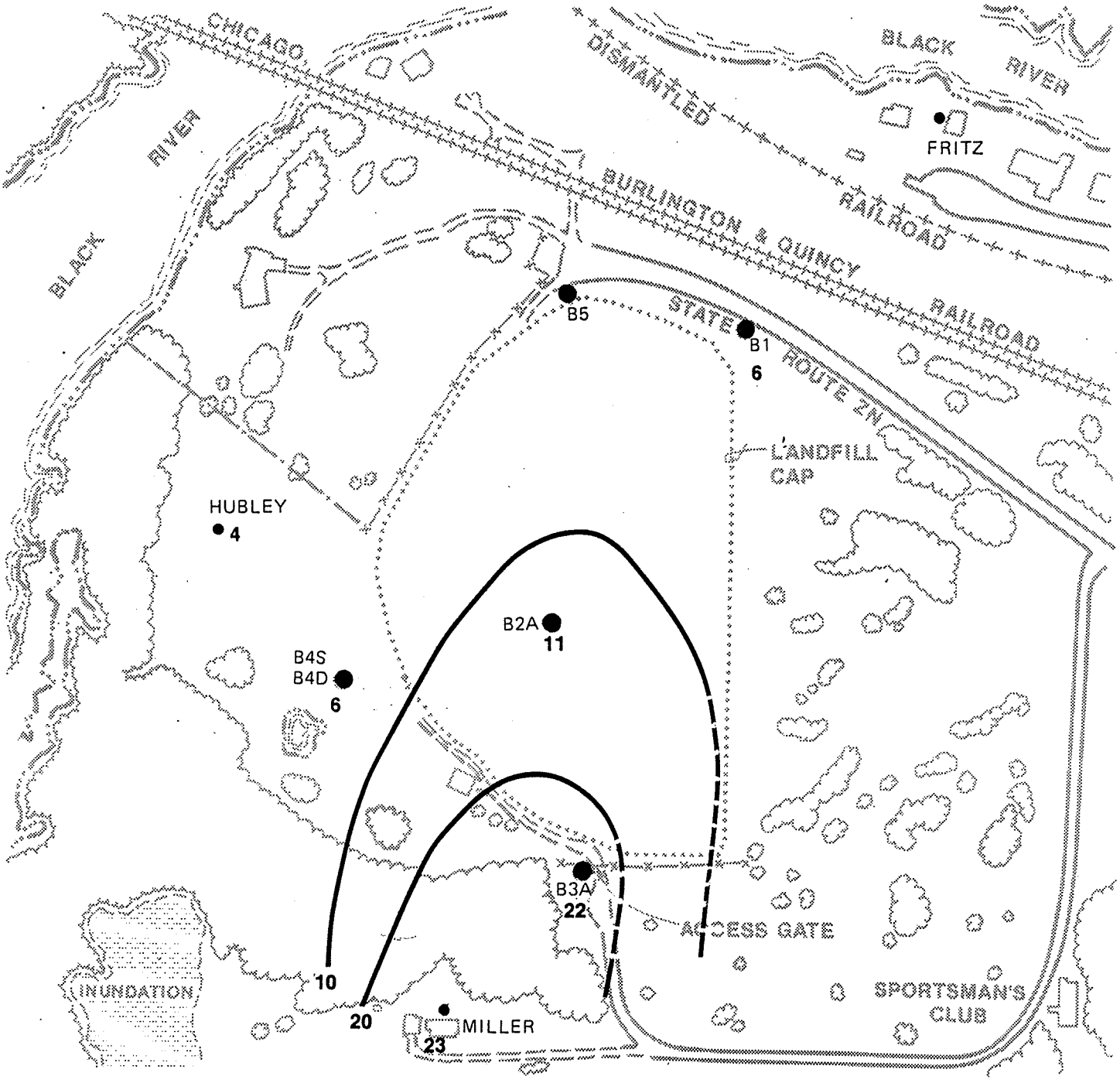


FIGURE 2-8
GROUNDWATER CHLORIDE CONTOURS (mg/l) 1986-1988
ONALASKA LANDFILL QAPP

- Concentrations of chloride, barium, iron, and manganese are above background both on and immediately south of the site.

- The migration of chloride and measurements of conductivity over time appear consistent with the calculated average groundwater direction and velocity of 80 feet/year to the south-southwest.

- Capping the site in 1982 appears to have resulted in diminished concentrations of chloride, total dissolved solids (as measured by conductivity) and oxidizable organics (as measured by COD) in groundwater beneath and immediately south of the site.

- Barium continues to exceed the primary MCL in groundwater onsite and immediately south of the site. Iron and manganese greatly exceed secondary MCLs in groundwater onsite.

- VOCs were routinely detected in wells B4S, B4D, B3A, and the Miller well. Concentrations in B4S were orders of magnitude greater than the other wells.

PRELIMINARY RISK ASSESSMENT

The preliminary risk assessment is based on available data for the Onalaska site. It identifies potential contaminants of concern based on the existing data and identifies potential exposure pathways based on current knowledge of site characteristics and waste/contaminant characteristics. It also compares existing environmental concentrations to standards and criteria and estimates the risk associated with those levels. The preliminary risk assessment is summarized here. More detail can be found in Section 2 of the Work Plan.

The major potential exposure pathways associated with the site are:

- Release of contaminants to the groundwater, contaminant migration through the groundwater, and use of the groundwater as a drinking water source

- Release of contaminants to the groundwater, contaminant migration through the groundwater, discharge of the groundwater to the Black River or

Lake Onalaska, and the exposure of fish and wildlife in the Upper Mississippi Wildlife and Fish Refuge

- Erosion of the cap and exposure of landfill contents, leading to the release of contaminants to the air and exposure of nearby residents

- Erosion of the cap and exposure of landfill contents, leading to the exposure of trespassers on the site

The only exposure pathway that can be quantitatively assessed at this time is the release of contaminants to groundwater with subsequent use of the groundwater as a water supply. Other pathways cannot be addressed for lack of data.

Contaminant concentrations detected in the groundwater were compared to drinking water standards and criteria using the last three sampling rounds in each monitoring well and the Miller well. The Miller well and monitoring wells 2A, 3A, 4S and 4D had contaminants exceeding criteria or standards. Monitoring well B4S had the greatest number of chemicals exceeding criteria or standards. Contaminants exceeding criteria or standards included barium, trichloroethene, 1,1-dichloroethene, 1,1,2,2-tetrachloroethane, 1,1,1-trichloroethane, ethylbenzene, xylene, and toluene.

The risks associated with drinking water containing contaminants were also evaluated. The risks are based on the highest levels detected in each of the last three sampling rounds. Cancer risks associated with the highest levels detected in the monitoring wells range from 1×10^{-3} to 1×10^{-5}. Reference dose values are exceeded for barium, ethylbenzene, manganese, and toluene.

As the analyses indicate, ingestion of the groundwater from the area south of the site could pose adverse health effects.

TARGET COMPOUNDS

The existing data were reviewed in proceeding sections on hazardous materials characterization and existing groundwater quality. The review helps suggest some of the potential chemicals of concern at the Onalaska site.

WASTES TYPES DISPOSES

The hazardous materials characterization identified the following specific chemical waste types as having been disposed of at the site:

Petroleum/coal tar derivatives

- VM&P naphtha
- High-flash naphtha
- Stoddard Solvent
- Mineral spirits
- Asphaltum

Other specific wastes

- Toluene
- Solvosol
- PTL-1009
- Transformer fluids

Most of the waste materials listed above consist of mixtures of chemicals. Chemicals typically identified with these waste types include:

- Saturated aliphatic hydrocarbons (paraffins) in the C5 to C14 range: VM&P naphtha, Stoddard Solvent, mineral spirits, high-flash naphtha

- Alicyclic hydrocarbons (cycloparaffins/naphthenes such as cyclohexane): VM&P naphtha, Stoddard Solvent, mineral spirits, high-flash naphtha

- Olefins (alkenes): mineral spirits, such as propene

- Alkylbenzenes (aromatic hydrocarbons such as toluene, xylene, ethylbenzene): VM&P naphtha, Stoddard Solvent, mineral spirits, and high-flash naphtha as sources of ethylbenzene, toluene, and xylene; waste toluene as a source of toluene

- Benzene: VM&P Naphtha, Stoddard Solvent, mineral spirits, and high-flash naphtha

- Polycyclic aromatic hydrocarbons: asphaltum

- Ethylene glycol monoethylether: solvosol

- Polychlorinated biphenyls (PCBs): transformer fluids

Other nonspecific wastes were identified and include:

- Paint and ink residues
- Water soluble degreasers
- Cutting oils
- Gun oil/gun cleaning solvents
- Pesticides

These waste categories cover a variety of waste types, so it is not possible to assign specific contaminants to them. Some general category might be discussed, however. Paint and ink residues could be a source of metals (used as pigments) and solvents. Cutting oils may include mineral and lard oil, sulfur compounds, chlorinated organic compounds, and additives such as formalin, mercurials, and phenols. Pesticides were reportedly disposed of at the site and may include DDT.

CHEMICALS DETECTED AT THE SITE

Monitoring has been performed for a limited set of chemicals. Chemicals detected in the groundwater include:

1,1-Dichloroethene	1,1,2,2-Tetrachloroethane
1,1-Dichloroethane	Toluene
1,1,1-Trichloroethane	Ethylbenzene
Trichloroethene	Xylene
Barium	Iron
Manganese	

Toluene, ethylbenzene, and xylene are consistent with the waste types identified as having been disposed of at the site. The other organic compounds are chlorinated ethane and ethenes and are constituents of widely used industrial materials such as degreasers.

Based on the foregoing discussion, the following target compounds will be analyzed (see Appendix A for specific compounds and detection limits):

- Volatile organic compounds
- Semivolatile organic compounds
- Pesticides and PCBs
- Inorganic chemicals

PROJECT OBJECTIVES

The objective of the RI for the Onalaska Landfill site is to provide data for selection of a remedy. To meet the objectives, the overall goals of the RI are to:

- Collect legally defensible data to support and complete a quantitative risk assessment of the site and to determine if remedial actions are warranted. Based on the preliminary risk assessment, it is assumed an FS will be performed.
- Approximate the size, extent, and characteristics of contaminant source areas for evaluating present and future releases.
- Identify and evaluate potential contaminant pathways to select remedial alternatives that will control, contain, or remove groundwater contaminants.
- Collect data used in the selection and evaluation of remedial alternatives that will contain, remove, treat, or destroy contaminant sources.

DATA QUALITY OBJECTIVES

Based on the evaluation of existing data, objectives are presented for the data type and quality needed to identify threats to the public health and environment and to develop and evaluate remedial action alternatives. Data quality objectives are presented here for each component of the source-pathway-receptor model of the site and for evaluation of alternatives.

SOURCES

Data will be obtained in the RI to help locate the cache of 300 buried drums, the 500-gallon tank truck, and the area where solvent dumping occurred. The integrity of the drums will be determined if they are found. If the solvent disposal area is found, the physical dimensions of the area and the degree of contamination will be defined. Field investigations to meet these objectives include geophysical investigations, a soil gas survey, and test pits.

The preliminary evaluation of remedial actions presented in Section 3 of the Work Plan concluded that locating other sources such as drums containing chemicals disposed of

throughout the landfill was not necessary since removal or treatment were not remedial actions likely to survive alternative screening.

NATURE AND EXTENT OF RELEASED CONTAMINANTS

Data to identify the type and magnitude of contamination in groundwater, surface water, and sediments will be obtained in the RI. Groundwater data will be obtained to identify three types of contaminant plumes: 1) a plume containing concentrations of inorganic chemicals and organic compounds sufficient to require substantial treatment if collected (e.g., precipitation, biological treatment processes); 2) a pure phase naphtha plume floating on the water table; and 3) a plume containing dissolved inorganic chemicals and organic compounds at levels above health-based criteria for drinking water but requiring less intensive treatment if collected (e.g., air stripping). Sampling and analysis of groundwater, surface water, sediment, and soil gas and geophysical investigations have been identified as field investigations to meet these objectives.

A close support laboratory (CSL) will provide screening analysis for target VOCs and COD. The rapid analytical turnaround available in the CSL will allow boring and monitoring well location to be selected based on earlier CSL results. The objective is to delineate the three plumes in one phase of field work. The CSL results will also allow judicious selection of samples to be sent to the CLP for analysis.

POTENTIAL PATHWAYS

Data obtained in the foregoing tasks will provide much information on the pathways of contaminant migration. In addition, data will be obtained on the surface topography, the subsurface stratigraphy, and the groundwater flow system sufficient to characterize the existing and potential future pathways to groundwater users and the surrounding surface waters. A topographic survey, an investigation of the effectiveness of the cap, and a hydrogeologic investigation will be performed to obtain the necessary data.

POTENTIAL RECEPTORS

Data on the location and number of groundwater users in the area and on land use patterns will be obtained as part of the risk assessment. Existing information on aquatic biota in the region will also be obtained during the risk assessment.

EVALUATION OF ALTERNATIVES

Data obtained in the foregoing tasks will be used to develop and evaluate remedial action alternatives. Additional data will be obtained in the RI to evaluate the remedial action alternatives and the permeability, thickness, and long-term effectiveness of the existing cap. Groundwater samples collected to determine the extent and magnitude of contamination will also be analyzed for conventional parameters to allow assessment of groundwater treatment alternatives. Soil samples from test pit investigations of the solvent disposal area will be analyzed for parameters important in evaluating soil treatment using incineration.

The data generated and collected during the RI will be used to achieve the goals and objectives discussed above. Table 2-4 addresses each RI field activity, lists the anticipated data generated from each task, and correlates the intended data usage as it relates to the project objectives.

SUMMARY

The following objectives have been identified for the Onalaska site:

- Determine the permeability of the existing cap.
- Evaluate the susceptibility of the cap to damage from freezing and drying.
- Determine the location, extent, and magnitude of the main drum disposal area and the location of the buried tank truck.
- Map the subsurface conductivity plume extending south of the landfill if sufficient conductivity differences occur.
- Locate the solvent disposal area.
- Evaluate the integrity of the drums buried in the drum disposal area.
- Determine the degree of contamination in the unsaturated subsurface soils of the solvent disposal area.

Table 2-4 (Page 1 of 2)
INTENDED DATA USAGE

	Fieldwork		Laboratory Analytical Work	
Task/Field Activity	Field Test and Measurement	Intended Data Usage	Laboratory Analysis	Intended Data Usage
Topographic Survey	Aerial topographic site survey Ground survey check	Develop a base map Determine existing grades and contours to upgrade cap	Determine cap cross section	
Cap Investigation	Test pits Infiltrometer test Nuclear density test Shelby tube samples HNu/OVA screen	Obtain samples for geotechnical analyses Measure in situ permeability Measure in situ density Soil Characterization	Geotechnical parameters: - Atterberg limits - moisture content - grain size - shrinkage limits - moisture-density relation test - flexible wall permeability test Shelby tube samples analyzed for: - in situ density - moisture content - flexible wall permeability test	Estimate infiltration capacity of existing cap Establish compaction characteristics of soil Evaluate susceptibility to freeze/thaw damage Evaluate remedial alternatives Determine present effectiveness of cap
Geophysical Investigation	Magnetometer survey Electromagnetic survey	Determine location, extent and relative magnitude of disposal area Locate buried tank truck Identify magnitude and extent of groundwater conductivity plume Locate solvent disposal area		
Solvent Disposal Area Investigation	Soil Gas Survey	Obtain samples for CSL analyses	Close support laboratory: - indicator VOCs	Determine required sampling depth Analyze headspace gases Select samples for CLP analyses Evaluate extent of pure phase naphtha migration Delineate location of solvent disposal area
	Test Pits	Evaluate competency of buried drums Obtain samples for CLP analyses	Close support laboratory - indicator VOCs CLP: - TCL organics and inorganics - incineration parameters - toxicity characteristics	Evaluate degree of contamination in unsaturated subsurface soils in the solvent disposal area Evaluate treatment alternatives

	Fieldwork		Laboratory Analytical Work	
Task/Field Activity	Field Test and Measurement	Intended Data Usage	Laboratory Analysis	Intended Data Usage
Hydrogeologic Investigation	Geotechnical borings - borehole logging - HNu/OVA screen	Determine need/depth of monitoring wells Define subsurface stratigraphy Identify potential migration pathways of released contaminants Allow collection of groundwater samples for CSL analyses	Geotechnical parameters: - grain size - Atterberg limits	Delineate extent of VOC and naphtha plumes Determine need/depth of monitoring wells Soil classification
	Groundwater samples - HNu/OVA screen		Close support laboratory: - selected VOCs	
	Monitoring well installation and monthly water level measurement	Better define potentiometric relationships Evaluate extent of groundwater contamination Determine groundwater flow system		
	Subsurface soil sampling - HNu/OVA screen		Geotechnical parameters: TCL organics and inorganics	Generate data on the physical properties of the local subsurface soils for predictions of contaminant migration Determine magnitude of subsurface soil contamination Detect organic vapors
	Pump test	Determine aquifer characteristics sufficient for evaluation and design of groundwater collection systems		
	Monitoring well sampling - HNu/OVA screen		Close support laboratory TCL organics and inorganics	Determine need/placement of monitoring wells. Determine extent and magnitude of groundwater contamination
Environmental Sampling	Residential well sampling	For laboratory analysis	TCL organics and inorganics	Determine whether groundwater contamination has migrated to residential wells and determine risk to users.
	Surface water and sediment sampling - HNu/OVA screen		Close support laboratory TCL organics and inorganics	Determine need/location for additional surface water and sediment sampling Determine extent and magnitude of surface water and sediment contamination

- Obtain data for evaluation of soil incineration and offsite disposal material from the solvent disposal area.
- Determine the extent of the floating naphtha outside the landfill.
- Confirm or refine the conceptual model of the groundwater flow system in relation to the underlying hydrostratigraphy and adjacent Black River.
- Identify the number and location of groundwater receptors and identify aquatic receptors.
- Evaluate the magnitude and extent of groundwater contamination attributable to the landfill and determine analytical parameters important for evaluation of groundwater treatment.
- Determine the magnitude of contamination in the saturated zone within the soil groundwater plume.
- Evaluate the aquifer properties and the aquifer's response to pumping.
- Determine whether site contaminants have migrated to residential wells.
- Evaluate whether site contaminants have migrated to surface water and sediment.

SAMPLE NETWORK DESIGN AND RATIONALE

Sample network design and rationale are discussed for each field investigation subtask identified below.

TOPOGRAPHIC SURVEY

The purpose of this subtask is to produce a topographic survey and base map of the landfill site. Existing grades and contours must be known before remedial actions involving the upgrade of the existing cap can be developed. An aerial topographic survey of the site and surrounding area will be conducted. The aerial survey will be field checked by a ground survey crew. The site topographic map covering 14 acres of the site and surrounding area will consist of contour lines on 1-foot intervals and use a scale of 1 inch equals 30 feet. A topographic map with 2-foot contour

intervals and a scale of 1 inch equals 100 feet will be developed for a broader area of 145 acres and include the surrounding surface waters, mostly west and south of the site.

CAP INVESTIGATION

The objectives of the cap investigations are to:

- Determine the permeability of the existing cap
- Evaluate the susceptibility to damage from freezing and drying

These investigations will aid in the predictions of contaminant migration to the groundwater and allow evaluation of the long-term effectiveness of the existing cap in reducing contaminant migration.

Permeability tests performed on the undisturbed (Shelby tube) samples and infiltrometer tests will be used to determine the effectiveness of the existing cap. Results of permeability and density tests on undisturbed and remolded samples will be compared to determine the susceptibility of the cap soil to damage from freezing and drying. Characterization and permeability testing will also be used to support evaluation of remedial alternatives, such as construction of a multilayer cap.

The following elements are intended to achieve the objectives.

A maximum of 11 test pits will be dug at locations shown in Figure 2-9 to establish the cross section of the cap. The pits will be dug by hand or with a narrow bucket backhoe to a depth of about 2 feet. An infiltrometer will be used to measure in situ permeability as each pit is being dug.

A nuclear density gauge will be used to determine in situ density and moisture contents at various locations across the site. The number and locations of the nuclear density tests will be determined in the field.

Samples from the test pits will be analyzed for grain size, Atterberg limits, and shrinkage limits through SAS requests. One moisture-density relation test will be performed using a soil sample taken from a representative test pit. A flexible wall permeability test will be performed on a remolded sample, compacted to 95 percent maximum dry density at the optimum moisture content.

Shelby tube samples will collected at each test pit location. The Shelby tubes will be pushed using the drill rig that is needed for the hydrology/hydrogeology investigation. If the characterization tests performed on the test pit samples indicate markedly different soil types, additional Shelby tube samples will be necessary. Shelby tube samples will be analyzed for in situ density and moisture. Flexible wall permeability tests will be performed on samples taken from each Shelby tube.

After the initial stage of geotechnical investigation and sampling is completed, the results will be evaluated to determine whether more field work is needed. Results of the permeability tests will be reviewed along with composition tests. The following additional tasks may be necessary to evaluate capping alternatives in the feasibility study:

- Additional permeability tests on locally available soils or onsite soil-bentonite clay mixtures may be necessary if the existing cover is highly permeable.

- Test patches may need to be constructed using the proposed cover material to determine the feasibility of achieving the desired relative compaction. Compaction over the landfill may be an issue because of potential problems with the soft (refuse) subgrade.

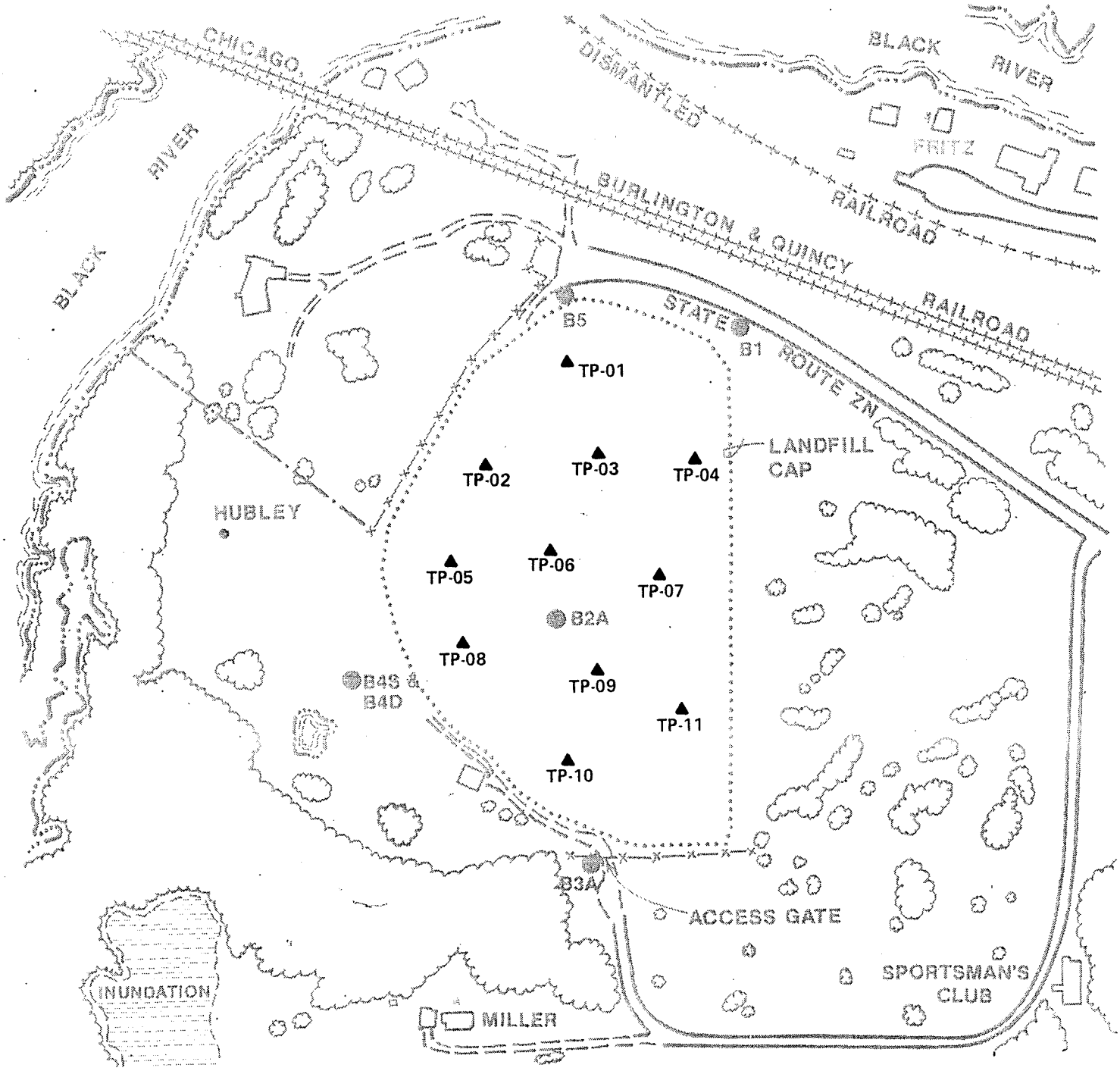
Geotechnical laboratory procedures and equipment will require close monitoring. Specifications for each test will be prepared and included as part of the drilling subcontract. The drilling subcontractor will be responsible for retaining a laboratory capable of conforming to the specifications. A CH2M HILL geotechnical engineer will visit the laboratory at least once to review the procedures and equipment being used.

GEOPHYSICAL INVESTIGATION

The objectives of the geophysical investigations are:

- To determine the location, extent and magnitude of the main drum disposal area, and the location of the buried tank truck

- To map the groundwater conductivity plume extending south of the landfill if sufficient conductivity differences occur



LEGEND

▲ SHALLOW TEST PIT LOCATIONS

FIGURE 2-9
CAP INVESTIGATION LOCATIONS
ONALASKA LANDFILL QAPP

- To locate the solvent disposal area if sufficient conductivity differences occur

Magnetometer Survey

A magnetometer survey will be conducted to determine the location and size of the main drum disposal area and to locate the buried tank truck. A 100- by 40-foot grid will be laid out over an area of about 12 acres (Figure 2-10). A magnetometer base station will also be established to monitor diurnal changes in the magnetic field (for correction purposes). Once the grid and base station have been located, magnetometer readings will be collected at 20-foot centers using EDA's OMNI PLUS magnetometer/ gradiometer. Readings made at locations not marked by a grid flag will be located by positioning a marked tape or rope along the appropriate line.

Upon completion of the magnetometer survey, data will be corrected for the effects of the diurnal changes in the local magnetic field. Once this has been done, a magnetic contour map will be prepared to aid in the interpretation of magnetic anomalies.

Electromagnetic Survey

The purpose of the electromagnetic survey is to identify the relative magnitude and extent of the groundwater conductivity plume migrating from the landfill and to locate the solvent disposal area. Conductivity variations in soils or fills may limit the ability to distinguish the solvent disposal area or a conductivity plume onsite. Data will be collected on 40-foot centers at 20- and 10-meter coil separations over an area of about 26 acres (Figure 2-10). If the conductivity plume is found to extend beyond that area, the survey will be continued to the south with a wider grid spacing. The different coil separations will help identify whether conductivity variations are due to shallow or deep sources. The data will then be plotted and contoured to describe the location of the solvent disposal area and the conductivity plume south of the landfill.

SOLVENT DISPOSAL AREA INVESTIGATION

The objectives of the solvent disposal area investigation are:

- To locate the major solvent disposal area within the landfill and to evaluate the degree of

contaminantion in the unsaturated subsurface soils of the area

- To obtain data for the evaluation of soil incineration and offsite disposal
- To determine the extent of the floating naphtha outside (downgradient of) the landfill

The following investigation is intended to achieve the objectives.

Soil Gas Survey

If the geophysical survey does not locate the major solvent disposal area and naphtha plume, a soil gas survey will be performed to locate them. The soil gas survey will be centered approximately 200 feet north of monitoring well B4S and B4D (Figure 2-11). Existing data indicate that is the location of the solvent disposal area. A soil gas survey and CSL analyses of the soil gas for a few selected VOCs may provide immediate information of the lateral extent of soil and groundwater contamination. The survey will also help minimize the number of geotechnical borings and monitoring wells to be drilled or installed.

Soil gas ground probes will be used for sampling. The stainless steel gas probes are 5/8-inch O.D. and 2.5 feet long. The lower section is vertically slotted to allow soil gas to be drawn into the probe. The probe is driven into the ground using an electrically operated hammer. Once the probe is in place, Teflon tubing is connected and air is withdrawn with an electrically operated air pump. After air purging for 2 minutes, a sample is collected onto a Supeleo Carbotrap. The Carbotrap is then thermally desorbed and analyzed in the CSL by GC/FID.

Samples will be analyzed in the onsite CSL using a Hewlett Packard Model 5890 gas chromatograph with ECD and FID detectors. Samples will be analyzed for up to five selected target VOCs including 1,1-dichloroethene, trichloroethene, 1,1,1-trichloroethane, toluene, and xylene.

Vertical profiles of organic gases present in the soil pore spaces will be measured and plotted for several locations. One will be alongside monitoring well B4S. Based on the vertical profiles a sampling depth of at least 4 feet will be selected. Sampling probe depth within the landfill may be limited by the presence of buried drums. Once the

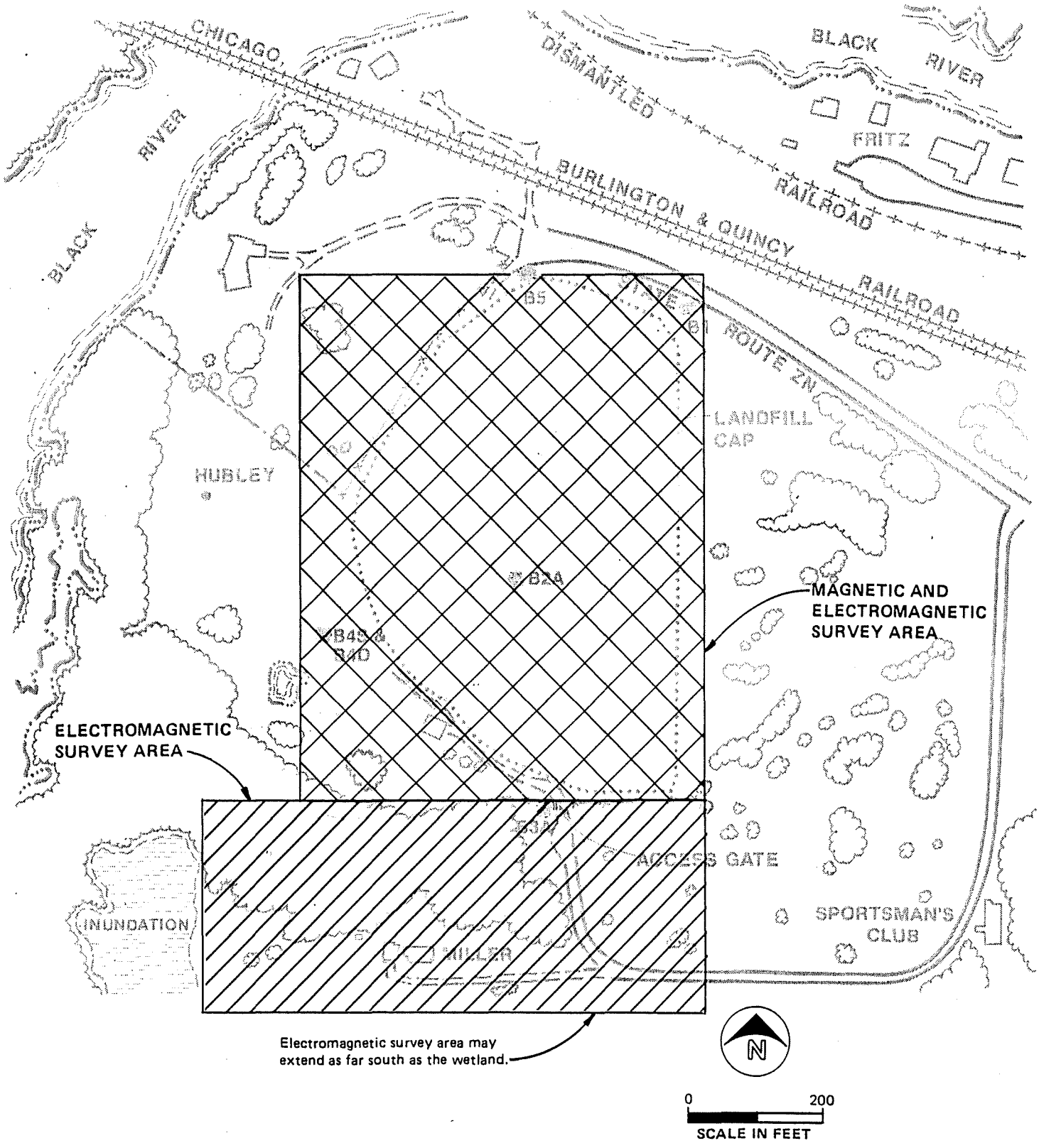


FIGURE 2-10
GEOPHYSICAL SURVEY AREAS
ONALASKA LANDFILL QAPP

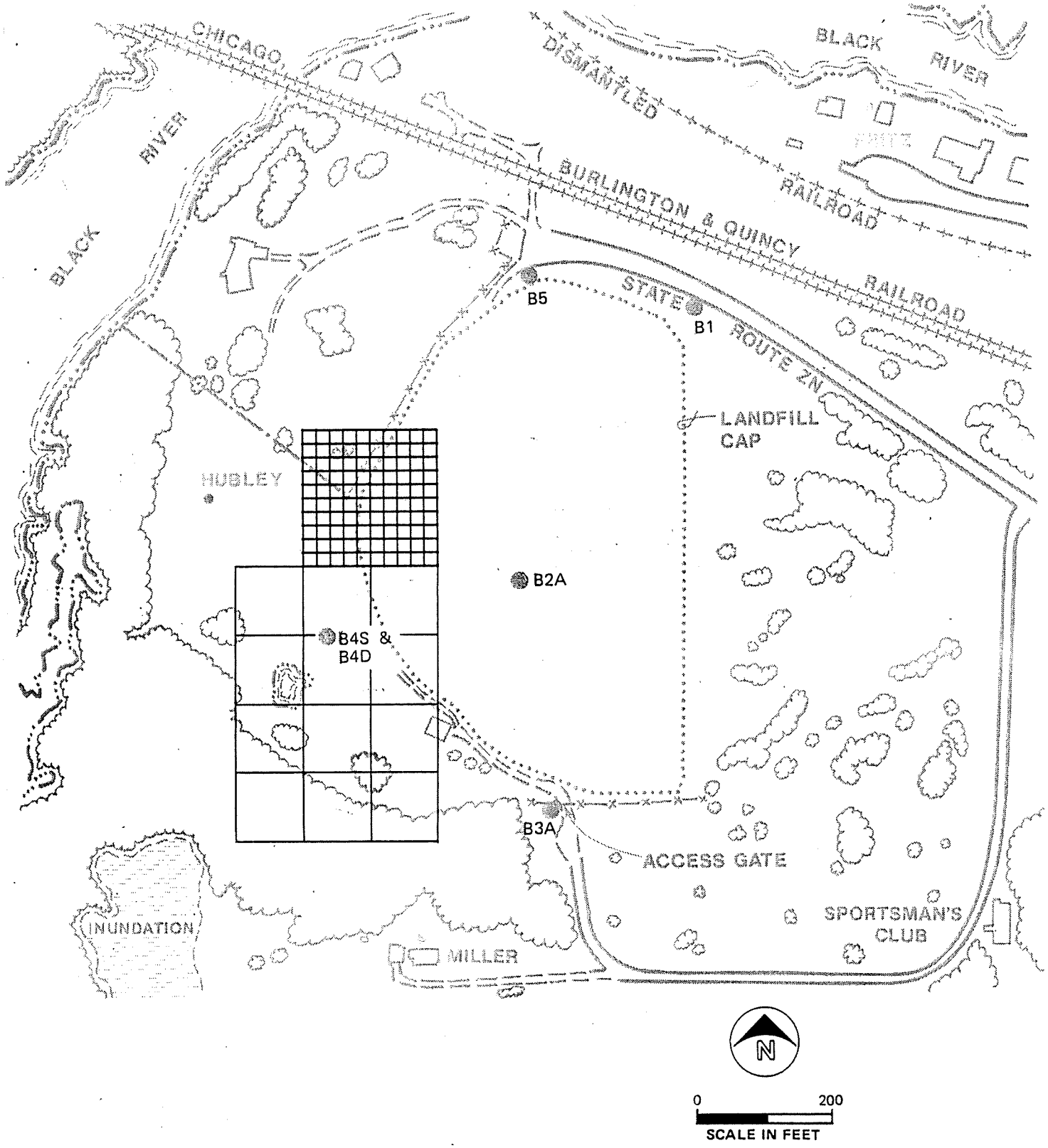


FIGURE 2-11
SOIL GAS SURVEY GRID
ONALASKA LANDFILL QAPP

constant sampling depth is established, soil gas samples will be collected across a grid. If the initial vertical profiles do not provide sufficient data to locate the naphtha plume, the soil gas survey will be discontinued.

Samples will be collected on a 20- by 20-foot grid laid out over an area measuring 200 by 200 feet. Initially, samples will be collected nearest the suspected disposal location. Analytical results will be plotted on site maps so that the areal trends in the selected VOCs can be identified. If an area of distinctly higher VOCs is identified, sampling will be done on a 10- by 10-foot grid to determine the limits of the area. A maximum of 80 soil gas samples will be taken. An additional maximum of 20 soil gas samples will be taken on a 100- by 100-foot grid to identify the extent of the floating naphtha south of the disposal area.

Test Pits

Review of the site history indicated the major drum disposal area and the solvent disposal area may be adjacent to each other. Three test pits will be excavated in the drum disposal area and solvent disposal area to a depth of 15 feet. The objective of the test pit investigation is to: 1) evaluate the integrity of the drums, and 2) determine the degree of contamination in the unsaturated subsurface soils in the solvent disposal area.

Subsurface soil samples will be collected every 2 feet while the pits are excavated and headspace gases will be analyzed by the onsite CSL for indicator VOCs. Based on the laboratory results and visual observations, a maximum of ten subsurface soil samples will be collected for analyses of organic compounds and inorganic chemicals. Total organic carbon (TOC), total organic halides (TOX), ash, sulfur, Btu content, and EP toxicity will also be analyzed for evaluation of soil incineration and offsite disposal.

HYDROGEOLOGICAL INVESTIGATION

The objectives of the hydrogeological investigation are to:

- Confirm or refine the conceptual model of the groundwater flow system in relation to the underlying hydrostratigraphy and the Black River
- Evaluate the magnitude and extent of groundwater contamination attributable to the landfill and determine analytical parameters important to evaluation of groundwater treatment

- Determine the magnitude of soil contamination in the saturated zone within the groundwater plume
- Evaluate aquifer properties and the aquifer's response to pumping

The following investigation is intended to achieve the above objectives.

Geotechnical Borings

Soil borings will be drilled and sampled to confirm or refine the conceptual model the subsurface stratigraphic relationships and delineate the extent of the VOC and naphtha plume near the landfill. The rationale and proposed depth of each boring shown in Figure 2-12 is presented in Table 2-5. If the stratigraphy is more complex or the groundwater contamination is more extensive than that presented in the evaluation of existing data, a maximum of 16 more geotechnical borings may be required in this phase of field sampling. All borings will be advanced using a 4.25-inch (I.D.) screened hollow-stem auger.

Two soil borings will be advanced to bedrock, about 135 feet below ground. The other six borings will be advanced about 70 feet to determine the stratigraphy of the sediments beneath the south portion of the landfill and south of the landfill near the potential migration pathways.

Each geotechnical boring will be sampled at 5-foot intervals using a standard split-spoon sampler following ASTM Standard D 1586 for the Standard Penetration Resistance Test. Boreholes where monitoring wells are not installed (four estimated) will be abandoned by injecting a thick bentonite slurry from the bottom of the borings to the ground surface using the tremie method.

Each boring will be logged by an experienced CH2M HILL hydrogeologist or geotechnical engineer. Samples will be described using the Unified Soil Classification System terminology. Samples will be collected for grain-size analysis or Atterberg limits based on changes in stratigraphy. The decision to submit a sample for geotechnical analysis will be made in the field by the supervising geologist or engineer but in no case will exceed three samples per boring. All samples will be screened for VOCs using an HNu or OVA. Up to ten samples from below the water table will be selected on the basis of visual

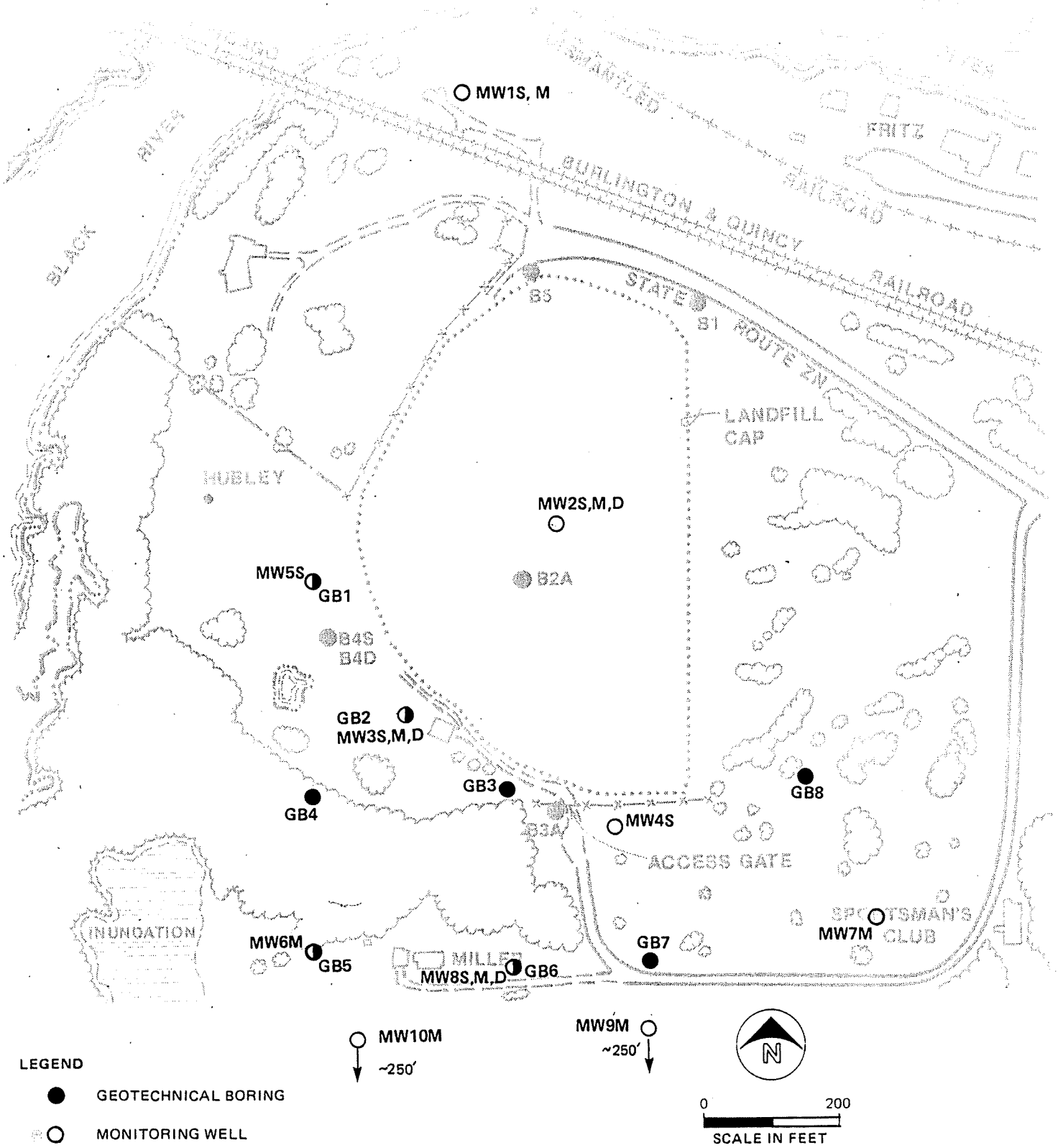


FIGURE 2-12
GEOTECHNICAL BORINGS
ONALASKA LANDFILL QAPP

Table 2-5
RATIONALE FOR SOIL BORING LOCATIONS

Boring Location	Proposed Depth	Rationale
GB-1	Bedrock	o To help determine stratigraphy on west side of site where data is scarce, and to help locate upgradient monitoring well nest
GB-2	70 Feet	o To help determine stratigraphy in SW portion of the site where data is scarce, and to help locate upgradient monitoring well nest
GB-3	70 Feet	o To help determine location of downgradient monitoring well nest
GB-4	70 Feet	o To help determine stratigraphy in SW corner of site where data is scarce, and to help locate downgradient monitoring well nest
GB-5	Bedrock	o To help determine stratigraphy of potential migration pathways and extent of contamination, and to help locate monitoring wells
GB-6	70 Feet	o To help determine stratigraphy of potential migration pathways and extent of contamination, and to help locate monitoring wells
GB-7	70 Feet	o To help determine downgradient stratigraphy and extent of contamination, and to help locate monitoring wells
GB-8	70 Feet	o To help determine stratigraphy in SE portion of the site where data is scarce

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observation and screening results for CLP analysis of organic and inorganic contaminants and TOC.

To allow delineation of the groundwater contaminant plume in one phase of field investigation, groundwater samples will be collected from within the hollow-stem auger and analyzed in the CSL for the five target VOCs. Groundwater samples will be collected at the water table, at mid-depth (about 70 feet below ground) and above bedrock (about 130 feet below ground). The potential exists for groundwater contaminants at elevations above the screened auger to be drawn down during groundwater sampling. This fact will be considered during evaluation of the CSL screening results. The relatively permeable sand at the site may alleviate this problem. The Level 2 screening data for the five target VOCs will be plotted on site plan maps and vertical cross sections. The rapid turnaround of analysis will allow judicious placement of subsequent borings or monitoring wells.

Monitoring Well Installation

To better define potentiometric relationships at the site and evaluate the extent of groundwater contamination, 17 monitoring wells will be installed (see Figure 2-12). The rationale for each location is presented in Table 2-6. The locations may be modified after results of the previous investigations are known. If the horizontal or vertical extent of groundwater contamination is greater than that estimated based on existing data, more monitoring wells may be necessary (a maximum of 13). The need for the wells and their location will be assessed in the field by CH2M HILL in conjunction with EPA's RPM.

Monitoring well nest MW1 will be installed upgradient of the landfill to establish upgradient groundwater quality. Monitoring well B5 is of questionable value for this purpose because of its unsecured cap and the fact that it is silted in above the screen. A second monitoring well nest (MW2) will be installed through the landfill to evaluate groundwater quality and stratigraphy beneath the landfill. Two additional nests are proposed south of the landfill (MW3 and MW8). The locations were selected to help evaluate the vertical distribution of contaminants downgradient of the landfill and to determine if a vertical gradient exists.

At least six monitoring wells will be installed in geotechnical borings immediately upon completion of the borings. The elevations of each monitoring well measuring

point will be determined and water levels recorded. This information is needed to determine the groundwater flow system. Of particular importance is to determine what portion of the year the Black River receives groundwater discharge. The information obtained from completion of this task will be important to the analysis of the fate and transport of constituents released from the Onalaska Landfill and to the identification of contaminant migration pathways.

The boreholes for the monitoring wells will be advanced using screened hollow-stem augers (6.25 inch I.D.). This size allows sufficient annular space between the well and the auger wall to introduce a filter pack and seal. If alternative drilling methods are required, only methods using clean water, air, or a cable tool will be considered.

As with the geotechnical borings, groundwater samples will be collected at up to three depths at each location, water table, mid-depth and above bedrock. Samples will be analyzed in the CSL for the five selected VOCs. Results will be plotted on site maps and cross sections and will be used to locate subsequent monitoring well locations. Samples will be collected from within the hollow stem auger after purging at least five well volumes.

Soil from the screened interval of monitoring wells MW2S, MW2M, MW2D and MW4 will be sampled with a split-spoon prior to well installation. Samples will be analyzed in the CLP for organic and inorganic contaminants and TOC.

Wells installed in the landfill and adjacent to B4S in the vicinity of the floating naphtha will be constructed of 2-inch nominal diameter threaded, flush joint stainless steel riser pipe. All remaining monitoring wells will be constructed of 2-inch nominal diameter flush joint schedule 40 PVC with factory slotted 5 and 10 foot screens. All screen bottoms will be capped prior to installation and joints wrapped with Teflon thread tape to ensure water tight seals. All riser pipes and screens will be steam cleaned prior to installation.

Wells will be joined section by section directly inside the auger or casing. Shallow wells will be installed such that the screened interval will be 4 feet above and 6 feet below the water table. If the formation is considered to be sufficiently coarse and graded, as determined from experienced field judgement, the formation will be allowed to collapse around the screen as the auger or casing is

Table 2-6
RATIONALE FOR MONITORING WELL LOCATIONS

Well Number	Proposed Depth	Rationale
Monitoring Wells*		
MW-1S	45 Feet	To help determine upgradient groundwater quality (background)
MW-1M	90 Feet	
MW-2S	45 Feet	To help determine groundwater quality and stratigraphy beneath the landfill
MW-2M	90 Feet	
MW-2D	135 Feet	
MW-3S	45 Feet	To help determine downgradient groundwater quality and vertical contaminant distribution
MW-3M	90 Feet	
MW-3D	135 Feet	
MW-4S	45 Feet	To help determine downgradient groundwater quality
MW-5S	45 Feet	To help determine downgradient groundwater quality
MW-6M	70 Feet	To help determine westward extent of groundwater contamination
MW-7M	70 Feet	To help determine eastward extent of groundwater contamination
MW-8S	45 Feet	To help determine downgradient groundwater quality and vertical contaminant distribution
M	90 Feet	
D	135 Feet	
MW-9M	70 Feet	To help determine southerly extent of groundwater contamination
MW-10M	70 Feet	To help determine southerly extent of groundwater contamination

Note: Location of monitoring wells dependent upon findings of soil boring program.

withdrawn, forming a natural filter pack. If the formation is not suitable, an artificial filter pack consisting of a medium to coarse flint or silica sand will be installed to a minimum of 3 feet above the top of the screened interval. The remainder of the annular space will be filled with a thick bentonite slurry to within 5 feet of the ground surface. The slurry will be introduced from the top of the filter pack to within 5 feet of the top of the ground surface using the tremie method. Slurry emplacement will be simultaneous with auger or casing removal.

Following installation, each monitoring well will be developed until substantially free of sediment, and until pH and conductivity are stable to the satisfaction of the CH2M HILL hydrogeologist. Wells will be developed using the double tube air lift or surge and bail methods.

Water Level Monitoring

All new and existing monitoring wells will be surveyed to establish horizontal location and elevation of the measuring points. Elevation measurements will be taken on the riser pipe with the measuring point designated by a chisel mark. All elevations will be referenced to the benchmark previously established at the site. All wells will be located horizontally to within ±5 feet. Vertical elevations of measuring points will be made to the nearest 0.01 foot.

Water levels will be measured prior to groundwater sampling and on a monthly basis at all monitoring wells, and at the staff gauge for the duration of the RI. This is assumed to be 5 months. An electric water level indicator graduated in 0.1-foot increments will be used.

Pump Test

The pump test will be used to determine the physical characteristics of the underlying aquifer. The pump test is important to understanding how the aquifer responds to pumping given the close proximity of the site to constant head boundaries. The test is needed to evaluate groundwater collection alternatives.

An 8-inch minimum I.D., fully penetrating production well would be drilled using mud rotary techniques for the purpose of conducting a 72 hour pump test. Eight monitoring wells will be used as observation wells for this test. Figure 2-13 illustrates the layout of the pumping and observation wells that will be used for the test. Data from

the pump test will be used in the pre-design work on a groundwater collection remedial action. The pump test may generate up to 1,000 gpm for 3 days. The volume of water, 14.3 million gallons, is too large to store onsite and will be discharged to the Black River. Discharge criteria and the need for treatment will be discussed with WDNR and EPA prior to beginning the test.

Groundwater Sampling

Groundwater samples will be collected from existing and newly installed monitoring wells in two sampling rounds separated by at least 2 months. In the first round, up to three samples floating on the water table will be taken prior to groundwater sampling. A clear bailer will be used to obtain the sample and to determine the thickness of the floating layer. Prior to groundwater sampling, all wells will be purged to remove stagnant water or stratified contaminants. Monitoring well sampling procedures are described in detail in the Field Sampling Plan (Appendix A). Groundwater samples will be analyzed in a CLP laboratory for organic and inorganic contaminants. A list of conventional pollutant parameters important for evaluation of groundwater treatment will also be analyzed as part of Round One analyses (Table 2-7).

RESIDENTIAL WELL SAMPLING

The objective of this task is to determine whether site contaminants have migrated to downgradient residential wells. Nine residential wells will be sampled. They will include the Hubley, Fritz, Marshall, and Sportsman's Club wells, both Miller wells, and three wells from the subdivision southeast of the site. An additional round of samples, not included in the expenditures limit, may be necessary if contamination is found in any of the wells other than the shallow Miller well.

Grab samples will be obtained from the cold water taps of a location upstream of water softeners or filters after the wells have been adequately purged to remove stagnant water. Samples will be analyzed for organic and inorganic contaminants at SAS detection limits.

SURFACE WATER AND SEDIMENT SAMPLING

Data for surface water and sediment contamination do not exist. As discussed in the preliminary risk assessment, site contaminants may have migrated by way of overland

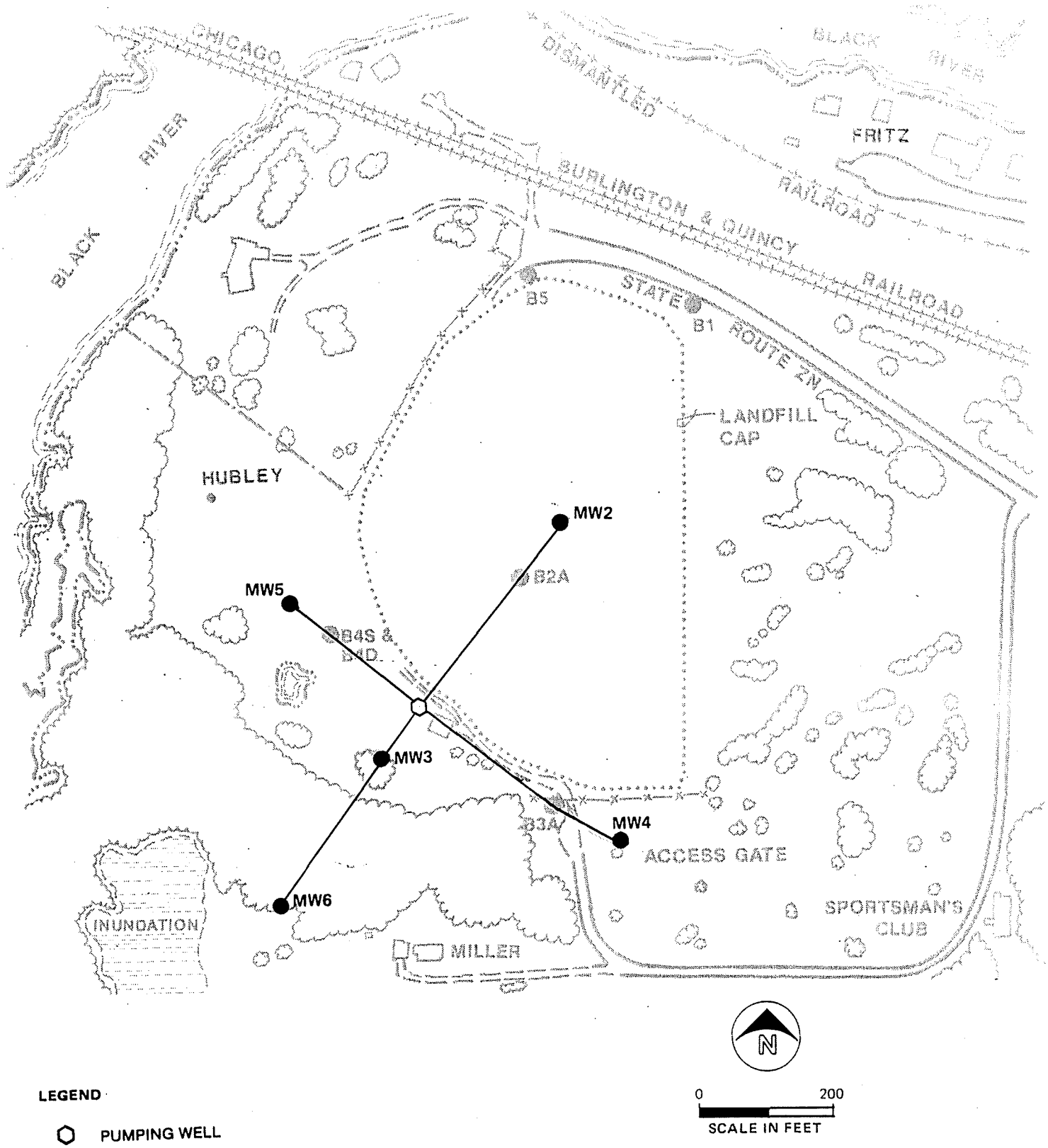


FIGURE 2-13
PUMP TEST LAYOUT
ONALASKA LANDFILL QAPP

Table 2-7
CONVENTIONAL POLLUTANTS FOR ANALYSES

BOD
COD
TOC
TDS
TSS
Chloride
Alkalinity as $CaCO_3$
Nitrate as N
Nitrite as N
Ammonia
Total Phosphorous
Sulfide
Sulfate
Total Carbon
Oil and Grease

Note: Total carbon will be determined as part of TOC analyses since the TOC method determines TOC by the following equation:

Total Organic Carbon = Total Carbon - Inorganic Carbon

GLT824/9-3

runoff to the Black River and adjacent wetlands during site operation. Currently, groundwater contaminants may be migrating to the Black river and wetlands.

The hydrogeologic investigations discussed earlier will be conducted onsite prior to this task. The results of those investigations may modify the location and number of surface water and sediment samples.

The objectives of this subtask are to evaluate whether site contaminants have migrated to the surface water and sediment.

Ten surface water and sediment samples will be obtained at the locations shown on Figure 2-14. Seven of the surface water samples will be taken from stagnant areas along the eastern bank of the Black River, in the "inundated area" southwest of the site, and in the wetland south of the site. One of these locations will be upgradient of the site but downgradient of the railroad tracks so that background contamination resulting from the railroad can be determined. At each location three sediment sample will be taken. Each sample will be analyzed in the CSL for the five target VOCs. Based on these screening results up to seven sediment samples will be sent to the CLP for TCL VOCs. At each of the seven location shown on Figure 2-14, samples for TCL semi-volatiles, pesticides/PCBs and inorganic compounds will be composited from the three subsamples. The three samples will be located within 20 feet of each other and taken from cores of the upper 6 inches of sediment.

The remaining three samples will be collected along the eastern bank of the main channel of the Black River. One of the samples will be a background sample. The surface water sample will be taken within 6 inches of the bank at mid depth. The sediment samples will be taken from within 6 inches of the bank. Otherwise sampling and analysis of sediment samples will be as described above. Surface water samples will be analyzed in the CSL for five target VOCs found onsite. If contamination is found in this analysis or the CLP analysis, an additional round of sampling may be necessary.

SAMPLING AND ANALYSES

All anticipated field sampling and analyses for the Onalaska site are summarized in Table 2-8. A detailed description can be found in the Field Sampling Plan (Appendix A). The target compound list in Appendix A (Table A-5) represents the compounds

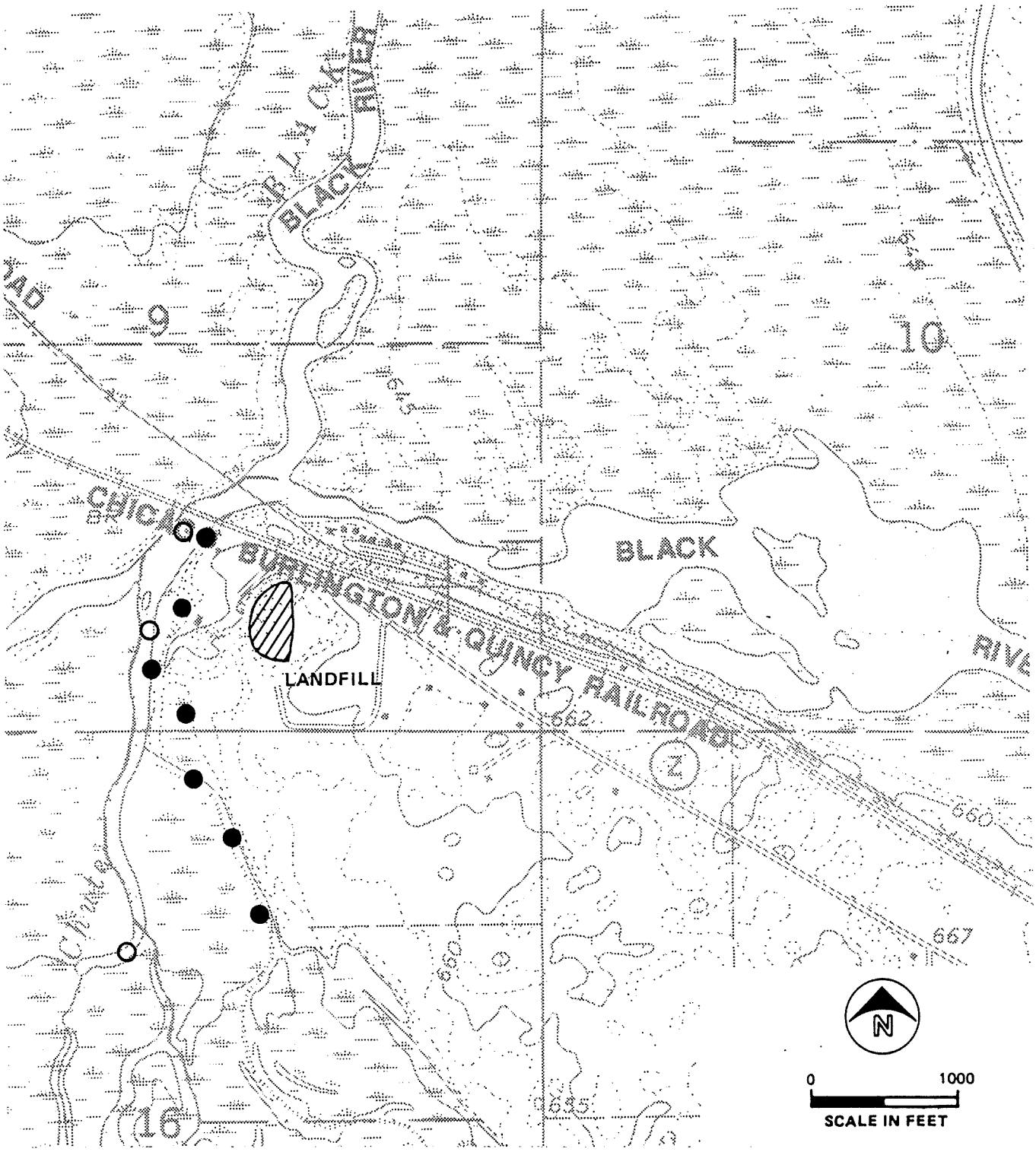
Table 2-8
FIELD INVESTIGATION SUBTASK SUMMARY

Subtask	Areas or Number of Locations	CSL VOC Analysis[a] Soil Gas	Water	Soil	Offsite Analysis	Comments
FM Topographic Survey	14 ac @ 1-inch = 30 feet 145 ac @ 1-inch = 100 feet					2-foot contours.
FS Cap Investigation Tests	11 Test Pits 11 Infiltrometer Tests				11 GA 11 PT	
FP Geophysical Investigation						
Magnetometer	12 ac					Reading on 20-foot centers.
Electromagnetic Survey	26 ac					Readings on 40-foot centers, 20-meter and 10-meter coil separations.
FG Solvent Disposal Area						
Soil Gas Survey	100 probe samples	100				Sampling on 10-foot to 100-foot grid.
Test Pits	3 Test Pits			30	10 PP, IN	Sampling every 2 feet over 20-foot depth.
FQ Hydrogeologic Invest.						
Geotechnical Borings	2(4) to 135 feet 6(20) to 70 feet		18(50)		24(72)GA 8(24)TOC	Maximum of three samples/borings analyzed for GA.
Monitoring Wells	6(8) to 45 feet 4(9) to 70 feet 4(8) to 90 feet 3(5) to 135 feet					
Water Levels	All 23(36) wells 3 times					
Pump Test	1 6-inch well					One 72-hour pump test
FO Environmental Sampling						
Monitoring Wells	46(72)		27(40)		46(72) PP 3 PP 23(36)CP	Two rounds of samples. Pure phase product sample. One round of samples.
Residential Wells					9(18)PP	
Surface Water	10(20)		10(20)		10(20)PP	
Sediment	10(20)			30(60)	10(20)PP	Sediment samples are a composite of three subsamples (except VOAs).
Subsurface Soil	10				10 GA, PP TOC	

Notes:
Numbers in parenthesis represent the maximum possible number of samples to be taken, as noted in the approved work plan.

GA = Geotechnical parameter analysis--grain size, Atterberg limits (shrinkage limits for task FS only)
PT = Permeability test on Shelby tube sample
IN = TOC, TOX, ash, sulfur, Btu, oil and grease, EP toxicity
CP = Conventional parameters listed in Table 4-3
PP = TCL organic compounds and inorganic chemicals

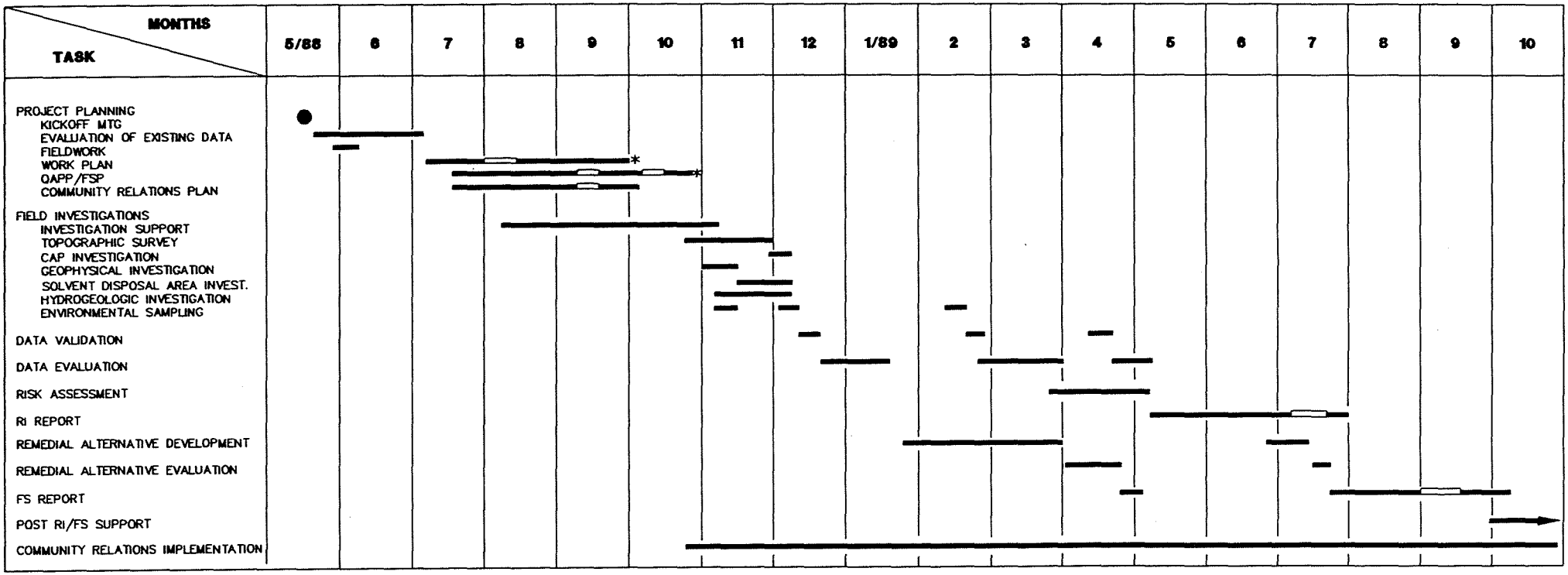
[a]Field GC analysis will be performed on up to five VOCs. Soil samples will be extracted with solvent followed by GC analysis.



LEGEND

- O — SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS IN MAIN RIVER CHANNEL
- ● — SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS IN IN STAGNANT WATER

FIGURE 2-14
SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS
ONALASKA LANDFILL QAPP



LEGEND

▭ AGENCY REVIEW

* WORK PLAN APPROVAL/QAPP APPROVAL

FIGURE 2-15
ONALASKA RI/FS SCHEDULE
ONALASKA LANDFILL QAPP

of interest for the RI that will be analyzed for through the CLP. Computer-assisted mass spectroscopy library searches will be conducted to identify as many as 30 additional organic compounds.

PROJECT SCHEDULE

The RI/FS schedule is presented in Figure 2-15. The schedule meets the 18-month RI/FS desired by U.S. EPA. It is based on the scope of work presented herein for the expenditure limit. If the maximum scope is necessary, additional time to complete the field investigations will be required.

Section 3
PROJECT ORGANIZATION AND RESPONSIBILITY

CH2M HILL has overall responsibility for all phases of the RI/FS. CH2M HILL will perform the field investigation, prepare the RI report, and perform the subsequent feasibility study. Project management will be provided by CH2M HILL.

The following responsibilities have been assigned for the project:

- Remedial Project Manager (RPM)
  Kevin Adler (U.S. EPA)

- Site Manager (SM)
  Phil Smith (CH2M HILL)

- Program Manager (PM)
  Mike Jury (CH2M HILL)

- Quality Assurance Manager (QAM)
  Greg Peterson (CH2M HILL)

- CH2M HILL Review Team Leader (RTL)
  John Fleissner (CH2M HILL)

- Sample Team Leaders (STL)
  Jeff Lamont/CH2M HILL
  Chris Lawrence/CH2M HILL
  Jeff Keiser/CH2M HILL

- Laboratory Operation
  EPA Close Support Laboratory (CSL; Operated by CH2M HILL), EPA Contract Laboratory Program (CLP), Region V Central Regional Laboratory (CRL)

- System/Performance Audits
  EPA EMSL--Las Vegas (CLP) and the Quality Assurance Section of the Region V CRL

- Field Audits
  CH2M HILL QA Manager

- Close Support Laboratory Audits
  Contract Project Management Section (CPMS) of the CRL

- Special Analytical Services Request Preparation
  Joe Sandrin/CH2M HILL
  Dean Charpentier/CH2M HILL

- Review of Special Analytical Services Data
  Dean Charpentier/CH2M HILL

- Review of Tentatively Identified Compounds
  Dean Charpentier/CH2M HILL

- CSL Standard Operating Procedure Development
  Dean Charpentier/CH2M HILL

- Review of CSL Data
  Dean Charpentier/CH2M HILL

- CLP Data Assessment and Review of Tentatively Identified Compounds
  EPA Region V, CPMS of the CRL

- QA/QC of CSL Data
  EPA Region V, CPMS of the CRL

- CLP Data Completeness
  CH2M HILL

- CSL Data Completeness
  CH2M HILL

Primary responsibility for project quality rests with the SM. Independent QA review is provided by the QA reviewers. A project organization chart is presented in Figure 3-1.

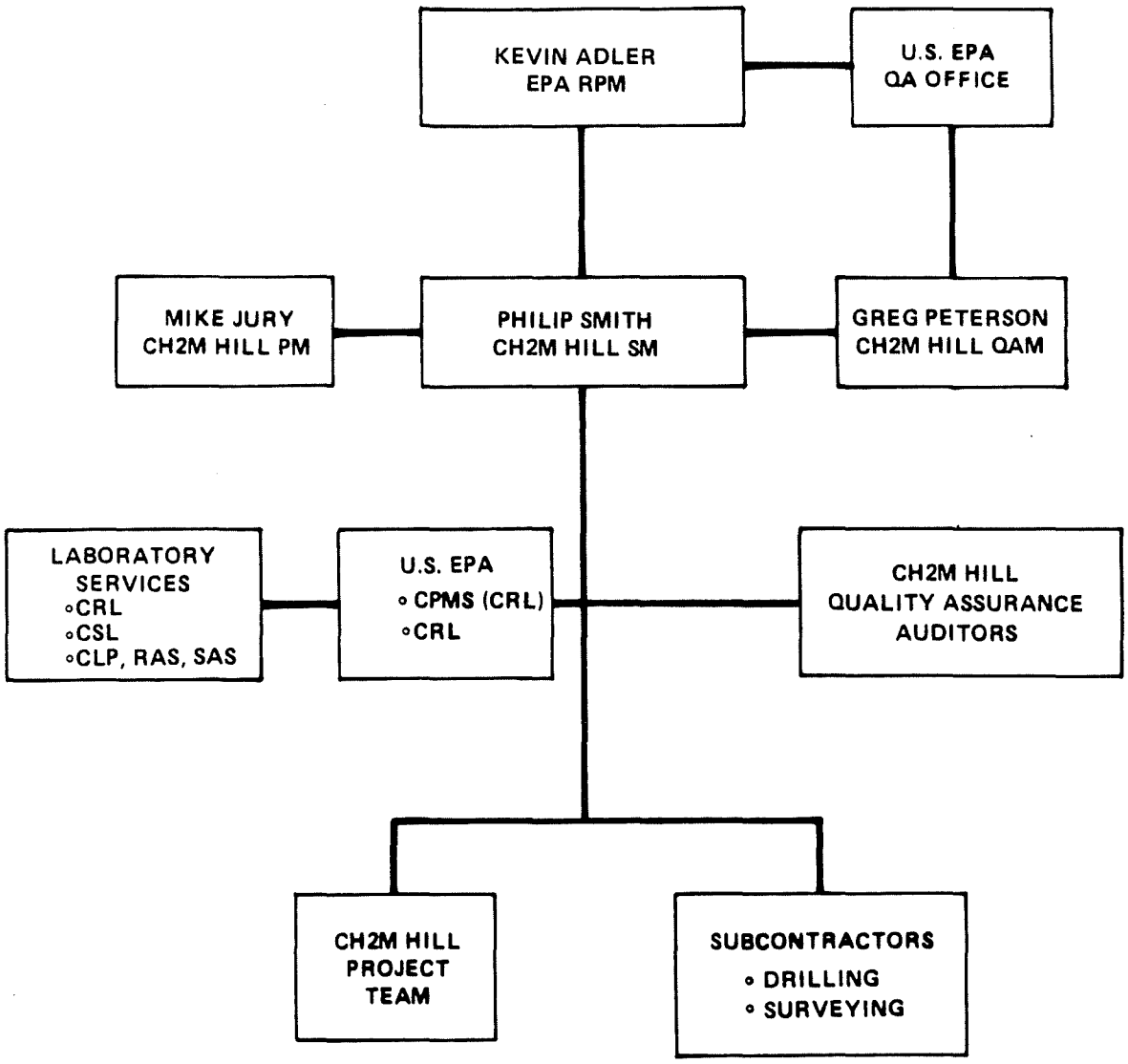


FIGURE 3-1
PROJECT ORGANIZATION
ONALASKA LANDFILL QAPP

Section 4
QUALITY ASSURANCE OBJECTIVES

The overall QA objectives are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide legally defensible results in a court of law. This section addresses objectives for field QC audits; accuracy, precision, and sensitivity of laboratory analyses; level of QA effort; method detection limits; and completeness, representativeness, and comparability.

FIELD QC AUDITS

REPLICATES AND BLANKS

Replicate and field blank groundwater, surface water, surface and subsurface soil, and sediment samples will be collected in the field and submitted to a CLP laboratory to assess the quality of data from field sampling efforts. Replicate samples will be used to assess the combined effects of sample collection, handling, and analysis on data precision. Field blanks will be analyzed to check for any procedural factors or ambient conditions at the site that may cause contamination. Trip blanks will also be prepared for groundwater, surface water, and residential well VOA samples to check cross-contamination that may occur during sample storage or shipment.

Replicate samples must be collected concurrently with actual samples in exactly equal volumes, at the same location, with the same sampling equipment, and in identical containers. Replicate samples will be preserved and handled in the same manner as the samples. Field blank samples will consist of deionized, contaminant-free water or clean sand that is collected, containerized, treated, and handled in the same manner as the samples. Replicates and field blanks will be collected at a rate of 1 replicate and 1 blank for every 10 or fewer samples collected each day.

The trip blank samples will be prepared at CH2M HILL's Milwaukee warehouse and sent to the site. They will accompany the groundwater samples to and through the CLP laboratory at a rate of 1 per shipping container per day.

The numbers of replicate and field blank samples to be collected are listed in Table 4-1. Sampling procedures are specified in the Field Sampling Plan (Appendix A).

FIELD MEASUREMENTS

Field measurements, including hydraulic conductivity, pH, specific conductivity, and temperature, involve measurements where QA concerns are appropriate but sample collection is not required. Procedures for field measurements, equipment calibration (where appropriate), and maintenance are documented in Appendix D. The primary QA objectives for field measurements are to obtain reproducible measurements with a degree of accuracy consistent with limitations of the analytical techniques used and with the intended use of the data.

SURVEY ACTIVITIES

Accuracy and reproducibility standards for survey activities will be consistent with those given in the standard survey reference, Manual of Survey Instructions 1973, prepared by the Bureau of Land Management. All horizontal locations will be obtained to an accuracy of ±1.0 foot. Vertical elevations will have an accuracy of ±0.1 foot for the ground surface and ±0.1 foot for well casings.

ACCURACY, PRECISION, AND SENSITIVITY OF LABORATORY ANALYSES

All surface and subsurface soil, sediment, groundwater, and surface water samples will be analyzed by a CLP laboratory. The QA goals of routine analytical services (RAS) are established under CLP guidelines, as stated in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987). The quality control limits of accuracy and precision for CLP inorganic and organic analyses are also stated in the guidelines. Besides the routine organic and inorganic analyses, CLP Special Analytical Services (SAS) will be performed on soil, sediment, groundwater, and surface water samples for additional parameters as needed. The accuracy and precision requirements for SAS are included in Appendix B.

COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Data completeness can be quantified during data assessment. The laboratories are expected to provide data meeting QC acceptance criteria for 90 percent or more of the requested determinations.

The objective of determining representativeness is to assess whether the information obtained during the investigation

accurately represents the actual site conditions. Requirements of representativeness were determined during the planning stages of the RI and are reflected in the sampling plan. Representativeness will be assessed after initial data validation.

To ensure samples are comparable and meet the objectives of the project, standard sampling procedures and recognized field and laboratory techniques will be used.

METHOD DETECTION LIMITS

Contract required method detection limits for RAS are given in Attachment B-1. Contract required method detection limits for SAS are given in Appendix B. The detection limits for field measurements are described in the procedures for operating the equipment (Appendix D).

Table 4-1
(1 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
SUBSURFACE SOIL (from geotech & MW borings)	HNu/OVA Screen	VOCs RAS Protocol	13	1	13	2	1	2	2	1	2	2	1	2	17
		Semivolatiles RAS Protocol	13	1	13	2	1	2	2	1	2	2	1	2	17
		Pesticides/PCBs RAS Protocol	13	1	13	2	1	2	2	1	2	2	1	2	17
		Metals RAS Protocol	13	1	13	2	1	2	2	1	2	2	1	2	17
		TOC (MSA, Part II, 2nd ed.)	13	1	13	2	1	2	2	1	2	2	1	2	17
		Atterberg Limits SAS (ASTM D4318)	24	1	24	---	---	---	---	---	---	---	---	---	24
		Percent Moisture SAS (ASTM D2216-80)	11	1	11	---	---	---	---	---	---	---	---	---	11
		Grain Size SAS (ASTM D422)	24	1	24	---	---	---	---	---	---	---	---	---	24

Notes:

1. See Table A-1 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

Table 4-1
(2 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
SUBSURFACE SOIL (from Test Pits)	HNu/OVA Screen	VOCs RAS Protocol	10	1	10	1	1	1	1	1	1	1	1	1	12
		Semivolatiles RAS Protocol	10	1	10	1	1	1	1	1	1	1	1	1	12
		Pesticides/PCBs RAS Protocol	10	1	10	1	1	1	1	1	1	1	1	1	12
		Metals RAS Protocol	10	1	10	1	1	1	1	1	1	1	1	1	12
		Btu SAS (ASTM 2015-77)	10	1	10	1	1	1	---	---	---	---	---	---	11
		% Ash SAS (ASTM 3174-82)	10	1	10	1	1	1	---	---	---	---	---	---	11
		% Moisture SAS (ASTM D2216)	10	1	10	1	1	1	---	---	---	---	---	---	11
		Sulfur Content SAS (ASTM D3177-82)	10	1	10	1	1	1	---	---	---	---	---	---	11
		TOC (MSA, Part II, 2nd ed.)	10	1	10	1	1	1	---	---	---	1	1	1	11
		Total Chlorine SAS (ASTM D808)	10	1	10	1	1	1	---	---	---	---	---	---	11
		EPTOX (SW846 Method 1310)	10	1	10	1	1	1	---	---	---	---	---	---	11
		Atterberg Limits SAS (ASTM D4318)	11	1	11	---	---	---	---	---	---	---	---	---	11
		Percent Moisture SAS (ASTM D2216-80)	11	1	11	---	---	---	---	---	---	---	---	---	11
		Grain Size SAS (ASTM D422)	11	1	11	---	---	---	---	---	---	---	---	---	11

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

TABLE 4-1
(3 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
SEDIMENT															
	HNU/OVA Screen	VOCs RAS protocol	20	1	20	2	1	2	2	1	2	2	1	2	24
		Semivolatiles RAS Protocol	20	1	20	2	1	2	2	1	2	2	1	2	24
		Pesticides/PCBs RAS Protocol	20	1	20	2	1	2	2	1	2	2	1	2	24
		Metals RAS Protocol	20	1	20	2	1	2	2	1	2	2	1	2	24

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

Table 4-1
(4 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
GROUNDWATER (Monitoring Wells-- Round One)	pH Specific Conductance Temperature HNu/OVA Screen	VOCs RAS Protocol Unfiltered Samples	36	1	36	4	1	4	4	1	4	4	1	4	48
		Semivolatiles RAS Protocol Unfiltered Samples	36	1	36	4	1	4	4	1	4	4	1	4	44
		Pesticides/PCBs RAS Protocol Unfiltered Samples	36	1	36	4	1	4	4	1	4	4	1	4	44
		Metals RAS Protocol Filtered Samples	36	1	36	4	1	4	4	1	4	4	1	4	44
		BOD5 SAS (EPA 405) Unfiltered	36	1	36	4	1	4	4	1	4	3	1	3	44
		Chemical Oxygen Demand SAS (Low: EPA 410.2, Medium: EPA 410.1) Unfiltered Samples	36	1	36	4	1	4	4	1	4	3	1	3	44
		Total Organic Carbon SAS (EPA 415.1) Unfiltered Samples	36	1	36	4	1	4	4	1	4	3	1	3	44
		Total Phosphorus SAS (EPA 365) Unfiltered Sample	36	1	36	4	1	4	4	1	4	3	1	3	44

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

3. Trip blank samples (for VOC water samples only) shall be collected at a frequency of one (two 40-ml vials) per shipping cooler of VOC water samples.

Table 4-1
(5 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
(Monitoring Wells-- Round One continued)	HNU/OVA Screen	Total Dissolved Solids SAS (EPA 160.1) Unfiltered Samples	36	1	36	4	1	4	4	1	4	---	---	---	44
		Total Suspended Solids SAS (EPA 160.2) Unfiltered Samples	36	1	36	4	1	4	4	1	4	---	---	---	44
		Alkalinity SAS (EPA 310.1) Unfiltered Samples	36	1	36	4	1	4	4	1	4	---	---	---	44
		Chloride SAS (EPA 325.1, 325.2 Unfiltered Samples	36	1	36	4	1	4	4	1	4	3	1	3	44
		Ammonia as N SAS (EPA 350) Unfiltered Sample	36	1	36	4	1	4	4	1	4	3	1	3	44
		Nitrate + Nitrite SAS (EPA 353) Unfiltered Sample	36	1	36	4	1	4	4	1	4	3	1	3	44
		Sulfate SAS (EPA 375.2) Unfiltered Sample	36	1	36	4	1	4	4	1	4	3	1	3	44
		Sulfide SAS (Std. Method 16th ed. 427, 421) Unfiltered Sample	36	1	36	4	1	4	4	1	4	---	---	---	44
		Oil & Grease SAS (EPA Method 413.2) Unfiltered	36	1	36	4	1	4	4	1	4	3	1	3	44
		Total Carbon (Std. Method 16th ed. 505)	36	1	36	4	1	4	4	1	4	3	1	3	44
		Pure Product Naptha (ASTM Method D3328-78)	3	1	3	1	1	1	1	1	1	1	1	1	5

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

3. Trip blank samples (for VOC water samples only) shall be collected at a frequency of one (two 40-ml vials) per shipping cooler of VOC water samples.

Table 4-1
(6 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
GROUNDWATER (Monitoring Wells-- Round Two)	pH Specific Conductance Temperature HNu/OVA Screen	VOCs RAS Protocol Unfiltered Samples	36	1	36	4	1	4	4	1	4	4	1	4	48
		Semivolatiles RAS Protocol Unfiltered Samples	36	1	36	3	1	3	3	1	3	4	1	4	42
		Pesticides/PCBs RAS Protocol Unfiltered Samples	36	1	36	3	1	3	3	1	3	4	1	4	42
		Metals RAS Protocol Filtered Samples	36	1	36	3	1	3	3	1	3	4	1	4	42

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

3. Trip blank samples (for VOC water samples only) shall be collected at a frequency of one (two 40-ml vials) per shipping cooler of VOC water samples.

Table 4-1
(7 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
GROUNDWATER (Residential wells)	pH	VOCs CLP SAS Unfiltered Samples	9	2	18	1	2	2	2	1	2	2	1	2	24
	Specific Conductance	Semivolatiles CLP SAS Unfiltered Samples	9	2	18	1	2	2	2	1	2	2	1	2	22
	Temperature														
	HNu/OVA Screen	Pesticides/PCBs CLP SAS Unfiltered Samples	9	2	18	1	2	2	2	1	2	2	1	2	22
		Metals CLP SAS Unfiltered Samples	9	2	18	1	2	2	2	1	2	2	1	2	22

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

3. Trip blank samples (for VOC water samples only) shall be collected at a frequency of one (two 40-ml vials) per shipping cooler of VOC water samples.

Table 4-1
(8 of 8)

SUMMARY OF SAMPLING AND ANALYSIS AT THE ONALASKA MUNICIPAL LANDFILL SITE

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	REPLICATES NO.	REPLICATES FREQ.	REPLICATES TOTAL	FIELD BLANKS NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL
SURFACE WATER															
	pH	VOCs RAS Protocol	20	1	20	2	1	2	2	1	2	2	1	2	26
	Specific Conductance	Unfiltered Samples													
		Semivolatiles	20	1	20	2	1	2	2	1	2	2	1	2	24
	Temperature	RAS Protocol													
	HNu/OVA Screen	Unfiltered Samples													
		Pesticides/PCBs	20	1	20	2	1	2	2	1	2	2	1	2	24
		RAS Protocol													
		Unfiltered Samples													
		Metals	20	1	20	2	1	2	2	1	2	1	1	1	24
		RAS Protocol													
		Unfiltered Samples													

Notes:

1. See Table A-5 of Appendix A for a complete list of parameters.

2. Matrix spikes (MS) and matrix spike duplicates (MSD) are not included in the total number of samples. These numbers represent samples for which extra volume must be collected for the laboratory to perform contract required QC analyses. Triple the normal volumes will be collected for VOA samples and double the normal volumes for semivolatiles, pesticides, and PCBs.

3. Trip blank samples (for VOC water samples only) shall be collected at a frequency of one (two 40-ml vials) per shipping cooler of VOC water samples.

Section 5
SAMPLING PROCEDURES

Detailed sampling procedures are provided in the Field Sampling Plan (see Appendix A). Analyses of the samples are summarized in Table 4-1.

Section 6
SAMPLE CUSTODY PROCEDURES

INTRODUCTION

It is U.S. EPA and Region V policy to follow the U.S. EPA Region V 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: sample collection, laboratory, and final evidence files. Final evidence files, including all originals or laboratory reports, and purge files are maintained under document control in a secure area.

A sample or evidence file is under your custody if it:

- Is in your possession
- Is in your view after being in your possession
- Was in your possession and you placed it in a secured location
- Is in a designated secure area

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact.

FIELD-SPECIFIC CUSTODY PROCEDURES

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.

All bottles will be tagged with sample numbers and locations. The Sample Management Office number and stickers will be affixed.

Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather.

The Contractor's site manager must 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 EPA Remedial Project Manager of a breach or irregularity in chain-of-custody procedures.

Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the form. 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/from a secure storage area.

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.

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.

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.

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 off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

LABORATORY CUSTODY PROCEDURES

CONTRACT LABORATORY

The chain-of-custody procedures for the Contract Laboratory Program are described in the SOWs for RAS. This same custody procedure applies to SAS. These custody procedures along with the holding time requirements for CLP samples are described in the appropriate SOW document.

CENTRAL REGIONAL LABORATORY CHAIN-OF-CUSTODY

The Central Regional Laboratory has its own regional custody scheme for Drinking Water Specific Samples. For Onalaska, the CRL may be involved in chain-of-custody sample tracking in the following way: samples are shipped directly from the field to a contract laboratory without ever being delivered to the CRL.

The internal CRL custody protocol has been revised so that it addresses this situation and also meets all National EPA custody requirements. Moreover, the revised protocol requires only one new internal document--the custody logbook. This logbook replaces the existing shipping and receiving log. The new procedure is applied to the custody situation as follows.

When samples are shipped directly from the field to a contract laboratory, no one at the CRL signs the chain-of-custody record.

The sampling team submits a report to the CRL describing its sampling activities. Please see Appendix E for instructions on filling out sample documentation.

The purge files from the Contract Laboratory Program are maintained by the Region V CRL Laboratory Support Team Data Coordinator and CPMS.

FIT maintains the SI files along with all relevant records, PA/SI reports, safety reports, logs, field notebooks, pictures, subcontractor reports, and CPMS data reviews for the specific site held in a secured, limited access area and under custody of the FIT-DCC.

FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final evidence files from the CRL and the CLP are maintained by the Region V CRL Laboratory Support Team Data Coordinator.

The Contractor maintains the RI files along with all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and CPMS data reviews in a secured, limited access area and under custody of the Contractor's site manager.

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Section 7
ANALYTICAL SERVICES

CLP ROUTINE AND SPECIAL ANALYTICAL SERVICES

All samples will be analyzed through RAS or SAS protocols of the CLP. QAPP elements of RAS protocols predetermined in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987) are:

- Calibration procedures and frequency
- Analytical procedures
- Internal quality control checks
- Data reduction, validation, and reporting
- Performance and system audits
- Preventive maintenance
- Data measurement assessment
- Corrective action

ANALYTICAL AND CALIBRATION PROCEDURES

All samples will be analyzed for TCL metals, VOCs, or both. All sample testing will conform to the guidelines in the User's Guide to the U.S. EPA Contract Laboratory Program and to those specified in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987). Computer-assisted library searches will be made in accordance with the CLP SOWs.

INTERNAL QUALITY CONTROL

Internal quality control procedures for soil, sediment, groundwater, and surface water samples will follow the guidelines of the CLP specified in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987).

DATA REDUCTION, VALIDATION, AND REPORTING

Data validation will be performed by the CPMS of the CRL to ensure the accuracy of data reporting and the acceptability of data reductions. Data handling standards guarantee the integrity of raw data and create a traceable audit trail from chain-of-custody through data reduction. The data base is assessed for numerical reasonableness and acceptability. Extremes are eliminated on the basis of review by qualified and experienced individuals. Validation procedures provide for the generation of defensible analytical data acceptable for use as evidence in legal proceedings.

Data reduction will be documented so checks can be accurately made to monitor the validity of the reduction process. Data reduction includes all processes that change the values or numbers of data items. Documentation will address the reliability of computations, appropriateness of models as a framework for investigating the study questions, and the overall correctness of the data reduction. Individuals responsible for work performed on data will date and sign (or initial) the generated material so questions about conclusions or methods can be directed quickly and resolved.

Reports will record the performance of all tasks and results. Missing data will be explained and the validation of data will be demonstrated each time data are recorded, calculated, or transcribed. Internal checks will be made to uncover or avoid errors in the data collection, recording, or transfer process.

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits for the CLP are the responsibility of the Support Services Branch, OERR, EPA and of EMSL--Las Vegas, EPA. Audits are performed as described in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987).

DATA ASSESSMENT

Data assessment is the responsibility of the CPMS of the CRL. Data completeness will be checked by CH2M HILL and the Sample Management Office.

DEFINITIONS

Accuracy and precision definitions for analyses performed at the CLP laboratories are listed in the CLP SOW for Organics and the CLP SOW for Inorganics (both July 1987).

CORRECTIVE ACTIONS

If quality control audits result in the detection of unacceptable conditions of data, the SM will be responsible for developing and initiating corrective action. The RM and QAM will be notified if nonconformance is of program significance or requires special expertise not normally available to the project team. Corrective action may include:

- Reanalyzing samples if holding time criteria permit
- Resampling and analyzing
- Evaluating and amending sampling and analytical procedures
- Accepting data acknowledging level of uncertainty

CLOSE SUPPORT LABORATORY

Standard operating procedures for the CSL are provided in Appendix C.

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Section 8
QUALITY ASSURANCE REPORTS TO MANAGEMENT

No separate QA report for this project is anticipated. The final RI report and FS report will contain separate QA sections that summarize data quality information collected during the project.

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Appendix A
FIELD SAMPLING PLAN

CONTENTS

CONTENTS (Continued)

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Appendix A
FIELD SAMPLING PLAN

OBJECTIVE

This sampling plan documents procedures and practices to be followed during the RI field activities at the Onalaska Municipal Landfill. Samples will be collected from various media including surface and subsurface soils, sediment, groundwater, and surface water. The sampling plan will be used for guidance in performing the soil borings, monitoring well installations, test pit excavations, and execution of the geophysical, cap, and soil gas survey investigations. Table A-1 summarizes field sampling activities and analyses.

SAMPLE DESIGNATION

A CH2M HILL sample numbering system will be used to identify each sample, including duplicates and blanks. A Sample Management Office (SMO) number and a Central Regional Laboratory (CRL) number will be assigned to each sample at the same time. Refer to the User's Guide to the Contract Laboratory Program for an explanation of the SMO numbers and to the CRL Sample Handling Manual for an explanation of the CRL numbers. A listing of sample identification numbers will be maintained in the sampling log book by the team leader. Each CH2M HILL sample number will consist of three components, as described below.

The designation ON identifies the sample as being from the Onalaska site. Each sample will have a two-digit letter code corresponding to the sample type:

- SB--subsurface soil
- TP--test pit
- SD--sediment
- SS--surface soil
- MW--monitoring well
- RW--residential well
- SW--surface water
- SG--soil gas

Samples will have an alphanumeric sample location code after the code for sample type. At sites with more than one well, a letter indicating well depth (S-shallow; M-medium; D-deep) will follow the location number. Field blanks will have an FB following the letter code for the type of sample (e.g., a

groundwater field blank will be GWFB). Trip blanks will be designated by the code TB.

All samples will have an identifier to indicate the sampling event. Replicates will be identified by "99." For sampling points that may have multiple samples collected over time (e.g., monitoring wells), a number indicating the sequential sampling event will follow the type/location code. Groundwater samples will be given the designation "01" to indicate Round 1 groundwater sampling, "02" to indicate Round 2 sampling, and so on.

Soil samples will be given a sequential code indicator to indicate the depth at which the sample is taken. For example, soil borings will be sampled at 5-foot intervals (with a maximum of 3 samples submitted for analysis), so the sequential code indicator of "03" will indicate a sample taken at a depth of 15 feet.

For example:

- ON-MW3S-02: Round 2 sample from shallow monitoring well MW3
- ON-RW22-02: Round 2 sample from residential well 22
- ON-SB22-03: Third subsurface soil sample from soil boring 22

SOIL BORINGS

DRILLING

Eight deep soil borings will be drilled at the locations shown in Figure A-1. The proposed depth of each boring is presented in Table A-2. Up to 16 additional borings may be needed depending on the stratigraphic conditions encountered.

Borings GB1 and GB5 will be advanced to bedrock, about 135 feet below ground. The other six borings will be advanced to a depth of about 70 feet to determine the stratigraphy of the soils beneath the south portion of the landfill and south of the landfill near the potential migration pathways.

Upon encountering the water table, a groundwater sample will be collected from within the screened hollow-stem auger.

Table A-1 (Page 1 of 2)
SUMMARY OF FIELD SAMPLING AND ANALYSES

Media	Number of Locations	Number of Samples	Parameters Analyzed or Tested				
			RAS	SAS	CSL	Geotechnical Lab	Field
Subsurface Soil	8-24 borings	24-72 soil samples (5-ft intervals, max 3 samples/borings)				Grain size Atterberg limits	HNu OVA screen Field inspection for free product Visual classification
		10 composite soil samples from zones of greatest contamination and 4 geotechnical borings	Volatiles, semivolatiles, pesticides/PCBs, metals	TOC			
	3 test pits (solvent disposal area)	30 soil samples (2-ft intervals)	10 samples selected for: volatiles, semivolatiles, pesticides/PCBs, metals	10 samples selected for: oil and grease, Btu, % ash, % moisture, sulfur content, TOC, total chlorine Extraction Procedure Toxicity (EP Tox)	Indicator VOCs		
Landfill Cap	11 test pits (2-ft depth)	11 soil samples (grab samples)				Atterberg limits Grain size Moisture content Shrinkage limits 3 moisture-density relation tests 3 permeability tests	11 infiltrometer tests 40 nuclear density tests HNu/OVA screen
		11 Shelby Tube samples				In situ density Moisture content Permeability test	

Media	Number of Locations	Number of Samples	Parameters Analyzed or Tested				
			RAS	SAS	CSL	Geotechnical Lab	Field
Groundwater	23-36 monitoring wells	Round 1: 23-36 water samples	Volatiles, semivolatiles, pesticides/PCBs metals	BOD, COD, TOC, total phosphorus, TDS, alkalinity, chloride, ammonia as N, nitrate, nitrite, sulfate, sulfide, total carbon	Indicator VOCs, chloride, COD		pH, specific conductivity, temperature, HNu/OVA screen
		3 pure phase floating product		High hazard volatiles, semivolatiles, pesticides/PCBs, metals, naphtha anlysis			
		Round 2: 23-36 water samples	Volatiles, semivolatiles, pesticides/PCBs, metals	Oil and grease			pH, specific conductivity, temperature, HNu/OVA screen
	9 residential wells	9-18 water samples		Volatiles, semivolatiles, pesticides/PCBs, metals, low detection limits			pH, specific conductivity, temperature
	45-90 samples from geotechnical and monitoring well borings	45-90 water samples			Indicator VOCs, chloride, COD		pH, specific conductivity, temperature, HNu/OVA screen
Surface water	10-20 sample points	10-20 water samples	Volatiles, semivolatiles, pesticides/PCBs, metals		Indicator VOCs		pH, specific conductivity, temperature, HNu/OVA screen
Sediment	10-20 sample points	10-20 sediment samples (composites)	Volatiles, semivolatiles, pesticides/PCBs, metals		Indicator VOCs		
Soil Gas	100 gas probe locations over sample grid	100 gas probe samples			GC analysis for volatiles		

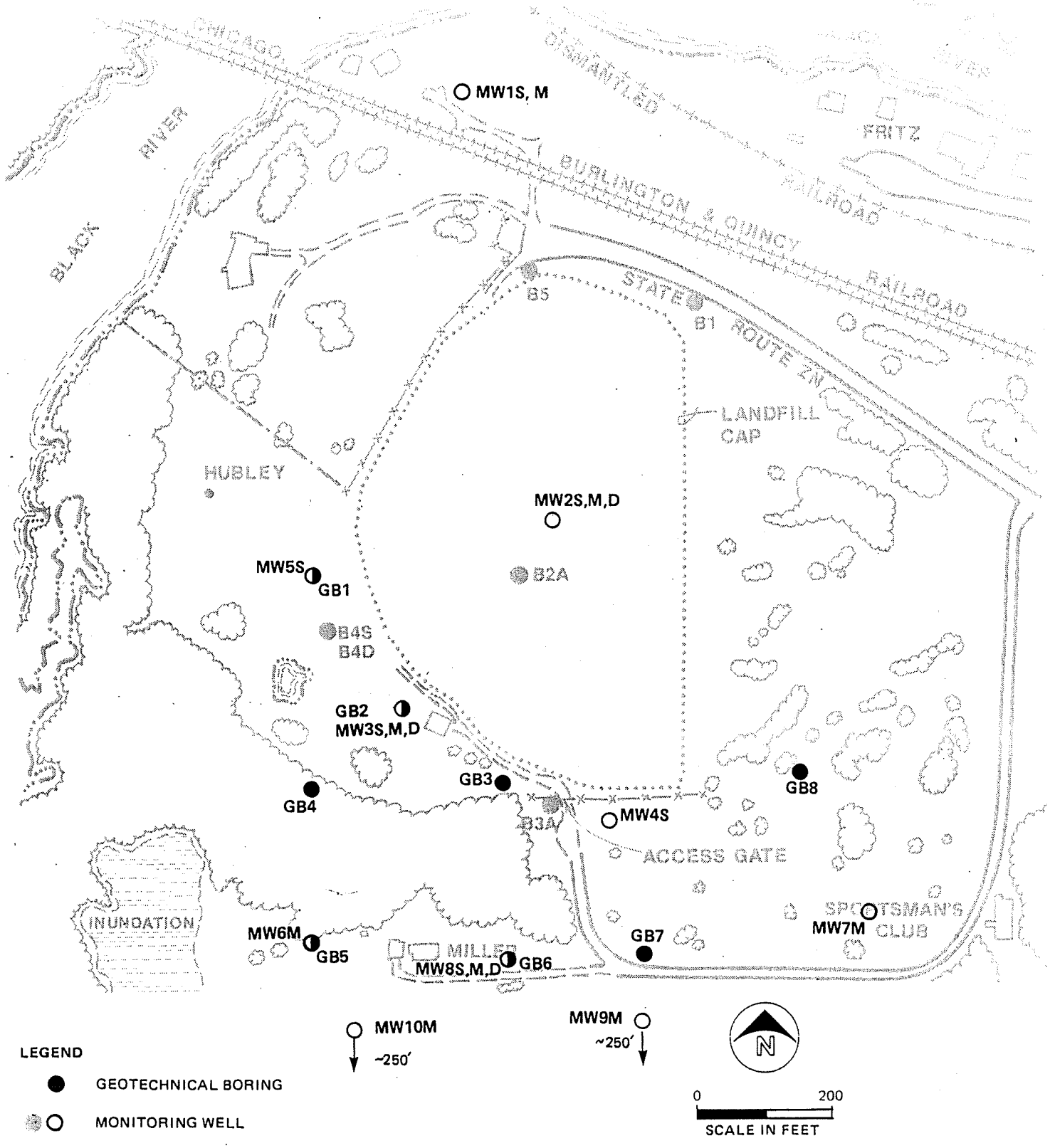


FIGURE A-1
GEOTECHNICAL BORINGS
ONALASKA LANDFILL FSP

Table A-2
RATIONALE FOR SOIL BORING LOCATIONS

Boring Location	Proposed Depth	Rationale
GB-1	Bedrock	o To help determine stratigraphy on west side of site where data is scarce, and to help locate upgradient monitoring well nest
GB-2	70 Feet	o To help determine stratigraphy in SW portion of the site where data is scarce, and to help locate upgradient monitoring well nest
GB-3	70 Feet	o To help determine location of down-gradient monitoring well nest
GB-4	70 Feet	o To help determine stratigraphy in SW corner of site where data is scarce, and to help locate downgradient monitoring well nest
GB-5	Bedrock	o To help determine stratigraphy of potential migration pathways and extent of contamination, and to help locate monitoring wells
GB-6	70 Feet	o To help determine stratigraphy of potential migration pathways and extent of contamination, and to help locate monitoring wells
GB-7	70 Feet	o To help determine downgradient stratigraphy and extent of contamination, and to help locate monitoring wells
GB-8	70 Feet	o To help determine stratigraphy in SE portion of the site where data is scarce

VOA samples will be collected using a stainless steel, bottom-filling bailer. After purging at least three volumes from the hollow-stem auger, two additional groundwater samples will be collected from each boring. GB1 and GB5 and one additional groundwater sample will be collected from the remaining borings.

Collected groundwater samples will be submitted to the CSL for a gas chromatograph screen for 1,2-dichloroethene, 1,1,1-trichloroethane, trichloroethene, toluene, and xylenes. Drill cuttings and purge water will be screened with an organic vapor analyzer to evaluate whether volatile organic contaminants are present. Soils visibly contaminated or with VOC concentrations greater than 5 ppm will be drummed and placed in a secure, onsite storage area.

Solid waste derived from drilling activities will be stored in barrels following the completion of each borehole. Barrels will be clearly marked so that contents can be identified. Barrels will be stored in a secure area on the site.

SAMPLING

The purpose of soil sampling is to obtain a representative portion of soil that can be submitted for chemical analysis, geotechnical analysis, or geomorphological classification. The following field information will be collected from each split-spoon sample:

- Visual inspection for free product
- Visual classification of stratigraphy
- HNu/OVA scan

Each soil boring will be sampled at 5-foot intervals using a standard split-spoon sampler following ASTM Standard Method D1586 for the Standard Penetration Resistance Test. Each boring will be logged by an experienced CH2M HILL hydrogeologist or geotechnical engineer and described using the Unified Soil Classification System.

Samples will be collected using a standard 2-foot-long, 2-inch-O.D. split-spoon sampler. The HNu or OVA results, sample depth, and visual inspection will be used to select samples for laboratory analyses.

Subsurface soil samples will be collected from 8 to 24 borehole locations surrounding the landfill proper. An average of 3 samples per borehole will be collected (24 to

72 samples). Samples will be analyzed for grain size and Atterberg limits.

Ten subsurface soil samples will be selected from the borings for RAS parameters (volatile and semivolatile organic compounds, pesticides/PCBs, and metals) and SAS parameters (TOC). The samples will be selected from the screened intervals of monitoring wells MW2S, MW2M and MW3S and from the zones of greatest contamination in borings GB1, GB2, GB3, and GB4. The zone of greatest contamination will be judged based on visual evidence, HNu/OVA screening of soil samples, or the results of the CSL analysis of boring groundwater samples. Three background soil samples will also be collected and submitted for the same analyses. These background samples will be collected from the installation of MW1M. Table A-1 lists the parameters for which soil will be tested.

All sample jars will be sealed and stored in a cooler at 4°C.

TEST PIT SAMPLING

CAP INVESTIGATION TEST PITS

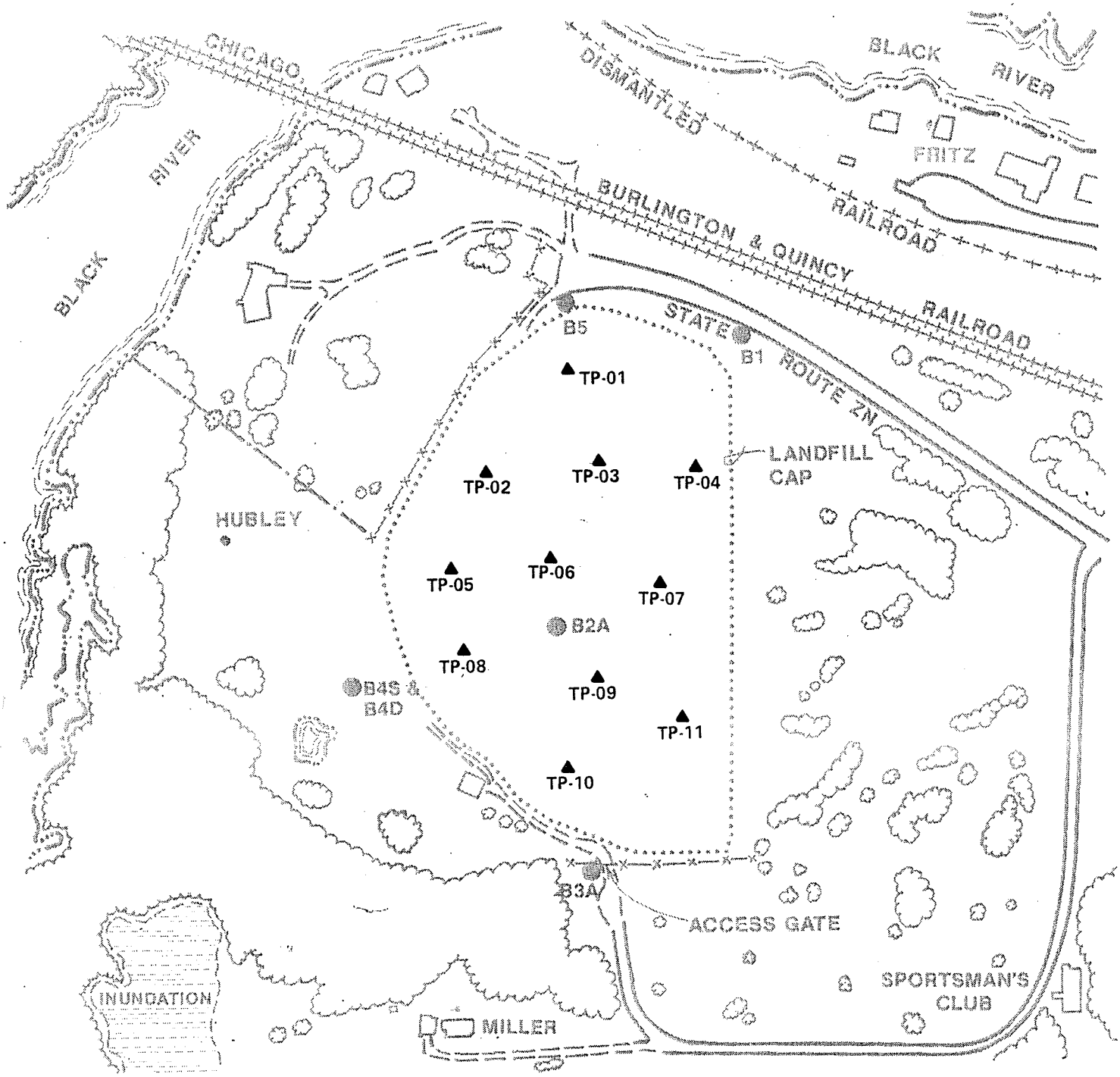
Eleven 2-foot-deep test pits will be excavated in the landfill cap to determine its integrity. Figure A-2 shows the proposed locations of the eleven test pits. One grab sample will be collected from each test pit. The sample will be analyzed for moisture content, grain size, shrinkage limits, and Atterberg limits. Three of the samples will be analyzed to determine the moisture-density relationship and permeability of the soil. The depth of the soil cap will be determined at each sampling location to estimate the infiltration capacity of the soil cover.

Shelby tube samples will also be collected at the test pit locations. They will be analyzed for in situ density, moisture content, and permeability.

SOLVENT DISPOSAL AREA TEST PITS

Thirty subsurface soil samples will be collected from three backhoe test pits in the solvent disposal area (Figure A-3).

All test pits will be excavated with a track-mounted backhoe equipped with a bucket. Each test pit will be approximately 20 feet long, 4 feet wide, and 10 to 15 feet deep. Actual test pit dimensions will be recorded on the test pit logs.



LEGEND

▲ SHALLOW TEST PIT LOCATIONS

FIGURE A-2
CAP INVESTIGATION LOCATIONS
ONALASKA LANDFILL FSP

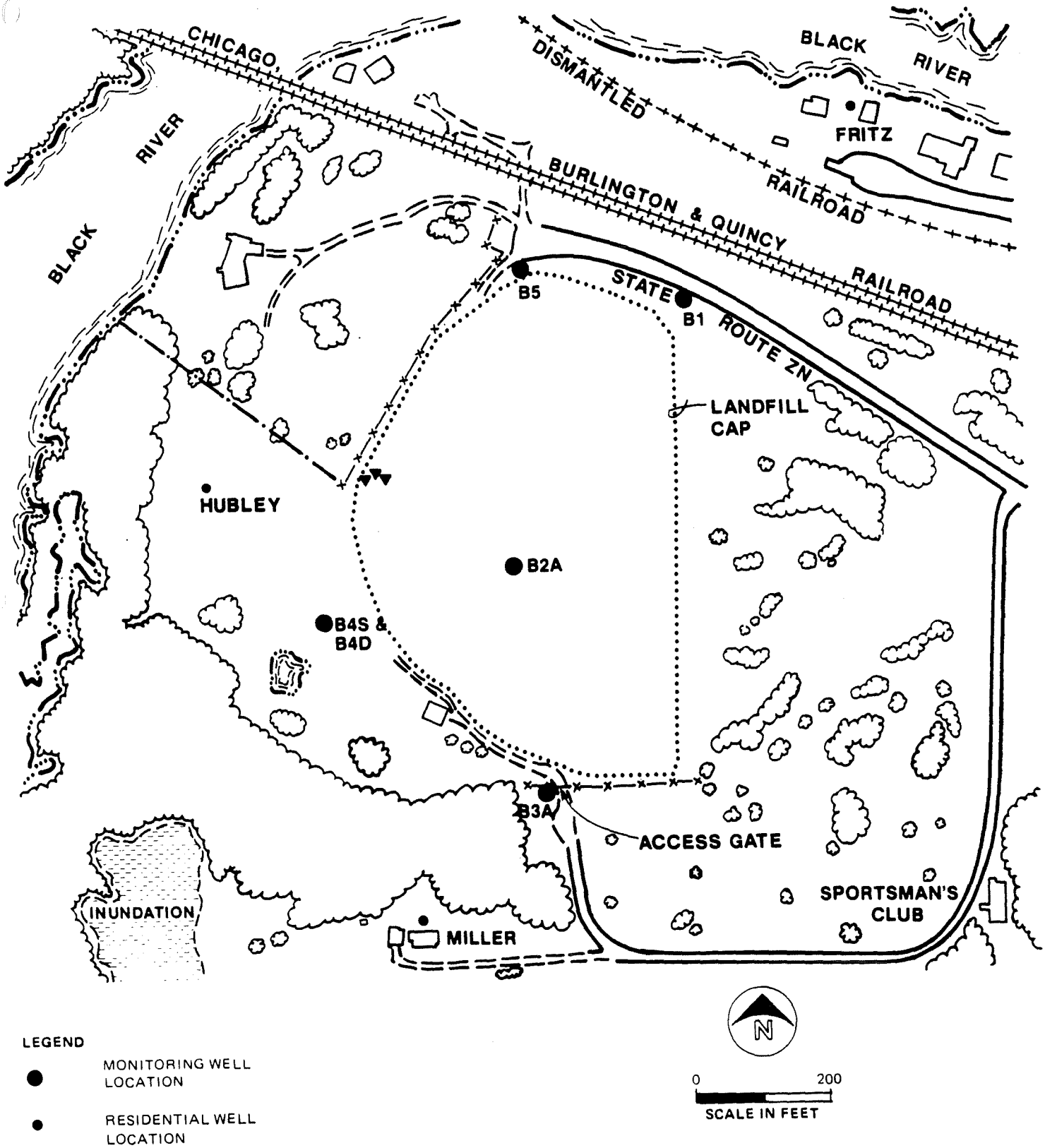


FIGURE A-3
SOLVENT DISPOSAL AREA
INVESTIGATION
ONALASKA LANDFILL FSP

Three sediment samples will be taken at each location. Each sample will be analyzed in the CSL for up to five target VOCs. Based on screening results, up to seven sediment samples will be sent to the CLP for VOC analysis. At each of the seven locations, samples for semivolatile organic compounds, pesticides, PCBs, and inorganic chemicals will be composited from the three sediment samples. The three samples will be located within 20 feet of each other and taken from cores of the upper 6 inches of sediment.

The remaining three samples will be collected along the eastern bank of the main channel of the Black River. One will be a background sample. The surface water sample will be taken within 6 inches of the bank at mid depth. The sediment samples will be taken from within 6 inches of the bank. Otherwise sampling and analysis of sediment samples will be as described above. Sediment samples to be submitted for VOC analysis shall be collected as grab samples.

MONITORING WELL INSTALLATION

Seventeen to thirty monitoring wells will be installed at the landfill (see Figure A-6). The proposed depth for each location is presented in Table A-3, but actual depth will be determined by the field hydrogeologist based on the stratigraphy and groundwater contamination identified in the geotechnical boring program.

Monitoring well nest MW1 will be installed upgradient of the landfill to establish upgradient (background) groundwater quality. Monitoring well nest MW2 will be installed through the landfill to evaluate groundwater quality and stratigraphy beneath the landfill. A third nest MW3 is proposed near the southern edge of the landfill to help evaluate the vertical distribution of contaminants at the downgradient edge of the landfill and to determine if a vertical gradient exists.

At least three of the remaining monitoring wells will be installed in geotechnical borings immediately after completion of the borings. The relative elevations of each monitoring well measuring point will be determined and water levels recorded.

The boreholes for the monitoring wells will be advanced using screened, hollow-stem augers (6.25-inch I.D.). The selected auger size will allow sufficient annular space

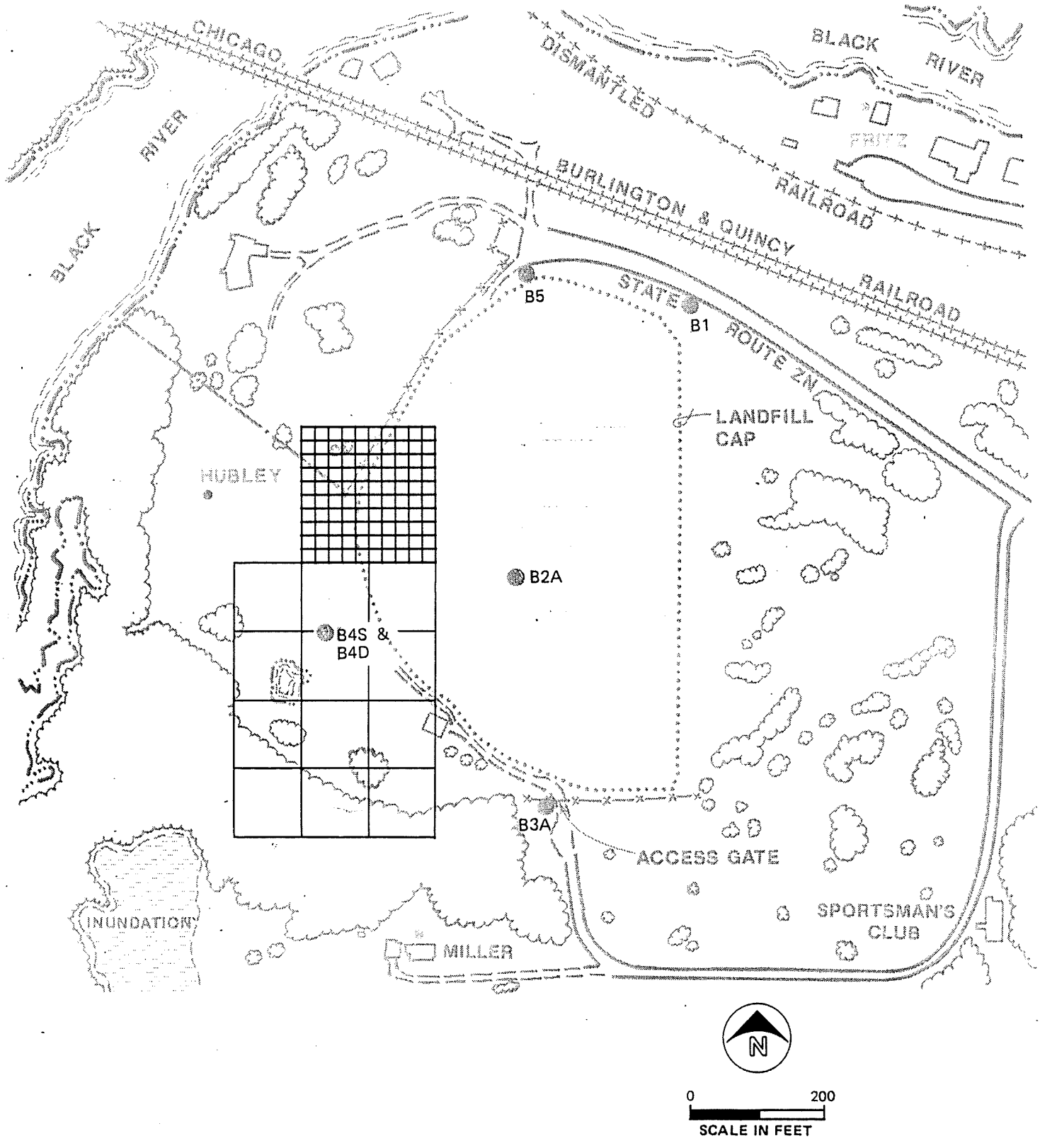
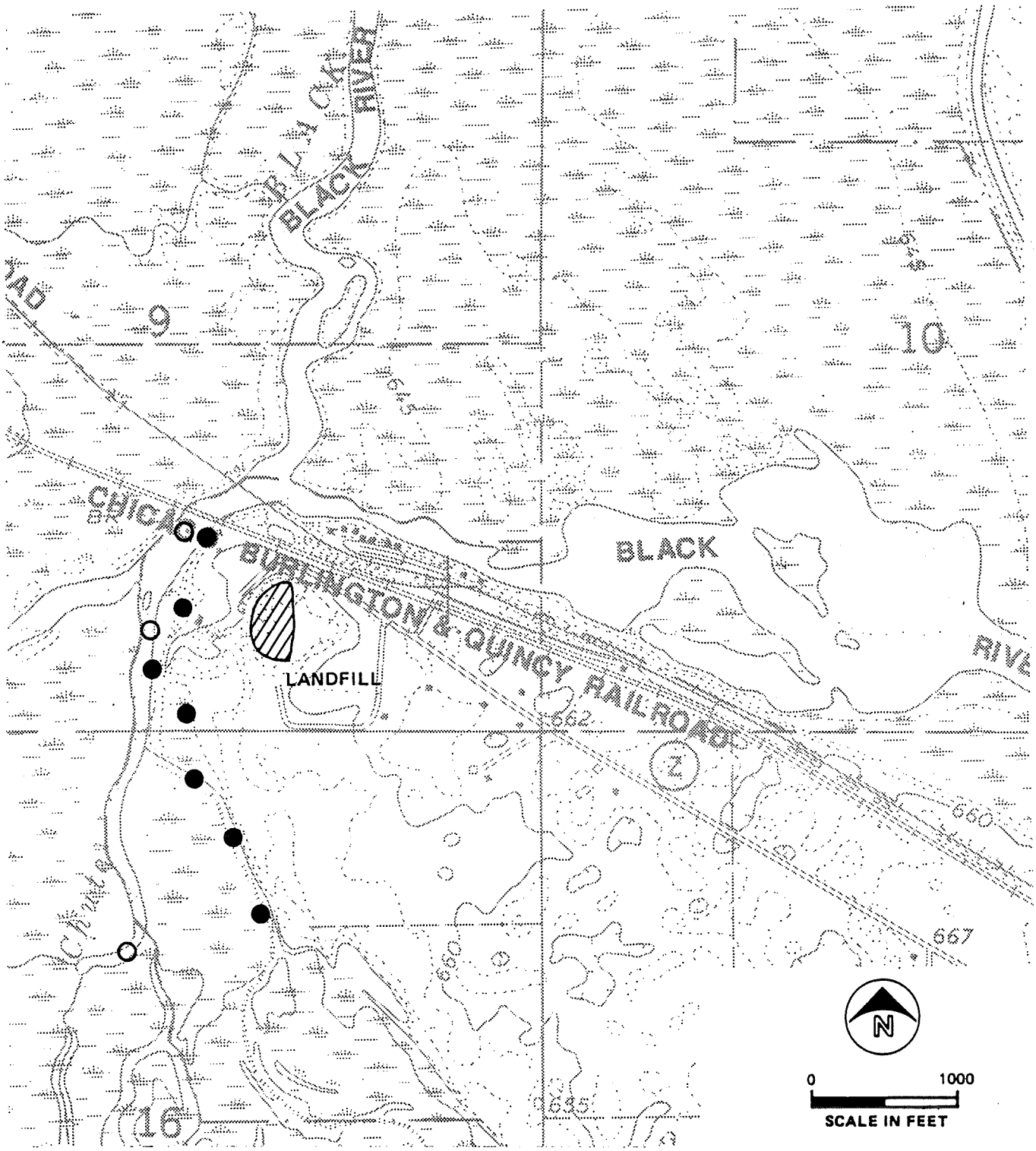


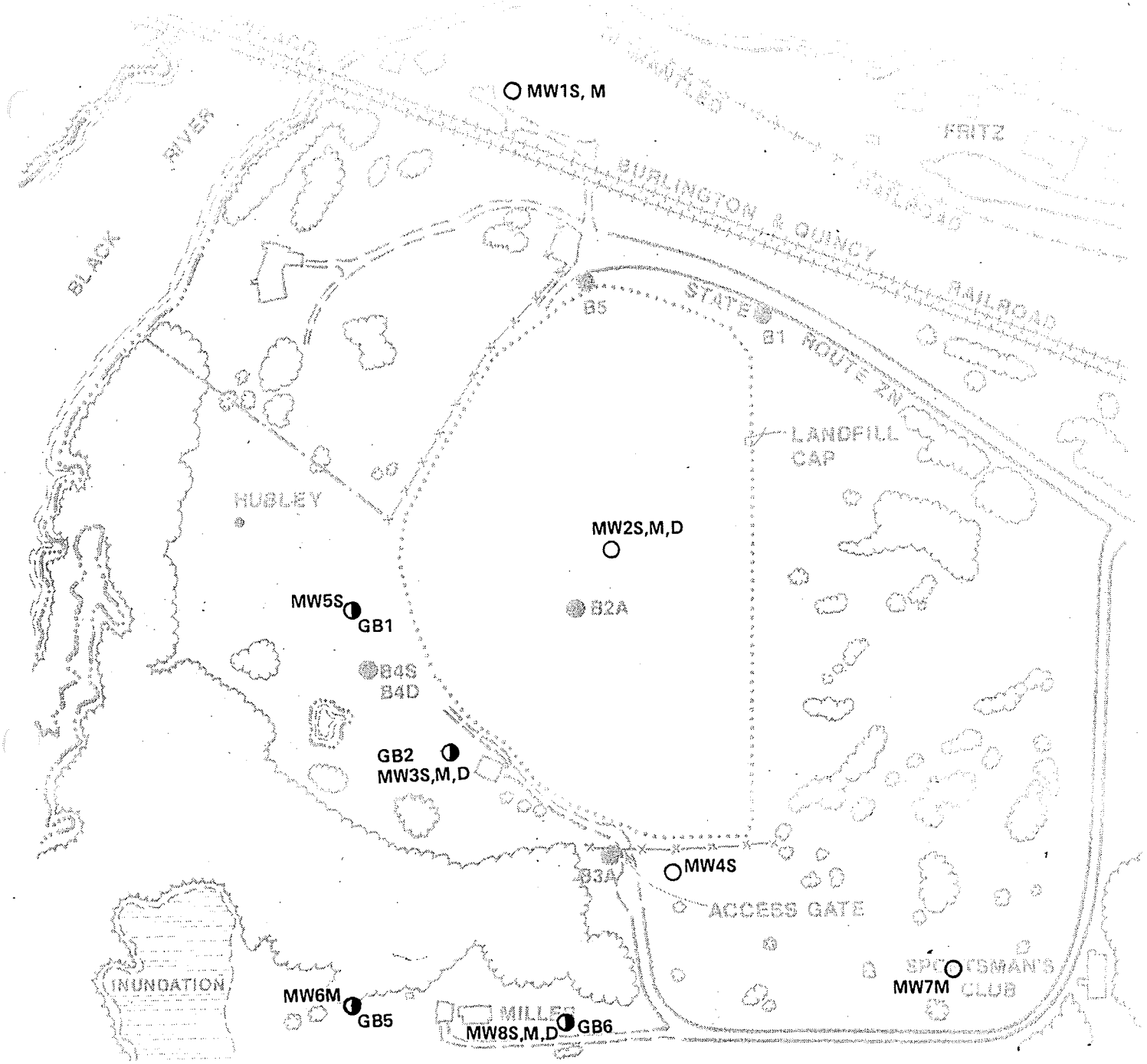
FIGURE A-4
SOIL GAS SURVEY GRID
ONALASKA LANDFILL FSP



LEGEND

- ○ SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS IN MAIN RIVER CHANNEL
- ● SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS IN IN STAGNANT WATER

FIGURE A-5
SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS
ONALASKA LANDFILL QAPP



LEGEND

- ○ MONITORING WELL
- ◑ MONITORING WELL INSTALLED IN GEOTECHNICAL BORING

FIGURE A-6
GROUNDWATER SAMPLING LOCATIONS
ONALASKA LANDFILL QAPP

Table A-3
RATIONALE FOR MONITORING WELL LOCATIONS

Well Number	Proposed Depth	Rationale
Monitoring Wells*		
MW-1S	45 Feet	To help determine upgradient groundwater quality (background)
MW-1M	90 Feet	
MW-2S	45 Feet	To help determine groundwater quality and stratigraphy beneath the landfill
MW-2M	90 Feet	
MW-2D	135 Feet	
MW-3S	45 Feet	To help determine downgradient groundwater quality and vertical contaminant distribution
MW-3M	90 Feet	
MW-3D	135 Feet	
MW-4S	45 Feet	To help determine downgradient groundwater quality
MW-5S	45 Feet	To help determine downgradient groundwater quality
MW-6M	70 Feet	To help determine westward extent of groundwater contamination
MW-7M	70 Feet	To help determine eastward extent of groundwater contamination
MW-8S	45 Feet	To help determine downgradient groundwater quality and vertical contaminant distribution
M	90 Feet	
D	135 Feet	
MW-9M	70 Feet	To help determine southerly extent of groundwater contamination
MW-10M	70 Feet	To help determine southerly extent of groundwater contamination

Note: Location of monitoring wells dependent upon findings of soil boring program.

between the well and the auger wall to introduce a filter pack (if necessary) and seal.

Wells installed in the landfill will be constructed of 2-inch threaded, flush joint, stainless steel riser pipe. All other monitoring wells will be constructed of 2-inch flush joint schedule 40 PVC with factory slotted 10-foot screens. All screen bottoms will be capped before installation, and the joints will be wrapped with Teflon thread tape to promote watertight seals. All riser pipes and screens will be cleaned with high pressure hot water before installation.

Each well will be fitted with a threaded or slip cap. Each well will have a protective casing and a minimum 2-foot stickup that will be painted a bright color to make it easily seen. Wells located in potentially high traffic areas will be surrounded by three concrete-filled, 4-inch diameter guard posts set at 120° angles to each other.

Each monitoring well will be developed until substantially free of sediment and until pH and conductivity are stable to the satisfaction of the CH2M HILL hydrogeologist. Wells will be developed using a 2-inch submersible pump.

GROUNDWATER SAMPLING

Seventeen new wells and eight geotechnical boring groundwater samples will be collected during drilling (Figure A-6). Two groundwater samples will be collected from the medium depth monitoring wells, and three samples will be collected from the deep monitoring wells. Samples will be collected using the same procedures employed for the geotechnical boring groundwater samples. The CSL will analyze the groundwater samples for indicator VOCS. Temperature, pH, and specific conductivity will be measured in the field. The sampling data will be used to analyze trends in contaminant migration and aid in the location of additional borings and in the placement of monitoring wells.

Two rounds of groundwater sampling will be conducted. Round 1 samples will be collected from the existing and proposed monitoring wells, for a total of 23 to 36 samples (Figure A-6). Round 2 sampling will take place about 2 months later.

As part of the Round 1 sampling, the CLP will conduct analyses for RAS parameters. As part of SAS, the CLP will analyze for biological oxygen demand, chemical oxygen

demand, total organic carbon, total dissolved solids, total phosphorus, alkalinity, chloride, ammonia, nitrate/nitrite, sulfate, sulfide, and total carbon. Three pure phase samples of floating product will be collected and analyzed for volatile and semivolatile organic compounds, pesticides, PCBs, and the constituents of naphtha.

As part of Round 2 sampling, samples will be analyzed for RAS parameters and for oil and grease (see Table 4-1). Temperature, pH, and specific conductivity will be measured for both rounds of groundwater sampling.

GROUNDWATER SAMPLE COLLECTION

Before each well is purged for sampling, a water level measurement will be taken to the nearest 0.01 foot using an electronic water level indicator. The well volume will be calculated and five well volumes will be removed during purging.

Each well will be purged immediately before sampling using a hand piston pump when the total head exceeds 20 feet or a peristaltic pump when the head is 20 feet or less. For each well purged with a peristaltic pump, a length of Teflon tubing sufficient to reach the well screen and a 2-foot section of silicone tubing (for the peristaltic pump head) will be dedicated to the well. The tubing will be elevated above the water table and affixed to the well cap between sampling events. Wells will be purged by pumping from as near the water table as possible to prevent stagnant water from remaining in the well. Wells will be sampled for all parameters except VOCs. Samples will be withdrawn from the screened portion of the well. Metals samples will be filtered in-line through a 0.45-micron membrane filter. The flow rate to the filter will be controlled with a stopcock on a branch line from the pump discharge line. Temperature, pH, and specific conductivity will be measured in a plastic container with well water flowing through. VOC samples will be taken with a bottom-filling bailer lowered to the screened section of the well. PVC bailers will be used and dedicated to each well where a floating layer is detected. Stainless steel bailers will be used in other wells. VOC sample vials will be filled directly from the bailer using a bottom-emptying device. Vials will not be rinsed with groundwater before filling.

SURFACE WATER SAMPLING

Surface water samples will be taken from the Black River immediately west of the site. One or two samples will be collected at each of the 10 locations shown in Figure A-5. The samples will be collected near the water/sediment interface. Surface water will be analyzed for indicator VOCS at the CSL and for RAS parameters by the CLP laboratory. Samples will also be field tested for pH, specific conductivity, and temperature.

Surface water samples will be collected by submerging the sample container at mid-depth. The mouth of the container will be positioned so that it faces downstream to avoid collecting suspended detritus, and sampling personnel will stand downstream so that sediment they may stir up will not be collected. Sample collection will begin at the downstream location and proceed upstream. The main channel samples will be collected at mid-depth and within 6 inches of the east bank of the Black River.

RESIDENTIAL WELL SAMPLING

Nine residential wells (Figure A-7) will be sampled to determine if the water supply presents a potential hazard to human health. In the event that some of the residential wells are contaminated, a second round of samples will be collected. The samples will be analyzed by the CLP laboratory for SAS parameters (volatile and semivolatile organic compounds, pesticides, PCBs, and metals) with low detection limits. Temperature, pH, and specific conductivity will be measured in the field.

Before a residential well is sampled, a tap will be turned on and allowed to run for at least 10 minutes (if possible). Samples will be collected directly from a tap that is upstream of (or bypasses) any water softeners or filters. Any aerators on the tap or faucet will be removed.

DECONTAMINATION PROCEDURES

SOIL BORING EQUIPMENT

Between boreholes, all casing, rods, samplers, and other equipment used in the boreholes will be decontaminated. The cleaning process will consist of steam cleaning or hosing the drilling equipment with a high pressure hot water (50 to 60°C) rinse. After cleaning, the drilling equipment will be placed on a clean surface to air dry.

All drilling equipment, including the drill rig, casings, rods, tools, and miscellaneous equipment, will also be decontaminated before leaving the site.

SAMPLING EQUIPMENT

Sampling equipment must be decontaminated in the following way before a new sample can be taken:

1. Brush off visible debris or dirt, then scrub wash with clean water. Tap water may be used for this initial washing.

2. Scrub and wash equipment with a 2.5 percent (weight/volume) solution of trisodium phosphate and water.

3. Next, scrub and wash equipment with a 10 percent (volume/volume) solution of methanol and water.

4. Rinse with deionized or distilled water.

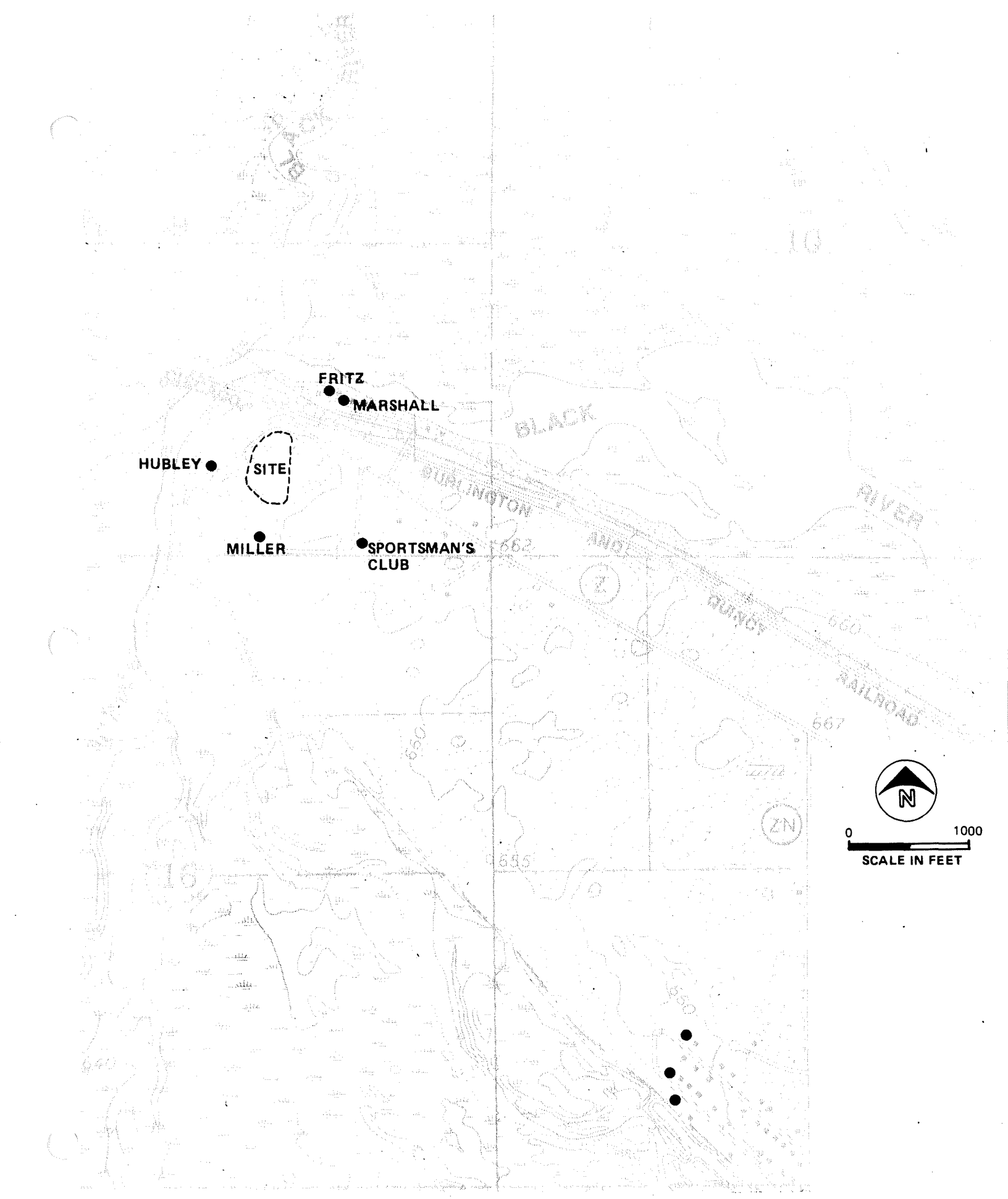
5. Finally, place equipment on a clean plastic sheet and allow to air dry.

FIELD BLANKS

Field blanks will be collected to check the effectiveness of sampling equipment decontamination practices. For soil, test pit, and sediment sampling, the blanks will be prepared by collecting clean, washed sand with the same decontaminated trowel, scoop, or spoon used to collect other samples.

For groundwater sampling, the blanks will be obtained by pouring deionized water directly into the bailer and transferring it into the appropriate sampling container. The blanks will then be preserved as specified in Table A-4. Blanks for samples collected with the pump will be passed through the pump, tubing, and filter apparatus, if applicable.

Field blanks for surface water and residential well sampling will be obtained by pouring deionized water directly into the appropriate sampling containers and preserving them.



LEGEND

● RESIDENTIAL WELL SAMPLING LOCATION

FIGURE A-7
RESIDENTIAL WELL SAMPLING LOCATIONS
ONALASKA LANDFILL QAPP

TABLE A-4

CONTAINERS, PRESERVATIVES, AND REQUIRED SAMPLE AMOUNTS

Matrix	Parameter	Required Container Type	Required Sample Amount	Preservative	Holding Time
GROUNDWATER	Volatile Organics	Two 40-ml Glass Vials w/Teflon Seal	Fill Completely, No Headspace	Iced to 4 C	10 days
	Semivolatiles	4-L Amber Bottle w/Teflon Lined Caps	Fill to Shoulder	Iced to 4 C	Extraction within 5-days, analyze within 40 days
	Pesticides & PCBs	4-L Amber Bottle w/Teflon Lined Caps	Fill to Shoulder	Iced to 4 C	Extraction within 5-days, analyze within 40 days
	Metals	1-Liter Polyethylene Bottle	Fill to Shoulder	HNO3 to pH < 2 Iced to 4 C	6 months
	Mercury	1-Liter Polyethylene Bottle	Fill to Shoulder	HNO3 to pH < 2 Iced to 4 C	14 days
	Oil & Grease	1-Liter Glass Bottle w/Aluminum Foil Under Cap	Fill to Shoulder	Add 2 ml/l of H2SO4 to pH < 2 Iced to 4 C	10 days
	Total Organic Carbon	1-Liter Polyethylene Bottle	Fill to Shoulder	Add 1 ml/l of H2SO4 to pH < 2 Iced to 4 C	28 days
	Biochemical Oxygen Demand	1-Liter Polyethylene Bottle	Fill to Shoulder	Iced to 4 C	48 hours
	Chemical Oxygen Demand Nitrate & Nitrite	1-Liter Polyethylene Bottle	Fill to Shoulder	Add 1 ml/l of H2SO4 to pH < 2 Iced to 4 C	28 days
	Total Phosphorus	1-Liter Polyethylene Bottle	Fill to Shoulder	Add 1 ml/l of H2SO4 to pH < 2 Iced to 4 C	28 days
	Total Suspended Solids Total Dissolved Solids Alkalinity	1-Liter Polyethylene Bottle	Fill to Shoulder	Iced to 4 C	7 days 7 days 14 days
	Ammonia	1-Liter Polyethylene Bottle	Fill to Shoulder	Add 1 ml/l of H2SO4 to pH < 2 Iced to 4 C	28 days
	Sulfate	1-Liter Polyethylene Bottle	Fill to Shoulder	Iced to 4 C	28 days
	Sulfide	1-Liter Glass Bottle	Fill Completely, No Headspace	Add 1.5 ml/l of 2N Zn(acac) NaOH to pH < 11 and PH > 9 Iced to 4 C	7 days
	Chloride	1-Liter Polyethylene Bottle	Fill to Shoulder	Iced to 4 C	28 days
	Pure Product (Naphtha)	Two 40-ml Glass Vials w/Teflon Seal	Fill Completely, No Headspace	Iced to 4 C	7 days

TABLE A-4

CONTAINERS, PRESERVATIVES, AND REQUIRED SAMPLE AMOUNTS

Matrix	Parameter	Required Container Type	Required Sample Amount	Preservative	Holding Time
SUBSURFACE SOIL	Volatile Organics	120-ml Wide Mouth Glass Vials	Fill Completely, No Headspace	Iced to 4 C	10 days
	Semivolatiles	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Extraction within 10-days, analyze within 40 days
	Pesticides & PCBs	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Extraction within 10-days, analyze within 40 days
	Metals	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	6 months
	Mercury	8-oz. Wide Mouth Glass Jar	Fill to Shoulder	Iced to 4 C	26 days
	Total Organic Carbon	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	28 days
	Oil & Grease	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Grain Size Analysis	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Atterberg Limits	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Heating Value	4-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Percent Ash	4-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Percent Moisture	4-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Sulfur Content	4-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	Total Chlorine	4-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days
	EPTOX	8-oz. Wide Mouth Glass Jar	Fill 3/4 Full	Iced to 4 C	Analyze within 40 days

TABLE A-4

CONTAINERS, PRESERVATIVES, AND REQUIRED SAMPLE AMOUNTS

Matrix	Parameter	Required Container Type	Required Sample Amount	Preservative	Holding Time
SEDIMENT	Volatile Organics	120-ml Wide Mouth Glass Vials	Fill Completely, No Headspace	Iced to 4 C	10 days
	Semivolatiles	8-oz. Wide Mouth Glass jar	Fill 3/4 Full	Iced to 4 C	Extraction within 10-days, analyze within 40 days
	Pesticides & PCBs	8-oz. Wide Mouth Glass jar	Fill 3/4 Full	Iced to 4 C	Extraction within 10-days, analyze within 40 days
	Metals	8-oz. Wide Mouth Glass jar	Fill 3/4 Full	Iced to 4 C	6 months
	Mercury	1-Liter Polyethylene Bottle	Fill 3/4 Full	Iced to 4 C	26 days

TABLE A-4

CONTAINERS, PRESERVATIVES, AND REQUIRED SAMPLE AMOUNTS

Matrix	Parameter	Required Container Type	Required Sample Amount	Preservative	Holding Time
SURFACE WATER	Volatile Organics	Two 40-ml Glass Vials w/Teflon Seal	Fill Completely, No Headspace	Iced to 4 C	10 days
	Semivolatiles	4-L Amber Bottle w/Teflon Lined Caps	Fill to Shoulder	Iced to 4 C	Extraction within 5-days, analyze within 40 days
	Pesticides & PCBs	4-L Amber Bottle w/Teflon Lined Caps	Fill to Shoulder	Iced to 4 C	Extraction within 5-days, analyze within 40 days
	Metals	1-Liter Polyethylene Bottle	Fill to Shoulder	HNO_3 to pH < 2 Iced to 4 C	6 months
	Mercury	1-Liter Polyethylene Bottle	Fill to Shoulder	HNO_3 to pH < 2 Iced to 4 C	14 days

TRIP BLANKS

Trip blanks are used as a means of monitoring the influence of outside elements that could contaminate samples during sampling shipping and site sampling. Trip blanks will be prepared for groundwater, surface water, and residential well samples at CH2M HILL's Milwaukee warehouse for VOA water samples only.

At the warehouse, chain-of-custody seals will be broken on boxes containing VOA vials. A set of vials will then be filled with deionized water only. There should be no air bubbles in the trip blanks. The VOA vials will then be returned to the appropriate box, and new chain-of-custody seals will be affixed. The trip blanks will be sent to the site as part of the project bottle shipment.

At the site, the new chain-of-custody seals will be broken, and each trip blank will be carried to the well head where it will remain during sampling. After a day's sampling effort has been completed, the sample batch, including the trip blank, will be sent to the laboratory. One trip blank will accompany each cooler containing VOA samples.

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Table A-5 (Page 1 of 4)
TARGET COMPOUND LIST AND
CONTRACT REQUIRED QUANTIFICATION LIMITS

Volatiles	CAS Number	Quantification Limits Low Concentration Analysis[a] Water (ug/l)	Soil/Sediment[b] (ug/kg)
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-34-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-2	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. Cis-1,3-Dichloropropene	10061-02-5	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. Trans-1,3-Dichloropropene	10061-01-6	5	5
26. Bromoform	75-25-2	5	5
27. 2-Hexanone	591-78-6	10	10
28. 4-Methyl-2-pentanone	108-10-1	10	10
39. Tetrachloroethene	127-18-4	5	5
30. Toluene	108-88-3	5	5
31. Chlorobenzene	108-90-7	5	5
32. Ethyl Benzene	100-41-4	5	5
33. Styrene	100-42-5	5	5
34. Xylenes (total)	133-02-7	5	5
Semivolatiles			
35. Phenol	108-95-2	10	330
36. bis(2-Chloroethyl)ether	111-44-4	10	330
37. 2-Chlorophenol	95-57-8	10	330
38. 1,3-Dichlorobenzene	541-73-1	10	330
39. 1,4-Dichlorobenzene	106-46-7	10	330
40. Benzyl Alcohol	100-51-6	10	330
41. 1,2-Dichlorobenzene	95-50-1	10	330
42. 2-Methylphenol	95-48-7	10	330
43. bis(2-Chloroisopropyl)ether	39638-32-9	10	330

Note: Specific quantification limits are highly matrix dependent. The quantification limits listed herein are provided for guidance and may not always be achievable.

[a] Quantification limits listed for soil/sediment are based on wet weight. The quantification limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the

[b] contract, will be higher.
Medium Soil/Sediment Contract Required Quantification Limits (CRQL) for Volatile TCL Compounds are 100 times the individual Low Soil/Sediment CRQL; for Semivolatile TCL Compounds they are 60 times the individual Low Soil/Sediment CRQL.

Semivolatiles (Continued)	CAS Number	Quantification Limits Low Concentration Analysis[a] Water (ug/l)	Soil/Sediment[b] (ug/kg)
44. 4-Methylphenol	106-44-5	10	330
45. N-Nitroso-Dipropylamine	621-64-7	10	330
46. Hexachloroethane	67-72-1	10	330
47. Nitrobenzene	98-95-3	10	330
48. Isophorone	78-59-1	10	330
49. 2-Nitrophenol	88-75-5	10	330
50. 2,4-Dimethylphenol	105-67-9	10	330
51. Benzoic Acid	65-85-0	50	1,600
52. bis(2-Chloroethoxy)methane	111-91-1	10	330
53. 2,4-Dichlorophenol	120-83-2	10	330
54. 1,2,4-Trichlorobenzene	120-82-1	10	330
55. Naphthalene	91-20-3	10	330
56. 4-Chloroaniline	106-47-8	10	330
57. Hexachlorobutadiene	87-68-3	10	330
58. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
59. 2-Methylnaphthalene	91-57-6	10	330
60. Hexachlorocyclopentadiene	77-47-4	10	330
61. 2,4,6-Trichlorophenol	88-06-2	10	330
62. 2,4,5-Trichlorophenol	95-95-4	50	1,600
63. 2-Chloroanaphthalene	91-58-7	10	330
64. 2-Nitroaniline	88-74-4	50	1,600
65. Dimethyl Phthalate	131-11-3	10	330
66. Acenaphthylene	208-96-8	10	330
67. 2,6-Dinitrotoluene	606-20-2	10	330
68. 3-Nitroaniline	99-09-2	50	1,600
69. Acenaphthene	83-32-9	10	330
70. 2,4-Dinitrophenol	51-28-5	50	1,600
71. 4-Nitrophenol	100-02-7	50	1,600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. Diethylphthalate	84-66-2	10	330
75. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
76. Fluorene	86-73-7	10	330
77. 4-Nitroaniline	100-01-6	50	1,600
78. 4,6-Dinitro-2-methylphenol	534-52-1	50	1,600
79. N-nitrosodiphenylamine	86-30-6	10	330
80. 4-Bromophenyl Phenyl ether	101-55-3	10	330
81. Hexachlorobenzene	118-74-1	10	330
82. Pentachlorophenol	87-86-5	50	1,600
83. Phenanthrene	85-01-8	10	330
84. Anthracene	120-12-7	10	330
85. Di-n-butylphthalate	84-74-2	10	330
86. Fluoranthene	206-44-0	10	330
87. Pyrene	129-00-0	10	330
88. Butyl Benzyl Phthalate	85-68-7	10	330

Note: Specific quantification limits are highly matrix dependent. The quantification limits listed herein are provided for guidance and may not always be achievable.

[a] Quantification limits listed for soil/sediment are based on wet weight. The quantification limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

[b] Medium Soil/Sediment Contract Required Quantification Limits (CRQL) for Semivolatile TCL Compounds are 60 times the individual Low Soil/Sediment CRQL.

Semivolatiles (Continued)	CAS Number	Quantification Limits Low Concentration Analysis[a] Water (ug/l)	Soil/Sediment[b] (ug/kg)
89. 3,3'-Dichlorobenzidine	91-94-1	20	660
90. Benzo(a)anthracene	56-55-3	10	330
91. Chrysene	218-01-9	10	330
92. bis(2-ethylhexyl)phthalate	117-81-7	10	330
93. Di-n-octyl Phthalate	117-84-0	10	330
94. Benzo(b)fluoranthene	205-99-2	10	330
95. Benzo(k)fluoranthene	207-08-9	10	330
96. Benzo(a)pyrene	50-32-8	10	330
97. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
98. Dibenz(a,h)anthracene	53-70-3	10	330
99. Benzo(g,h,i)perylene	191-24-2	10	330
Pesticides/PCBs			
100. alpha-BHC	319-84-6	0.05	8.0
101. beta-BHC	319-85-7	0.05	8.0
102. delta-BHC	319-86-8	0.05	8.0
103. gamma-BHC (Lindane)	58-89-9	0.05	8.0
104. Heptachlor	76-44-8	0.05	8.0
105. Aldrin	309-00-2	0.05	8.0
106. Heptachlor Epoxide	1024-57-3	0.05	8.0
107. Endosufan I	959-98-8	0.05	8.0
108. Dieldrin	60-57-1	0.10	16.0
109. 4,4'-DDE	72-55-9	0.10	16.0
110. Endrin	72-20-8	0.10	16.0
111. Endosulfan II	33213-65-9	0.10	16.0
112. 4,4'-DDD	72-54-8	0.10	16.0
113. Endosulfan Sulfate	1031-07-8	0.10	16.0
114. 4,4'-DDT	50-29-3	0.10	16.0
115. Endrin Ketone	53494-70-5	0.10	16.0
116. Methoxychlor	72-43-5	0.5	80.0
117. Alpha-chlordane	5103-71-9	0.05	80.0
118. gamma-chlordane	5103-74-2	0.05	80.0
119. Toxaphene	8001-35-2	1.0	160.0
120. PCB Arochlor-1016	12674-11-2	0.5	80.0
121. PCB Arochlor-1221	11104-28-2	0.5	80.0
122. PCB Arochlor-1232	11141-16-5	0.5	80.0
123. PCB Arochlor-1242	53469-21-9	0.5	80.0
124. PCB Arochlor-1248	12672-29-6	0.5	80.0
125. PCB Arochlor-1254	11097-69-1	1.0	160.0
126. PCB Arochlor-1260	11096-82-5	1.0	160.0

Note: Specific quantification limits are highly matrix dependent. The quantification limits listed herein are provided for guidance and may not always be achievable.

[a] Quantification limits listed for soil/sediment are based on wet weight. The quantification limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

[b] Medium Soil/Sediment Contract Required Quantification Limits (CRQL) for Semivolatile TCL Compounds are 60 times the individual Low Soil/Sediment CRQL; for Pesticide/PCB TCL Compounds they are 15 times the individual Low Soil/Sediment CRQL.

Table A-5 (Page 4 of 4)

Inorganic Target Analyte	Quantification Limit Low Concentration Analysis[a] (ug/l)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5,000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5,000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5,000
Selenium	5
Silver	10
Sodium	5,000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

[a]The quantification limits for samples may be considerably higher depending on the sample matrix.

GLT824/68

Appendix B
SPECIAL ANALYTICAL SERVICES

5/002__-0-6/87 Alkalinity (H_2O) 6/29/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved For Scheduling
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name:

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 waters (surface waters, groundwaters, drinking waters, leachates, etc.). Samples will be unfiltered. Reports are reported as 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 concentration):

Analyze 44 groundwater samples for alkalinity.

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: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

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

1) Alkalinity EPA Method 310.1 (Titrimetric, ph 4.5) 2) Standard Methods, 16th Edition, Method 403 4c and 4d.

Samples will be stored at 4°C until analysis and validation of results.

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

Samples holding time should not exceed 14 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity > 20 mg/l as $CaCO_3$. For concentrations <20 mg/l, use EPA Method 310.1 (Section 6.3) or Standard Methods, Method 403 4d. Do not use titrant volumes greater than 50ml. Obtain approval of CPMS, CRL prior to use of any other method.

Use Na_2CO_3 to standardize titrant. Standardize the pH meter and the titrant each day.

Standardize the pH meter using at least two buffers which bracket the end point.

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 the order of analysis including pH meter calibration, titrant standardization, lab blanks, samples, lab control standards, duplicates, etc., with resulting titrant volumes or readouts will be provided along with calculation worksheets. All records will be legible and sufficient to recalculate all sample concentrations and QA audit results. Report method of titrant standardization.
EPA QC Reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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	2 mg/l for low level	± 2 mg/l for Conc. < 20 mg/l $CaCO_3$
	20 mg/l for high level	± 10% for Conc. > 20 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on allowable methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. The QA audits below will be done for each group of low-level and high-level akalinity determinations.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
lab blank	at least 1 per group of 10 or fewer samples	<10 mg/l for high-level samples tested. ≤2 mg/l≤ for low-level samples tested.
lab duplicate	at least 1 per group of 10 or fewer samples	± 10% or ± 2 mg/l
lab control sample 1 set of EPA QC mineral reference samples	1 per sample set	90-110% recovery.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanlyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

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.

5/013__-0-6/87 Ammonia in water 6/30/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling _____ _____

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

E. Site Name: Onalaska Sanitary Landfill

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 ammonia in waters (surface waters, ground waters, drinking waters, etc.) All samples will be unfiltered. Results will be reported as mg/l N.

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 concentration):

Analyze 44 groundwater samples for ammonia.

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: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

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

1) EPA Method 350.1 (Automated Phenate), or

2) EPA Method 350.3 (Potentiometric, Ion Selective Electrode).

Samples will be stored at 4° C until analysis and validation of results. Sample aliquots will be preserved in the field with sulfuric acid (1 ml/l to pH < 2).

The working concentration range of Method 350.1 Auto Analyzer should be 0.1 to 10 mg/l NH_3-N or lesser concentration.

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

Check sample pH (wide range pH paper). If pH >2 contact Jay Thakkar, CPMS, CRL for instructions. Dilute and rerun samples with peak heights or concentrations higher than the highest standard. The holding time is not to exceed 28 days from sample collection. All solutions should be made with amonia-free water. For Method 350.3 calibrate the electrometer with standards in order of increasing concentration of ammonia. The pH of the solution after the addition of NAOH must be above 11. Use only the method(s) specified above. Standard curve for Method 350.1 must include at least 5 standards (one of which is zero concentration). Standard curve for Method 350.3 must include at least 4 standards between 0.1 and 10.0 mg/l NH3-N. All standards, blanks, dilution water, and diluted samples must be acidified with 1 ml/l H_2SO_4.
Analyze MS and MSD as specified by Section II of the QC Requirements.

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 the order of calibration standards, lab blanks, samples, lab control standards, spikes, duplicate, etc. with resulting peak heights, millivolts, or concentration readouts, will be provided along with copies of worksheets used to calculate ammonia results. If Method 350.3 is used, the standard curve should be provided. A photocopy of the instrument readout i.e. strip charts, printer tapes, etc. must be included. All records analyses and calculation must be legible and sufficient to recalculate all concentrations. Results are to be in mg/-N per liter.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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.)
Ammonia	0.1 mg/l-N	Duplicate results must agree to within 10% for concentrations ≥ 1mg/l or to within 0.1mg/l for concentrations <1 mg/l Results will be reported to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1/mg/l-N.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

GENERAL STATEMENT

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

a) For Method 350.1

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± 10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤0.1 mg/l
Calibration verification	1 per group of 10 samples	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2	1 per sample set	85% - 115%

b) For Method 350.3

Lab Duplicate	at least 1 per group of 10 or fewer samples	10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤ 0.1 mg/l
Calibration verification standard	1 per 10 samples and end of set	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2	1 per sample set	85% - 115%

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Jay Thakkar (312) 886-1972) or Chuck Elly (312) 353-9087.

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.

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

SAS Number

Approved for Scheduling

SPECIAL ANALYTICAL SERVICES
Client Request

[ ] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Onalaska Sanitary Landfill

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 biological oxygen demand (BOD) in water and wastewater. Samples will be unfiltered. Results are reported as mg/l oxygen.

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 concentration):

Analyze 44 groundwater samples for biological oxygen demand (BOD).

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: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

_____

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

BOD "Standard Methods for the Examination of Water and Wastewater" 15th or 16th Edition, Method 507. All samples will be seeded unless otherwise stated.

_____

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

Set-up 3 or more sample dilutions so that two or more sample dilutions overlap to result in a residual D.O. > or = to 1 mg/l and a D.O. depletion ≥ 2 mg/l. Measure the seed BOD using 2 or more dilutions (Section 5d). BOD results for 2 dilutions should agree within + or - 15%. Analyze unseeded dilution water blanks , and glucose-glutamic acid checks (Section 5b of Method 507), both in duplicate, in addition to sample dilutions. Determine the initial and final D.O. for each bottle. Store samples at 4°C until analysis. The holding time is not to exceed 48 hours from the time of the beginning of sample collection. Dilution water will be seeded so that calculated DO uptake from BOD of seed will be between 0.6 and 1.0 mg/l (Section 5d of Method 507). Do not use seeded blanks to estimate seed corrections. All procedures defined in the Method must be followed precisely. Check for interferences (Section 5e). Analyze MS and MSD as dictated by Section II of the QC requirements.

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.

All measurements and calculations must be documented and submitted. Submit all raw data. Report initial and final D.O. from each bottle. Report BOD in mg/l for each bottle and the average of each fitting the depletion range listed above using calculations specified by "Standard Methods"(Section 6 of Method 507). Report results of duplicates, unseeded dilution water blank, BOD of seed, calculated DO uptake of seed in seeded dilution water, and glucose-glutamic acid check.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis will be providec for all reference samples used.

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.)
BOD	2 mg/l	Differences in duplicate series of sample results shall not exceed 2 mg/l for concentrations less than 20mg/l.

II. QC REQUIREMENTS Do not use any field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Glucose-Glutamic acid checks	1 pair per set of samples	160-240 mg/l
Duplicate (full dilution series)	at least 1 per group of 10 or fewer samples	+ or -(10% or 2 mg/l)
Unseeded Dilution Water Blanks	1 pair per set of samples, including 1 pair for each lot of dilution water	< or = to 0.2 mg/l
DO Uptake of seed in seeded dilution water (calculated)	calculated for each lot of seeded dilution water	0.6 to 1.0 mg/l
1 set of EPA QC Demand Reference Samples (if specified) Yes x No ___	1 set of 2 per sample set	75 - 125% Recovery

EPA will provide the QC samples

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

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.

5/005__-0-7/87 Chloride in Water 7/30/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling _____ _____

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312)353-2720

D. Date of Request: _____

E. Site Name: Onalaska Sanitary Landfill

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 chloride in water (surface waters, groundwater, drinking water, leachate, etc.). Samples will be unfiltered. This SAS is meant for routine monitoring of waters at a waste site. A A companion SAS is available for more precise, accurate, and sensitive chloride measurements in water. Results are reported as mg/l Cl.

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 concentration):

Analyze 44 groundwater samples for chloride.

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: Daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples:

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

1. EPA Method 325.1 (Colorimetric, Automated Ferricyanice, AA-I) 1983ed., or

2. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA-II) 1983ed., or
Note: A Region V CRL Auto Analyzer Manifold is attached for Method 325.2 to correct errors in Method 325.2's manifold diagram.

3. ASTM Colorimetric Method (Manual Method) -ASTM D 512C-81, or
4. Method 407C (Potentiometric Titration) Standard Methods, 16th ed. Samples will be kept at 4°C until analysis and validation of results.

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

For colorimetric methods (1) use a standard curve between 0 and 300 mg/l or less,(2) the calibration curve must include 5 points or more (including a zero concentration standard), and (3) samples with absorbances or peak heights greater than highest standard must be diluted and reanalyzed. For titrimetric ethod 1) use either 0.0141 or 0.025 N titrant, 2) automated potentiometric titrators are acceptable, 3) do not use more than 20 ml titrant for 50 ml or 100 ml sample aliquots, 4) dilute and reanalyze any sample aliquots requiring more than 20 ml titrant, 5) remove any interfering chromate, ferric iron, sulfide, and sulfite, and 6) standardize titrants daily. Obtain approval of CPMS, CRL prior to use of any other method. Analyze MS and MSD as specified by SEction II of the QC Requirements.

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. For the colorimetric methods, 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 worksheets used to calculate results. For the titration method, any potentiometric titration curves and all bench records tabulating titrant standardization, samples, aliquot volumes, matrix spikes, etc. will be provided. Records of titrant standardization and titrant blanks will be provided. A photocopy of instrument readouts, ie. strip charts, printer tapes, etc. must be included for all analyses. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA audit results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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.)
Chloride	5 mg/l	Differences in duplicate sample results are to be <5 mg/l for concentrations <50 mg/l and are to be < 10% for concentrations exceeding 50 mg/l. The significant figures to report depend on sensitivity of colorimetric curve or number of significant figures in titrant volume.
Note: These are minimum requirements Report actual detection limit used, based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
a) For Methods 325.1, 325.2, and ASTM D 512C		
Matrix Spike*	1 per group of 10 or fewer samples	85 - 115% Recovery
Lab Duplicate	" "	± (10% or 5 mg/l)
Lab Blank	" "	<5 mg/l
Calibration Verification Std.	" "	90-110% Recovery
1 Set of EPA QC Mineral Ref. Samples - 2 Concentrates	1 per sample set	85-115% Recovery
b) For Method 407C		
Same as Item IIa for Matrix Spike*, Lab Duplicate, and QC Mineral Reference Samples.		
Lab Blank (Not Titration Blank)	Beginning and end of sample set	-3 to +3 mg/l
Calibration verification Standard (Same as Titrant Standardazation)	At end of sample set	95 - 105% Recovery

*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.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Jay Thakkar (312) 886-1972 or Charles T. Elly (312) 353-9087.

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.

5/018__-0-6/87 COD (Hi- and Lo-levels) 6/26/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request Approved for Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Onalaska Sanitary Landfill

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 chemical oxygen demand (COD) (Hi-level) (50 - 800 mg/l) and COD (Lo-level) (5 - 50 mg/l) in water (surface water, groundwater, leachate, wastewater, drinking water, etc.). Samples will be unfiltered. Any sample with COD values less than 50 mg/l will be determined and reported as COD (Lo-level). Samples with COD values greater than or equal to 50 mg/l will be determined and reported as COD (Hi-level). The COD (Lo-level) method is used for optimum precision and accuracy of measurement of low concentration COD values.

If field specific conductance values are greater than or equal to 5000 umhos/cm, field personnel will indicate this on the SAS Packing Lists or any future RAS/SAS Traffic Report Forms, the field conductance values and the instruction "Check for Possible Chloride Interference in COD Test" for any such samples. If this is the case or if there is other definite knowledge of chlorides exceeding 2000 mg/l, the laboratory will determine the chloride content and inhibit chloride interference pursuant to Section 7.1 of EPA Method 410.3.

NOTE: It is expected that few waters from Region V (<3%), will have chloride concentrations in excess of 2000 mg/l, however, it is the responsibility of field personnel to first identify any samples having a chloride interference for COD so that the COD test can be modified to compensate for any chloride interference.

Results are reported as mg/l COD.

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 concentration):

Analyze 44 groundwater samples for chemical oxygen demand (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: Daily by overnight courier.

6. Number of days analysis and data required after laboratory receipt of samples:

_____

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

EPA Method 410.1 (Titrimetric, Mid-level) for COD $\geq$ 50 mg/l.
EPA Method 410.2 (Titrimetric, Low-level) for COD $<$ 50 mg/l.
Use Section 7.1 of Method 410.3 if chloride concentration exceeds 2000 mg/l in a sample. If titration blank is necessary for each different amount of mercuric sulfate used for inhibition of chloride interference, SAS Packing Lists will note the samples requiring assessment of chloride interferences. Measurement of chloride will be done using any method of "Standard Methods",16th ed., or "EPA Methods for Chemical Analysis of Water and Wastes", 1983 ed., whenever possible chloride interference is noted.
Samples will be preserved with 1 ml of H_2SO_4 to pH less than 2 and kept at 4°C until sample analysis and validation of results are completed. Holding time is not to exceed 28 days from date of sample collection.

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

1. Check sample pH (wide range pH paper). If pH>2, contact CPMS, CRL for further instructions.
2. Use a) 50 ml sample aliquots for both methods, b) 0.250 N $K_2Cr_2O_7$ reagent and 0.25 N ferrous ammonium sulfate titrant for Method 410.1, and c) 0.0250 N $K_2Cr_2O_7$ reagent and 0.025 N ferrous ammonium sulfate titrant for Method 410.2.
3. Dilute and reanalyze (by Method 410.1) any samples with COD values > 800 mg/l or titrant volumes < 5.0 ml. Reanalyze samples (by Method 410.1) if initial sample values are > 50 mg/l COD by Method 410.2. Reanalyze samples (by Method 410.2) if initial sample values are < 50 mg/l COD by Method 410.1.
4. Any sample aliquots < 50 mls will be diluted to 50 mls so that the COD reaction mixture will be 50% H_2SO_4/ 50% water by volume.
5. Titration blanks will be determined, at least in duplicate each day of analysis and will not differ more than $\pm$ 0.1 ml titrant for Method 410.1 and $\pm$ 1.0 ml titrant for Method 410.2.
6. Separate sets of QA Audits will be performed for each method, if both methods are used.
7. Use potassium hydrogen phthalate as a matrix spike compound. Use 20 mg/l matrix spike concentration for Method 410.2.
8. Samples will be refluxed for at least 2 hours.
9. Homogenize sample aliquots, as necessary, to obtain sample aliquots of representative suspended solids.
10. Use only the method specified.
11. Analyze MS and MSD as dictated by Section II of the QC Requirements.

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, tabulating titrant standardization, titration volumes for titration or sample blanks (2 or more in number), samples, and QA Audits will be provided for each method used. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results. Records of chloride analysis will be provided for any samples so specified on the RAS/SAS Traffic Report or SAS Packing List. Separate bench records will be provided for any COD determinations of high chloride samples (>2000 mg/l Cl) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type. EPA QC Reference samples, or any other reference samples, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

5/018G-0-6/87 COD (Hi- and Lo-Levels) 6-26-87

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
COD (Method 410.1)	50 mg/l	Method 410.1: Differences in sample duplicates are to be < or = to 0.2 ml titrant or ≤ 8 mg/l for concentrations < 80 mg/l and < 10% for COD concentrations exceeding 80 mg/l.
COD (Method 410.2)	5 mg/l	Method 410.2: Differences in sample duplicate results are to be ≤ 1.0 ml titrant or ≤ 4 mg/l for concentrations less than 40 mg/l and are to be ≤ 5 mg/l for concentrations between 40 50 mg/l.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike (KHP) Method 410.1* Method 410.2(Use 20 mg/l spike)	at least 1 per group of 10 or fewer samples	85 - 115% Recovery (410.1) 75 - 125% Recovery (410.2)
Lab duplicate	" " " "	Diff ≤ (8 mg/l or 10%) (410.1) Diff ≤ (4 mg/l - 5 mg/l) (410.2)
Titration blank (used for calculation of results)	at least 2 per sample set for each method used	Diff in titrant volumes shall not exceed 0.1 ml for 410.1 and 1.0 ml for 410.2
1 set of EPA QC Demand Reference samples - 2 concentration levels	1 per sample set for each method used	90 - 110% Recovery or ≤ 8 mg/l error for 410.1 and ≤ 5 mg/l error for 410.2 in aliquot tested

* - Matrix spike will be greater than 30% of the sample concentration, but spiked sample shall not exceed 800 mg/l for Method 410.1.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

Contact Region V RSCC Dennis Wesolowski (312) 886-1971 concerning questions on chloride interferences and modifications of COD test.

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.

5/014__-0-6/87 Nitrate/nitrite 6/29/87

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

SAS Number

APPROVED FOR SCHEDULING

SPECIAL ANALYTICAL SERVICES
Client Request _____ _____

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

E. Site Name: Onalaska Sanitary Landfill

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 nitrate plus nitrite (as mg/l N) in water (surface water ground water, drinking water, leachates, etc.) Samples will be unfiltered.

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 concentration):

Analyze 44 groundwater samples for nitrate/nitrite.

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: Daily by overnight courier.

6. Number of days analysis and data required after laboratory receipt of samples:

_____

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1) EPA Method 353.1 (colorimetric, automated hydrazine reduction).
2) EPA Method 353.2 (colorimetric, automated cadmium reduction).
3) EPA Method 353.3 (colorimetric, manual cadmium reduction).

For all methods:
Samples will be stored at 4°C until analysis and validation of results. Samples will be preserved in the field with sulfuric acid (1 ml/l) to pH<2. The analytical working range shall not exceed 0.1 to 10.0 mg/l N.

For Methods 353.2 or 353.3: If more than one reduction column is used separate calibrations, QA audits, and records are required for each column. The column used must be identified for each analytical result.

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

Analyze the samples within 28 days after collection. Check the sample pH (wide range pH paper is acceptable). If the pH>2 contact CPMS, CRL for instructions. Use only the methods specified in item 7. Obtain approval of CPMS, CRL before using any other method.
For Methods 353.2 and 353.3: After checking the pH it is recommended that the laboratory check for residual chlorine (or oxidizing reagents) and sulfide using test strips such as starch iodide and lead acetate papers. Contact CPMS, CRL if these interferences are present; however, the laboratory must remove these interferences prior to analysis. The laboratory must also minimize interferences due to metals in order to prolong column life. (See Section 7.1.2 of method 353.3) It is suggested that the laboratory may dilute samples up to ten-fold prior to analysis (Section 7.4 of Method 353.3) provided that the final analytical working range does not exceed 0.1 to 10.0 mg/l N.
For all methods: Neutralize samples to pH 5-9 (or to phenolphthalein color end-point) prior to analysis. Dilute and reanalize the neutralized samples if the concentrations exceed that of the highest standard. Use at least five calibration standards (including a zero standard). Prepare the lab blank using 1 ml of H_2SO_4/l. Neutralize and analyze it like a sample. Analyze MS and MSD as specified by Section II of the QC Requirements.

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 must be clearly identified. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, duplicates, etc., with resulting absorbances or concentration readouts will be provided. Worksheets used to calculate results will be included. Any sample treatment to remove interferences will be documented. The laboratory shall submit photocopies of the instrument readout (strip-charts, printer tapes, etc.) All records of analysis and calculations must be legible and sufficient to recalculate all concentrations. Results are to be reported as mg N/l.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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.)
Nitrate + Nitrite	0.10 mg/l as N	Duplicate results must be within 10% for concentrations >1mg/l or within 0.1 mg/l for concentrations ≤ 1mg/l Results will be reported to the nearest 0.1 mg/l for conc. less than 1.0 mg/l and to 2 significant figures for conc. exceeding 1 mg/l-N.
Note: These are minimum requirements. Report actual detection limits used based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use any designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	1 per group of 10 or fewer samples	±(10% - or 0.1 0 mg/l)
Lab Blank (1ml/l H_2SO_4)	2 per sample set	<0.1 mg/l
Calibration verification standard	1 per group of 10 or fewer samples and at end of run	90% - 110%
Calibration blank	1 per group of 10 samples or less	< 0.1 mg/l
1 set of EPA Nutrient QC reference samples-conc. 1 and 2, or EPA F/NO_3 QC sample, WS series Conc. 1 and 2	1 per sample set	85% - 115%

*Matrix spike concentrations will be 30% or larger, of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact Jay Thakkar (312) 886-1972) or Chuck Elly (312) 353-9087.

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.

5/021__-0-7/87 Oil and Grease (IR)(Water) 7-30-87

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

SAS Number

Scheduled for Approval

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V Onalaska Sanitary Landfill
B. Regional Representative: Jan Pels
C. Telephone Number: ( ) (312) 353-2720
D. Data request:
E. Site Name: Onalaska Sanitary Landfill

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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

pectrophotometric, Infra-Red) in water (surface water, groundwater, leachate, etc.).

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 concentration):

Analyze 44 samples for oil and grease.

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: Daily by overnight courier.

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

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

EPA Method 413.2 (Spectrophotometric, Infra-Red), 1983 ed. Samples will be in 1 qt. or 1 liter glass bottles and preserved with 2 ml/l H_2SO_4 to pH < 2. Sample holding time is 10 days from date of sample collection. Samples will be kept at 4 °C until analysis. Sample volume is best calculated by weighing sample bottle full and empty to the nearest 5 g. Any duplicate samples will be field duplicates.

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

Check sample pH (wide range pH paper). If pH > 2, contact CPMS, CRL for further instructions. A solvent blank is necessary for each solvent lot, and will be free of interferences. Each 30 ml solvent extraction will be added to sample bottle (Section 7.3 and 7.5 of Method 413.2), sample bottle and cap extracted, and solvent added to separatory funnel. Use only the method specified. Calibration standard is prepared only by Section 6.4 of Method 413.2. An instrument cell path of 50 mm or larger is required (See Section 7.7 of Method 413.2). Prepare 5 point calibration curve containing a zero concentration standard for each cell between zero 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 sample absorbance exceeds that of highest standard or exceeds 0.8 absorbance. Analyze MS and MSD as dictated Section II QC Requirements.

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. EPA QC reference samples, or any other reference sample, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Oil and Grease	< 0.4 mg/l	Any designated field duplicate values should not exceed ± 25% or 4 mg/l.
NOTE: This is a minimum requirement. Report actual detection limits used down to 0.1 mg/l (See Sections 1.3 and 7.7 of Method 413.2)		

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Solvent Blank (90 ml of Freon)	once per solvent lot and sample set	< 0.2 mg
Lab Blank (1 liter of tapwater at pH < 2)	at least 1 per group of 10 or fewer samples	-0.4 to +0.4 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
1 set of EPA QC Oil and Grease Reference samples	once per set of samples	within 95% confidence levels provided by EMSL-Cincinnati

III. *Action Required if Limits are Exceeded:

Take corrective action - Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

5/016__-0-6/87 Total Phosphorus in H_2O 6/29/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved For Scheduling
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Onalaska Sanitary Landfill

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 phosphorous in waters (surface waters, ground waters, drinking waters, leachate, etc.). Most samples will be unfiltered although certain aliquots can be filtered and preserved at time of collection. Results will be reported as mg/l P.

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 concentration):

Analyze 44 groundwater samples for total phosphorus.

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: Daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days after receipt of samples.

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

Total Phosphorus EPA Method 365.1 (Automated, Colorimetric, Ascorbic Acid)

Total Phosphorus EPA Method 365.2 (Automated, Colorimetric, Single Reagent)

Total Phosphorus EPA Method 365.4 (Block Digestor)

Samples will be preserved in the field with 1 ml/l H_2SO_4 to pH <2 and stored at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.) Check sample pH using wide-range pH paper. If the pH>2, contact CPMS, CRL for instructions:

Dilute and redigest samples with absorbances or peak heights higher than the highest standard. All standards, blanks, audits, etc. must be digested. The holding time is not to exceed 28 days from sample collection. Use only the method(s) specified above. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

Analyze MS and MSD as specified by Section II of the QC Requirements.

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 and all records of analysis and calculations for samples, blanks, duplicates, spikes and all control checks with peak height or response and concentrations will be provided with copies of worksheets. Results will be reported as mg/l P. Any digestion log will be provided showing sample aliquots and concentrations of all samples tested. Records must be legible and sufficient to recalculate all concentrations. A photocopy of the instrument readout i.e. stripcharts, printer tapes, etc. must be included. EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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.)
Total P	0.05 mg/l	Duplicate results must agree to within 10% for concentrations > 0.5 mg/l or within 0.05 mg/l for concentrations < 0.5 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± (10% or 0.05 mg/l)
Lab Blank (Also serves as a calibration blank).	at least 1 per group of 10 or fewer samples	<0.05 mg/l
Calibration verification standard	1 per group of 10 samples and end of sample set	90% - 110%
1 set of EPA nutrient QC reference samples conc. 3&4	1 per sample set	85% - 115%

*The matrix spike concentrations will be approximately 30% or larger of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

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.

5/011__-0-7/87 Sulfate in Water July 30, 1987

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

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling _____

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

E. Site Name: Onalaska Sanitary Landfill

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 sulfate in water (surface water, groundwater, drinking water, leachate, etc.). Samples will be unfiltered. Results are reported as mg/l SO_4.

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 concentration):

Analyze 44 groundwater samples for sulfate.

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: Daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples:

_____

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

1. EPA Method 375.2 (Colorimetric Methylthmol Blue) - 1983 ed.
- Note: This method requires 0.75 mg/l SO_4 in Dilution Water(See Reagent Section 6.8)

2. Method 426C of Standard Methods, 16th ed. (Turbidimetric)
- Note; this last method provides for measurement of sulfate using 2 standard curves- 1 for sulfate concentrations between 0 and 10mg/l, and 1 between 10 and 40 mg/l sulfate.

Samples will be kept at 4°C until validation of results.

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

Sample holding time is not to exceed 28 days from date of sample collection. Sulfate standards will be prepared daily from stock solution. Samples with absorbances or turbidities greater than that in the highest standard will be diluted and rerun. For Method 426C, 1) the reanalysis solution should contain between 20 and 40 mg/l sulfate, and 2) concentrations must be corrected for background turbidity and color per Section 5d of Method 426C using pH adjusted sample aliquots. Use only the methods specified. Calibration curves must include at least 6 points (including a zero concentration standard) for Method 375.2 and Buffer A of Method 426C. Analyze MS and MSD as dictated by the Section II QC requirements

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 must be clearly identified. Results shall be reported as mg/l SO_4. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, etc., with resulting absorbances or concentration readouts, will be provided along with copies of worksheets used to calculate results. Background absorbances used for turbidity corrections must be tabulated for each sample aliquot tested. A photocopy of the instrument readout (ie. strip charts, printer tapes, etc.) must be included. All records of analysis must be legible and sufficient to calculate all concentrations and results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source,lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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.)
Sulfate	5 mg/l	Method 375.2: Differences in duplicate sample results are to be < 5 mg/l for concentrations < 50 mg/l, and < 10% for concentrations > 50 mg/l.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		Method 426 C: Differences in duplicate sample results are to be ≤ 2 mg/l for concentrations ≤ 20 mg/l and ≤ 10% for concentrations > 20 mg/l in aliquot tested.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85-115%
Lab Duplicate	" "	± (10% or 5 mg/l) for Method 375.2
		± (10% or 2 mg/l) for Method 426C
Lab Blank (0 mg/l SO_4)	" "	< 5 mg/l - Method 375.2 -2 to +2mg/l-Buffer B of Method 426C or
Lab Blank (10 mg/l SO_4)	" "	8 to 10mg/l - Buffer A of Method 426C
Calibration Verification Standard	1 per group of 10 samples and at end of sample set	90 - 110%
1 Set of EPA QC Mineral Reference Samples	once per sample set	85-115% for each concentration.

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

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.

5/012__-0-7/87 S - Titr. 7/30/87

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

SAS Number

Scheduled for Approval

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V Onalaska Sanitary Landfill
B. Regional Representative: Jan Pels
C. Telephone Number: (312) 353-2720
D. Data request:
E. Site Name: Onalaska Sanitary Landfill

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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 sulfide in water (groundwater, wastewater, leachates, etc.) The detection limit using this method is 1 mg/l $S^{=}$. (If a lower detection limit is necessary the Colorimetric method must be used.)

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 concentration):

Analyze 40 groundwater samples for sulfide.

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: Daily by overnight courier.

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

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

"Standard Methods for the Examination of Water and Wastewater", 16th edition: Method #427 B and D with #421 B 2e. for standardization of the iodine solution and #421 B 2f for standardization of the sodium thiosulfate solution. No other methods may be used.

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

Collect all samples in duplicate to allow for any reanalysis and lab duplicate. Avoid any head space. Sample volumes must be 500 ml or larger. Preserve samples by adding 2 N zinc acetate at the rate of 1.5 ml/l plus NaOH to pH between 9 and 11. Samples must be analyzed within 7 days of collection. Because of the preservation procedure, the entire sample must be used for analysis. Also samples must be prepared and analyzed using the filtration technique (Method 427 B, Sec. 3b and 427 D, Sec. 2b) The original volume of the sample must be accurately known and must be included in calculating the final results (427 D, Sec. 3).

Prepare and standardize the following solutions each day:

1) iodine standard, per Method 427, Sec. D, 1a.
2) sodium thiosulfate solution, per Method 421 B, Sec. 2e, and 2f.

Prepare fresh potassium bi-iodate standard each day per Method 421 B, Sec. 2f.

Complete all standardization titrations in duplicate. Duplicate results for solution standardizations must agree to within 0.2 ml. Use the average of the duplicate results 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 tabulating all standardization titrations (in duplicate), sample and QA audit titrations, sample volumes, calculations, etc., will be provided. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA audits.

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

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

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.

5/012G-0-7/87 S - Titr. 7/30/87

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Sulfide	1.0 mg/l	Difference in duplicate results should not exceed ± 10% for concentrations > 3 mg/l or .3 mg/l for concentrations < 3 mg/l. Report results to the nearest 0.1 mg/l (above 1 mg/l) or to 2 significant figures above 10 mg/l.

II. QUALITY CONTROL REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Field duplicates	1 per group of 5 or fewer samples	± (10% or 0.3 mg/l)

III. *Action Required if Limits are Exceeded:

Repeat all associate sample analyses with second sample bottle.

5/024__-0-6/87 Total Dissolved Solids 6/29/87

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

SAS Number

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V Onalaska Sanitary Landfill
B. Regional Representative: Jan Pels
C. Telephone Number: (312) (312) 353-2720
D. Data request:
E. Site Name: Sanitary Landfill

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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 (180°C) in water (surface waters, wastes, groundwaters, drinking water, leachate, etc.) 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 concentration):

Analyze 44 groundwater samples for total dissolved solids ( ).

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: Daily by overnight courier

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

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

1. EPA Method 160.1, 1983 ed., or

2. Method 209B, "Standard Methods", 16th ed. Samples will be kept at 4°C until sample analysis and validation of results. Holding time is 7 days from date of sample collection.

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

1) Use standard aliquots of 100ml; however do not use sample aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller sample aliquot. 2) If the pH value is less than 4.0, raise the pH of the aliquot (using NaOH titrant) to between pH 4 and 8 and subtract the weight of sodium added from the weigth of the residue. 3) Residue will be weighed either to constant weight pursuant to Section 7.6 of Method 60.1 the final weight is to be used for calculations. Constant weight is defined as a) less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller, or b) dried overnight (12 hours drying time) with a single weight 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.

Identify the QC reference sample lot numbers used and their true values with 95% confidence intervals. Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, duplicate samples, and reference samples will be provided with copies of work sheets used to calculate results . Dates and time of 1) determination of tare weights, 2) sample filtration, and 3) determination of residue weights and constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

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

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
TDS	20 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.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. QUALITY CONTROL REQUIREMENTS Do not use any designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1. 1 set of EPA QC Mineral Reference Samples*- 2 concentration levels.	1 per sample set	85-115% Recovery
2. Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg of 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

* Alternate reference samples must be approved by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contant Charles T. Elly (312/353-9087) or Jay Thakkar at (312/886-1972).

5/020__-0-6/87 Total Organic Carbon in Water 6/30/87

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

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request Approved For Scheduling

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V Onalaska Sanitary Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Onalaska Sanitary Landfill

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 in water (surface waters, groundwaters, drinking waters, leachate, etc.). 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 concentration):

   Analyze 44 groundwater samples for total organic carbon.

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: Daily by overnight carrier.

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

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

EPA Method 415.1 (combustion or oxidation).

Samples will be preserved with 1 ml/l H_2SO_4 to pH <2. Samples will be stored at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements) dilute and rerun samples with absorbances higher than the highest standard:

Check sample pH with (wide range pH paper). If pH >2 contact CPMS, CRL for instructions. The holding time is not to exceed 28 days from sample collection. Homogenize samples if necessary. Qualify results where suspended solids content may affect accuracy. Instruments with syringe injection will tilize 2 injections per measurement. If the 2 injections differ by more than 10% or _ mg/l, repeat and report the average of 4 injections. Inorganic carbon will be purged from solution or, if determined separately, subtracted from total carbon values. Obtain approval of CPMS, CRL, prior to use of any other method. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

Analyze MS and MSD as specified by Section II of the QC Requirements.

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 used will be clearly identified. Bench records tabulating order of calibration standards, lab blanks, samples lab control standards, spikes, duplicates etc., with resulting output on concentration readout will be provided along with worksheets used to calculate results. Specify the organic compound used to prepare standards and spikes. A photocopy of the instrument read-out, i.e. stripcharts, printer, tapes, etc. must be included. Results are to be reported in mg/l C. Records of analysis and calculations must be legible and sufficient to re-calculate all concentrations.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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

REPORT BOTH TOTAL CARBON AND TOTAL ORGANIC CARBON VALUES.

11. Name of sampling/shipping contact: Dave Shekoski

Phone: (414) 272-2426

DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
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.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± (10% or 2.0 mg/l)
Lab Blank	at least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Calibration verification standard	1 per group of 10 samples and end of set	90% - 110%
1 set of EPA demand QC reference samples (conc. 1 and 2)	1 per sample set	85% - 115%

*The matrix spike concentrations will be approximately 30% of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

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.

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

SAS Number

Approved for Scheduling

_____

Special Analytical Services
Regional Request

[X]	Regional Transmittal
[ ]	Telephone Request

A. EPA Region and Site Name: Region V /ONALASKA MUNICIPAL LANDFILL
B. Regional Representative: Jan Pels
C. Telephone Number: ( ) (312) 353-2720
D. Data request:
E. Site Name: ONALASKA MUNICIPAL LANDFILL

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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 suspended solids (103-105°C) in water (surface waters, groundwater, drinking water, leachate, etc.) Results are reported as mg/l total suspended 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 concentration):

Analyze 44 groundwater samples for total suspended solids (TSS).

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: Daily by overnight carrier.

6. Approximate number of days results required after lab receipt of samples: 30

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

1. EPA Method 160.2, 1983 ed., (Gravimetric, Dried at 103° - 105° C) using glass fiber filter discs without organic binder such as: Millipore AP-40, Reeve Angel 934-AH, Gelman A/E, or equivalent. Use only membrane filter apparatus with 47 mm diameter glass fiber filter and a coarse (40-60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples will be held at 4°C until sample analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

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

1. Sample aliquot volumes are selected on the basis of the following factors. a) During initial sample filtratrion, filtration rate should not drop rapidly, or require more than 5 minutes of filtratrion time. (Increase the filter area or decrease the sample volume as needed for sample reanalysis), b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200ml in volume, and c) Sample aliquots should not exceed 200ml in volume. 2. Duplicate sample aliquots will be filtered with 2 or more intervening samples. 3. Final residues are to be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 (The final weight is to be used for calculations), or dried overnight (12 hours of drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller. 4. Use only the method specified above in items 7 and 8.

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.

Identify EPA QC reference sample lot numbers used and their true values and 95% confidence intervals. Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples (all in the order filtered) will be provided along with copies of worksheets used to calculate results. Dates and time of a) filtration of initial 100ml volume, b) determination of tare weights, c) sample filtration, and d) determination of constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

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

11. Name of sampling/shipping contact: _____

Phone: _____

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.

DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Suspended Solids	2-3 mg/l for 200 ml	Difference in duplicate results shall not exceed 0.5 mg for duplicate aliquots filtered.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.	sample aliquot	

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1) Lab Duplicates (See item 8.3 on Page 2)	1 per group of 10 or fewer samples	less than 0.5 mg
2) Lab Blanks 200 ml aliquots)	1 per group or 10 or fewer samples	-0.5 to +0.5 mg
3) 1 set of 2 EPA QC Residue Reference Samples-2 concentration levels	1 per sample set	< 5 mg/l error for concentrations < to 50 mg/l or < or = to 10% for nominal concentrations > than 50 mg/l

* Alternate reference samples must be approbed by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Suspended Solids	2-3 mg/l for 200 ml	Difference in duplicate results shall not exceed 0.5 mg for duplicate aliquots filtered.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.	sample aliquot	

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1) Lab Duplicates (See item 8.3 on Page 2)		
2) Lab Blanks (200 ml aliquots)		
3) 1 set of 2 EPA QC Residue Reference Samples-2 concentration levels		

* Alternate reference samples must be approbed by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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 drinking water residential wells for metals at low 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 concentration):

Analyze 18 low level residential well samples for metals.

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: Daily by Overnight Carrier

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

Ten days for analysis; final report and data due within 15 days of receipt of samples.

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

Protocol as per Statement of Work for inorganics, 1987, SOW No. 787

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

Exceptions to inorganic SOW- Attachment III; Required low level detection limits- Attachment II.

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.

All deliverables included in the SOW required including Quarterly Form XI if the lower required detection limits are met. Otherwise, a new detection limit study must be done per SOW to achieve the required detection limits. (See attachment II).

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

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Inorganics	See Attachment II	As in SOW

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Prep. blank	One per 10 samples	<+/-D.L.
Duplicate	One per 10 samples	<10% RPD
Spike Sample	One per 10 samples	85 - 115 %R
Int. Check Std.	Beg. and end of run.	90 - 110 %R
Lab Control Std.	One per 10 samples	90 - 110 %R

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Attachment 2
INSTRUMENT DETECTION LIMIT FOR DRINKING WATER

Compound	Required Detection Limits (ug/l)	Method
Metal:		
1. Aluminum	100	ICP
2. Antimony	2	GFAA
3. Arsenic	2	GFAA
4. Barium	50	ICP
5. Beryllium	5	ICP
6. Cadmium	2	ICP
7. Calcium	1000	ICP
8. Chromium	10	ICP
9. Cobalt	10	ICP
10. Copper	10	ICP
11. Iron	100	ICP
12. Lead	2	GFAA
13. Magnesium	1000	ICP
14. Manganese	10	ICP
15. Mercury	0.2	Cold Vapor
16. Nickel	20	ICP
17. Potassium	2000	ICP
18. Selenium	2	GFAA
19. Silver	5	ICP
20. Sodium	1000	ICP
21. Thallium	2	GFAA
22. Vanadium	10	ICP
23. Zinc	20	ICP
24. Cadmium	0.2	GFAA
25. Tin	40	ICP
26. Cyanide	5.0	Colorimetric

Instrument Detection Limits (IDL) must be met before any samples are run. Laboratory may submit their quarterly form XI with each case if all IDLs meet the detection limits.

GLT824/44

Attachment 3
Table 2
CONCENTRATION LEVELS OF STANDARD SOLUTIONS
OF FURNACE METALS

ANTIMONY	0 ppb	10.0 ppb	20.0 ppb	30.0 ppb
Amt. of Nitric Acid	0.2%			
Inj. Vol.	20 ul			
ARSENIC	0 ppb	10.0 ppb	20.0 ppb	30.0 ppb
Amt. of Nitric Acid	0.5%			
Matrix Modifier	2 ml of 5% Ni, 2 ml of 30% H_2O_2			
Inj. Vol.	20 ul			
CADMIUM	0 ppb	0.5 ppb	1.0 ppb	1.5 ppb
Amt. of Nitric Acid	0.5%			
Inj. Vol.	10 ul			
LEAD	0 ppb	10 ppb	20 ppb	30 ppb
Amt. of Nitric Acid	0.5%			
Inj. Vol.	10 ul			
SELENIUM	0 ppb	10 ppb	20 ppb	30 ppb
Amt. of Nitric Acid	0.5%			
Matrix Modifier	2 ml of 5% Ni, 2 ml of 30% H_2O_2			
Inj. Vol.	20 ul			
THALLIUM	0 ppb	10 ppb	20 ppb	30 ppb
Amt. of Nitric Acid	0.5%			
Inj. Vol.	20 ul			

GLT824/47

Attachment 3

All samples should be preserved in the field as follows: 5 ml of 8N HNO_3 per liter with a final pH <2 for metals, 0.5 percent HNO_3 v/v and 0.05 percent K_2CrO_7 w/v for mercury determination, and 5 ml of 6N NaOH per liter with a final pH >12 for cyanide determination.

Digestion Procedure:

1. All beakers used for digestion should be rinsed with distilled water. Each beaker should be heated on a warm hot plate with 50 ml of 1:1 HCl for 30 minutes and rinsed again with distilled water.

2. Shake sample and transfer 50 ml of thoroughly mixed sample to the beaker.

3. Add 6 ml of 1:1 HNO_3 to the beaker.

4. Cover the beaker with a watch glass and heat on a hot plate at approximately 95°C without boiling until sample is reduced to approximately 10 ml.

5. Remove the beaker from the hot plate and cool the sample.

6. Add 5 ml of 6N HCl to each beaker and heat on a hot plate at approximately 90°C for 10 to 15 minutes. Cool, filter, and adjust the volume to 50 ml with type II (de-ionized metal-free) water in a volumetric flask.

7. A preparation blank, sample duplicate, sample spike, and laboratory control sample shall be digested with the samples for ICP analysis and for furnace AA metals when sample digestion is required.

8. All AA furnace metals can be analyzed without digestion if the samples are clean. If samples show any suspended particles, then samples must be digested using 2 ml of 1:1 HNO_3 and 2 ml of 30 percent H_2O_2. Cover the beakers with water glasses and heat for 2 hours at 95°C or until sample is reduced to approximately 10 ml. Cool, filter, and adjust the volume to 50 ml with distilled water in a volumetric flask. Use ICP digestate for antimony analysis on furnace.

9. Analyze all AA furnace metals by the method of standard additions (MSA) running sample and three spikes consecutively. Report the correlation coefficient, and if it is less than 0.995, re-analyze the sample. If the correlation coefficient is less than 0.995 after the second analysis, dilute the sample and rerun to get a

correlation coefficient from 0.995 or better. Blank, duplicate, and laboratory control sample should also be run by MSA.

10. Preparation standards for metals as per Table 2 for As, Pb, Sb, Se, Tl, and Cd. Cadmium can be analyzed by ICP if instrument detection limit (2 ppb) can be obtained.

11. Use mid-range concentration for EPA QC/LCS.

12. Initial calibration verification can be used as LCS and should be run in the beginning of the analysis, after every 5 samples for furnace metals, and after every 10 samples for ICP.

GLT824/43

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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 drinking water/residential wells for volatiles, semivolatiles and pesticides/PCBs with low 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 concentration):

Analyze 18 low level residential well samples for volatiles, semivolatiles, and pesticides/PCBs.

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: Daily by Overnight Carrier

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

Ten days for analysis; final report and data due within 15 days of sample receipt

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

Protocol as per Statement of Work for Organics, 1987, SOW No. WA-87J001, WA-87J002, WA-87J003.

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

1. Exceptions to Organic SOW - Attachment I
2. Required low level detection limits - Attachment II
3. Requirements for determining sensitivity limits: Easily recognizable spectra for all compounds using 10 ng injection for ABNs and 1.5 ug/l for VOAs.
4. Initial calibrations: %RSD for RFs should be < 40 for each VOA and ABN compound before beginning analyses.
5. Continuing calibration: Run daily calibration standard before running analyses. %D should be <25% for all compounds in both VOAs and ABNs. If any %Ds are greater than 25%, the standard should be reinjected. If still out, rerun 3 point curve.

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.**

All deliverables included in the SOW required including instrument sensitivity determinations.

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

11. **Name of sampling/shipping contact:** Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Organics	See Attachment II	See Attachment I

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Organics - As in SOW	As in SOW	See Attachment I

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

***Action Required if Limits are Exceeded:**

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Attachment 1

VOA

Increase sample volume up to 20 ml to meet sensitivity limits.

Initial Calibration: 5 ug/L, 10 ug/L, 20 ug/L for all compounds except acrolein and acrylonitrile, which should be run at 200 ug/L, 300 ug/L, 500 ug/L.

Continuing Calibration: 10 ug/L except all those compounds that have a detection limit greater than 3.0 ug/L which are to be run at 20 ug/L. Acrolein and acrylonitrile should be run at 300 ug/L.

Surrogates: As in IFB, but at 10 ug/L with percent recovery 80 to 120 percent.

Matrix spike: As in IFB, but at 10 ug/L with percent recovery 80 to 120 percent.

All RFs must be ≥0.05.

ABN

Extract entire bottle, rinse cap and bottle, and add to sample.

Decrease extract volume to help meet sensitivity limits.

Initial Calibration: 20, 50, and 100 total nanograms.

Continuing Calibration: 20 nanograms except for the following: Benzoic acid, 2,4-dinitrophenol, all three nitroaniline isomers, 4-nitrophenol, 2,4,5-trichlorophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol which are to be injected at 50 nanograms.

Surrogates: 20 ppb BN compounds with percent recoveries as listed in IFB. 40 ppb Acid compounds with percent recoveries as listed in IFB.

Matrix spike: 20 ppb BN compounds with percent recoveries as listed in IFB. 40 ppb Acid compounds with percent recoveries as listed in IFB.

All RFs must be ≥0.05.

PESTICIDE/PCB

Extract entire bottle, rinse cap and bottle, and add to sample. Decrease extract volume to help meet sensitivity limits.

Calibration: As in IFB using an attenuation setting capable of achieving the sensitivity limits in Attachment 2. 72 hour run sequence as in IFB.

Surrogates: Use 20 percent of IFB amounts with percent recoveries as listed in IFB.

Matrix Spike: Use 20 percent of the IFB amounts with percent recoveries as listed in IFB.

GLT824/45

Attachment 2 (Page 1 of 3)
ORGANIC ANALYTES AND DETECTION LIMITS

Analyte	CAS #	Method Detection Limit in Reagent Water (ug/l)
Benzene	71-43-2	1.5
Bromodichloromethane	75-27-4	1.5
Bromoform	75-25-2	1.5
Bromomethane	74-83-9	10
Carbon Tetrachloride	56-23-5	1.5
Chlorobenzene	108-90-7	1.5
Chloroethane	75-00-3	1.5
2-Chloroethyl Vinyl Ether	110-76-8	1.5
Chloroform	67-66-3	1.5
Chloromethane	74-87-3	10
Dibromochloromethane	124-48-1	1.5
1,1-Dichloroethane	75-34-3	1.5
1,2-Dichloroethane	107-06-2	1.5
1,1-Dichloroethene	75-35-4	1.5
Total-1,2-Dichloroethene	156-60-5	1.5
1,2-Dichloropropane	78-87-5	1.5
cis-1,3-Dichloropropene	10061-01-5	2
trans-1,3-Dichloropropene	10061-02-6	1
Ethylbenzene	100-41-4	1.5
Methylene Chloride*	75-09-2	1
1,1,2,2-Tetrachloroethane	79-34-5	1.5
Tetrachloroethene	127-18-4	1.5
Toluene*	108-88-3	1.5
1,1,1-Trichloroethane	71-55-6	1.5
1,1,2-Trichloroethane	79-00-5	1.5
Trichloroethene	79-01-6	1.5
Vinyl Chloride	75-01-4	10
Acrolein	107-02-8	100
Acetone*	67-64-1	10
Acrylonitrile	107-13-1	50
Carbon Disulfide	75-15-0	3
2-Butanone	78-93-3	10
Vinyl Acetate	108-05-4	10
4-Methyl-2-Pentanone	108-10-1	(3)
2-Hexanone	519-78-6	10
Styrene	100-42-6	1
m-Xylene	108-38-3	2
o-Xylene**	95-47-6	
p-Xylene**	106-42-3	2.5
Aniline	62-53-3	1.5
Bis(2-Chloroethyl)Ether	111-44-4	1.5
Phenol	108-96-2	2
1-Chlorophenol	95-57-8	2
1,3-Dichlorobenzene	541-73-1	2
1,4-Dichlorobenzene	106-46-7	2

Analyte	CAS #	Method Detection Limit in Reagent Water (ug/l)
1,2-Dichlorobenzene	95-50-1	2.5
Benzyl Alcohol	100-51-6	2
Bis(2-Chloroisopropyl)Ether	39633-32-9	2.5
2-Methylphenol	95-48-7	1
Hexachloroethane	67-72-1	2
N-Nitrosodipropylamine	621-64-7	1.5
Nitrobenzene	98-95-3	2.5
4-Methylphenol	106-44-5	1
Isophorone	78-59-1	2.5
2-Nitrophenol	88-75-5	2
2,4-Dimethylphenol	105-67-9	2
Bis(2-Cholorethoxy)Methane	111-91-1	2.5
2,4-Dichlorophenol	120-83-2	2
1,2,4-Trichlorobenzene	120-82-1	2
Naphthalene	91-20-3	2
4-Chloroaniline	106-47-8	2
Hexachlorobutadiene	87-68-3	2.5
Benzoic Acid	65-85-0	(30)
2-Methylnapthalene	91-57-6	2
4-Chloro-3-Methylphenol	59-50-7	1.5
Hexachlorocyclopentadiene	77-47-4	2
2,4,6-Trichlorophenol	88-06-2	1.5
2,4,5-Trichlorophenol	95-96-4	1.5
2-Chloronapthalene	91-58-7	1.5
Acenapthylene	208-96-8	1.5
Dimethyl Phthalate	131-11-3	1.5
2,5-Dinitrotoluene	606-20-2	1
Acenaphthene	83-32-9	1.5
3-Nitroaniline	99-09-2	2.5
Dibenzofuran	132-64-9	1
2,4-Dinitrophenol	51-28-5	(15)
2,4-Dinitrotoluene	121-14-2	1
Fluorene	86-73-7	1
4-Nitrophenol	100-02-7	1.5
Ethyl Phthalate	84-66-2	1
4,6-Dinitro-2-Methylphenol	534-52-1	(15)
1,2-Diphenylhydrazine	122-66-7	1
N-Nitrosodiphenylamine*	86-30-6	
Phenylamine*	122-39-4	1.5
4-Nitroaniline	100-01-6	3
4-Chlorophenyl Phenyl Ether	7005-72-3	1
4-Bromophenyl Phenyl Ether	101-55-3	1.5
Hexachlorobenzene	118-74-1	1.5
Pentachlorophenol	87-86-5	2
Phenanthrene	85-01-8	1
Anthracene	120-12-7	2.5
Di-n-butyl Phthalate	84-74-2	2

Analyte	CAS #	Method Detection Limit in Reagent Water (ug/l)
Fluoranthene	206-44-0	1.5
Pyrene	129-00-0	1.5
Butyl Benzyl Phthalate	85-68-7	3.5
Chrysene**	218-01-9	
Benzo(a)Anthracene**	56-55-3	1.5
Bis(2-Ethylhexyl)Phthalate	117-81-7	1
Benzo(a)Pyrene	117-84-0	1.5
Benzo(b)Fluoranthene**	205-99-2	
Benzo(k)Fluoranthene**	207-08-9	1.5
Di-n-octyl Phthalate	50-32-8	2
Indeno(1,2,3-cd)Pyrene	193-39-5	3.5
Dibenzo(a,h)Anthracene	53-70-3	2.5
Benzo(g,h,i)Perylene	191-24-2	4
2-Nitroaniline	88-74-4	1
Aldrin	309-00-2	0.005
alpha-BHC	319-84-6	(0.010)
beta-BHC	319-85-7	(0.005)
gamma-BHC (lindane)	58-89-9	0.005
delta-BHC	319-86-8	(0.005)
Chlordane	57-74-9	(0.020)
4,4'-DDD	72-54-8	(0.020)
4,4'-DDE	72-55-9	(0.005)
4-4'-DDT	50-29-3	0.020
Dieldrin	60-57-1	0.010
Endosulfan I	959-98-8	0.010
Endosulfan II	33213-65-9	0.010
Endosulfan Sulfate	1031-07-8	(0.10)
Endrin	72-20-8	0.010
Endrin Aldehyde	7421-93-4	(0.030)
Endrin Ketone	53494-70-5	(0.030)
Heptachlor	76-44-8	0.030
Heptachlor Epoxide	1024-57-3	0.005
4-4'-Methoxychlor	72-43-5	0.020
Toxaphene	8001-35-2	(0.25)
PCB-1242	53469-21-9	(0.10)
PCB-1248	12572-29-6	(0.10)
PCB-1254	11097-69-1	(0.10)
PCB-1260	11096-82-5	(0.10)

Values in parentheses are estimates. Actual values are currently being determined.

* Common laboratory solvent--blank limit is 5 times the method detection limit.

** Reported as a total.

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Atterberg limits analysis will be performed at a physical soil testing laboratory.

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 concentration):

Analyze 35 subsurface soil samples for atterberg limits.

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: Daily by Overnight Carrier

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

Laboratory will report results within 30 days of sample receipt.

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

Liquid Limit Plastic Limit, and Plasticity Index, Plastic Limit, and Natural Water Content of Soils, ASTM Method D4318.

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

Liquid Limit using the Multipoint Method. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology.

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.

Report all raw data. Results reported as percent water content.

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

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Liquid Limit	Not Applicable	
Plastic Limit	Not Applicable	
Natural Water Content		

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	One per 10 for sets >10 Two for sets <10	20%

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Designation: D 4318 – 84

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for LIQUID LIMIT, PLASTIC LIMIT, AND PLASTICITY INDEX OF SOILS[1]

This standard is issued under the fixed designation D 4318; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the liquid limit, plastic limit, and the plasticity index of soils as defined in Section 3.

1.1.1 Two procedures for preparing test specimens and two procedures for performing the liquid limit are provided as follows:

A Multipoint test using a wet preparation procedure, described in Sections 10.1, 11, and 12.

B Multipoint test using a dry preparation procedure, described in Sections 10.2, 11, and 12.

C One-point test using a wet preparation procedure, described in Sections 13, 14, and 15.

D One-point test using a dry preparation procedure, described in Sections 13, 14, and 15.

The procedure to be used shall be specified by the requesting authority. If no procedure is specified, Procedure A shall be used.

NOTE 1—Prior to the adoption of this test method, a curved grooving tool was specified as part of the apparatus for performing the liquid limit test. The curved tool is not considered to be as accurate as the flat tool described in 6.2 since it does not control the depth of the soil in the liquid limit cup. However, there are some data which indicate that typically the liquid limit is slightly increased when the flat tool is used instead of the curved tool.

1.1.2 The plastic limit test procedure is described in Sections 16, 17, and 18. The plastic limit test is performed on material prepared for the liquid limit test. In effect, there are two procedures for preparing test specimens for the plastic limit test.

1.1.3 The procedure for calculating the plasticity index is given in Section 19.

1.2 The liquid limit and plastic limit of soils (along with the shrinkage limit) are often collectively referred to as the Atterberg limits in recognition of their formation by Swedish soil scientist, A. Atterberg. These limits distinguish the boundaries of the several consistency states of plastic soils.

1.3 As used in this test method, soil is any natural aggregation of mineral or organic materials, mixtures of such materials, or artificial mixtures of aggregates and natural mineral and organic particles.

1.4 The multipoint liquid limit procedure is somewhat more time consuming than the one-point procedure when both are performed by experienced operators. However, the one-point procedure requires the operator to judge when the test specimen is approximately at its liquid limit. In cases where this is not done reliably, the multipoint procedure is as fast as the one-point procedure and provides additional precision due to the information obtained from additional trials. It is particularly recommended that the multipoint procedure be used by inexperienced operators.

1.5 The correlations on which the calculations of the one-point procedure are based may not be valid for certain soils, such as organic soils or soils from a marine environment. The liquid

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved Oct. 26, 1984. Published December 1984. Originally published as D 4318 – 83. Last previous edition D 4318 – 83[ε1].

limit of these soils should therefore be determined by the multipoint procedure (Procedure A).

1.6 The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on undried samples. If the liquid and plastic limits of soils are used to correlate or estimate the engineering behavior of soils in their natural moist state, samples should not be permitted to dry before testing unless data on dried samples are specifically desired.

1.7 The composition and concentration of soluble salts in a soil affect the values of the liquid and plastic limits as well as the water content values of soils (see Method D 2216). Special consideration should therefore be given to soils from a marine environment or other sources where high soluble salt concentrations may be present. The degree to which the salts present in these soils are diluted or concentrated must be given consideration if meaningful results are to be obtained.

1.8 Since the tests described herein are performed only on that portion of a soil which passes the 425-μm (No. 40) sieve, the relative contribution of this portion of the soil to the properties of the sample as a whole must be considered when using these tests to evaluate the properties of a soil.

1.9 The values stated in acceptable metric units are to be regarded as the standard. The values given in parentheses are for information only.

1.10 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

C 702 Methods for Reducing Field Samples of Aggregate to Testing Size[2]

D 75 Practice for Sampling Aggregates[3]

D 420 Recommended Practice for Investigating and Sampling Soil and Rock for Engineering Purposes[4]

D 653 Terms and Symbols Relating to Soil and Rock[4]

D 1241 Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses[4]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[4]

D 2240 Test Method for Rubber Property—Durometer Hardness[5]

D 2487 Test Method for Classification of Soils for Engineering Purposes[4]

D 2488 Recommended Practice for Description of Soils (Visual-Manual Procedure)[4]

D 3282 Recommended Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[6]

E 319 Methods of Testing Single-Arm Balances[6]

E 898 Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances[6]

3. Definitions

3.1 *Atterberg limits*—originally, seven "limits of consistency" of fine-grained soils were defined by Albert Atterberg. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limit.

3.2 *consistency*—the relative ease with which a soil can be deformed.

3.3 *liquid limit (LL)*—the water content, in percent, of a soil at the arbitrarily defined boundary between the liquid and plastic states. This water content is defined as the water content at which a pat of soil placed in a standard cup and cut by a groove of standard dimensions will flow together at the base of the groove for a distance of 13 mm (½ in.) when subjected to 25 shocks from the cup being dropped 10 mm in a standard liquid limit apparatus operated at a rate of 2 shocks per second.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vols 04.02, 04.03, and 04.08.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 09.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

NOTE 2—The undrained shear strength of soil at the liquid limit is considered to be 2 ±0.2 kPa (0.28 psi).

3.4 *plastic limit (PL)*—the water content, in percent, of a soil at the boundary between the plastic and brittle states. The water content at this boundary is the water content at which a soil can no longer be deformed by rolling into 3.2 mm (⅛ in.) in diameter threads without crumbling.

3.5 *plastic soil*—a soil which has a range of water content over which it exhibits plasticity and which will retain its shape on drying.

3.6 *plasticity index (PI)*—the range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

3.7 *liquidity index*—the ratio, expressed as a percentage, of (*1*) the natural water content of a soil minus its plastic limit, to (*2*) its plasticity index.

3.8 *activity number (A)*—the ratio of (*1*) the plasticity index of a soil to (*2*) the percent by weight of particles having an equivalent diameter smaller than 0.002 mm.

4. Summary of Method

4.1 The sample is processed to remove any material retained on a 425-μm (No. 40) sieve. The liquid limit is determined by performing trials in which a portion of the sample is spread in a brass cup, divided in two by a grooving tool, and then allowed to flow together from the shocks caused by repeatedly dropping the cup in a standard mechanical device. The multipoint liquid limit, Procedures A and B, requires three or more trials over a range of water contents to be performed and the data from the trials plotted or calculated to make a relationship from which the liquid limit is determined. The one-point liquid limit, Procedures C and D, uses the data from two trials at one water content multiplied by a correction factor to determine the liquid limit.

4.2 The plastic limit is determined by alternately pressing together and rolling into a 3.2 mm (⅛ in.) diameter thread a small portion of plastic soil until its water content is reduced to a point at which the thread crumbles and is no longer able to be pressed together and rerolled. The water content of the soil at this stage is reported as the plastic limit.

4.3 The plasticity index is calculated as the difference between the liquid limit and the plastic limit.

5. Significance and Use

5.1 This test method is used as an integral part of several engineering classification systems to characterize the fine-grained fractions of soils (see Test Method D 2487 and Recommended Practice D 3282) and to specify the fine-grained fraction of construction materials (see Specification D 1241). The liquid limit, plastic limit, and plasticity index of soils are also used extensively, either individually or together with other soil properties to correlate with engineering behavior such as compressibility, permeability, compactibility, shrink-swell, and shear strength.

5.2 The liquid and plastic limits of a soil can be used with the natural water content of the soil to express its relative consistency or liquidity index and can be used with the percentage finer than 2-μm size to determine its activity number.

5.3 The one-point liquid limit procedure is frequently used for routine classification purposes. When greater precision is required, as when used for the acceptance of a material or for correlation with other test data, the multipoint procedure should be used.

5.4 These methods are sometimes used to evaluate the weathering characteristics of clay-shale materials. When subjected to repeated wetting and drying cycles, the liquid limits of these materials tend to increase. The amount of increase is considered to be a measure of a shale's susceptibility to weathering.

5.5 The liquid limit of a soil containing substantial amounts of organic matter decreases dramatically when the soil is oven-dried before testing. Comparison of the liquid limit of a sample before and after oven-drying can therefore be used as a qualitative measure of organic matter content of a soil.

6. Apparatus

6.1 *Liquid Limit Device*—A mechanical device consisting of a brass cup suspended from a carriage designed to control its drop onto a hard rubber base. A drawing showing the essential features of the device and the critical dimensions is given in Fig. 1. The design of the device may vary provided that the essential functions are

preserved. The device may be operated either by a hand crank or by an electric motor.

6.1.1 *Base*—The base shall be hard rubber having a D Durometer hardness of 80 to 90, and a resilience such that an 8-mm (5/16-in.) diameter polished steel ball, when dropped from a height of 25 cm (9.84 in.) will have an average rebound of at least 80 % but no more than 90 %. The tests shall be conducted on the finished base with feet attached.

6.1.2 *Feet*—The base shall be supported by rubber feet designed to provide isolation of the base from the work surface and having an A Durometer hardness no greater than 60 as measured on the finished feet attached to the base.

6.1.3 *Cup*—The cup shall be brass and have a weight, including cup hanger, of 185 to 215 g.

6.1.4 *Cam*—The cam shall raise the cup smoothly and continuously to its maximum height, over a distance of at least 180° of cam rotation. The preferred cam motion is a uniformly accelerated lift curve. The design of the cam and follower combination shall be such that there is no upward or downward velocity of the cup when the cam follower leaves the cam.

NOTE 3—The cam and follower design in Fig. 1 is for uniformly accelerated (parabolic) motion after contact and assures that the cup has no velocity at drop off. Other cam designs also provide this feature and may be used. However, if the cam-follower lift pattern is not known, zero velocity at drop off can be assured by carefully filing or machining the cam and follower so that the cup height remains constant over the last 20 to 45° of cam rotation.

6.1.5 *Carriage*—The cup carriage shall be constructed in a way that allows convenient but secure adjustment of the height of drop of the cup to 10 mm (0.394 in.). The cup hanger shall be attached to the carriage by means of a pin which allows removal of the cup and cup hanger for cleaning and inspection.

6.1.6 *Optional Motor Drive*—As an alternative to the hand crank shown in Fig. 1, the device may be equipped with a motor to turn the cam. Such a motor must turn the cam at 2 ±0.1 revolutions per second, and must be isolated from the rest of the device by rubber mounts or in some other way that prevents vibration from the motor being transmitted to the rest of the apparatus. It must be equipped with an ON-OFF switch and a means of conveniently positioning the cam for height of drop adjustments. The results obtained using a motor-driven device must not differ from those obtained using a manually operated device.

6.2 *Flat Grooving Tool*—A grooving tool having dimensions shown in Fig. 2. The tool shall be made of plastic or noncorroding metal. The design of the tool may vary as long as the essential dimensions are maintained. The tool may, but need not, incorporate the gage for adjusting the height of drop of the liquid limit device.

6.3 *Gage*—A metal gage block for adjusting the height of drop of the cup, having the dimensions shown in Fig. 3. The design of the tool may vary provided the gage will rest securely on the base without being susceptible to rocking, and the edge which contacts the cup during adjustment is straight, at least 10 mm (3/8 in.) wide, and without bevel or radius.

6.4 *Containers*—Small corrosion-resistant containers with snug-fitting lids for water content specimens. Aluminum or stainless steel cans 2.5 cm (1 in.) high by 5 cm (2 in.) in diameter are appropriate.

6.5 *Balance*—A balance readable to at least 0.01 g and having an accuracy of 0.03 g within three standard deviations within the range of use. Within any 15-g range, a difference between readings shall be accurate within 0.01 g (Notes 4 and 5).

NOTE 4—See Methods E 898 and E 319 for an explanation of terms relating to balance performance.

NOTE 5—For frequent use, a top-loading type balance with automatic load indication, readable to 0.01 g, and having an index of precision (standard deviation) of 0.003 or better is most suitable for this method. However, nonautomatic indicating equal-arm analytical balances and some small equal arm top pan balances having readabilities and sensitivities of 0.002 g or better provide the required accuracy when used with a weight set of ASTM Class 4 (National Bureau of Standards Class P) or better. Ordinary commercial and classroom type balances such as beam balances are not suitable for this method.

6.6 *Storage Container*—A container in which to store the prepared soil specimen that will not contaminate the specimen in any way, and which prevents moisture loss. A porcelain, glass, or plastic dish about 11.4 cm (4½ in.) in diameter and a plastic bag large enough to enclose the dish and be folded over is adequate.

6.7 *Ground Glass Plate*—A ground glass plate at least 30 cm (12 in.) square by 1 cm (3/8 in.) thick for mixing soil and rolling plastic limit threads.

6.8 *Spatula*—A spatula or pill knife having a

blade about 2 cm (¾ in.) wide by about 10 cm (4 in.) long. In addition, a spatula having a blade about 2.5 cm (1 in.) wide and 15 cm (6 in.) long has been found useful for initial mixing of samples.

6.9 *Sieve*—A 20.3 cm (8 in.) diameter, 425-μm (No. 40) sieve conforming to the requirements of Specification E 11 and having a rim at least 5 cm (2 in.) above the mesh. A 2-mm (No. 10) sieve meeting the same requirements may also be needed.

6.10 *Wash Bottle*, or similar container for adding controlled amounts of water to soil and washing fines from coarse particles.

6.11 *Drying Oven*—A thermostatically controlled oven, preferably of the forced-draft type, capable of continuously maintaining a temperature of 110 ±5°C throughout the drying chamber. The oven shall be equipped with a thermometer of suitable range and accuracy for monitoring oven temperature.

6.12 *Washing Pan*—A round, flat-bottomed pan at least 7.6 cm (3 in.) deep, slightly larger at the bottom than a 20.3-cm (8-in.) diameter sieve.

6.13 *Rod* (optional)—A metal or plastic rod or tube 3.2 mm (⅛ in.) in diameter and about 10 cm (4 in.) long for judging the size of plastic limit threads.

7. Materials

7.1 A supply of distilled or demineralized water.

8. Sampling

8.1 Samples may be taken from any location that satisfies testing needs. However, Methods C 702, and Practice D 75, and Recommended Practice D 420 should be used as guides for selecting and preserving samples from various types of sampling operations. Samples which will be prepared using the wet preparation procedure, 10.1, must be kept at their natural water content prior to preparation.

8.2 Where sampling operations have preserved the natural stratification of a sample, the various strata must be kept separated and tests performed on the particular stratum of interest with as little contamination as possible from other strata. Where a mixture of materials will be used in construction, combine the various components in such proportions that the resultant sample represents the actual construction case.

8.3 Where data from this test method are to be used for correlation with other laboratory or field test data, use the same material as used for these tests where possible.

8.4 Obtain a representative portion from the total sample sufficient to provide 150 to 200 g of material passing the 425-μm (No. 40) sieve. Free flowing samples may be reduced by the methods of quartering or splitting. Cohesive samples shall be mixed thoroughly in a pan with a spatula, or scoop and a representative portion scooped from the total mass by making one or more sweeps with a scoop through the mixed mass.

9. Calibration of Apparatus

9.1 *Inspection of Wear:*

9.1.1 *Liquid Limit Device*—Determine that the liquid limit device is clean and in good working order. The following specific points should be checked:

9.1.1.1 *Wear of Base*—The spot on the base where the cup makes contact should be worn no greater than 10 mm (⅜ in.) in diameter. If the wear spot is greater than this, the base can be machined to remove the worn spot provided the resurfacing does not make the base thinner than specified in 6.1 and the other dimensional relationships are maintained.

9.1.1.2 *Wear of Cup*—The cup must be replaced when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep or when the edge of the cup has been reduced to half its original thickness. Verify that the cup is firmly attached to the cup hanger.

9.1.1.3 *Wear of Cup Hanger*—Verify that the cup hanger pivot does not bind and is not worn to an extent that allows more than 3-mm (⅛-in.) side-to-side movement of the lowest point on the rim.

9.1.1.4 *Wear of Cam*—The cam shall not be worn to an extent that the cup drops before the cup hanger (cam follower) loses contact with the cam.

9.1.2 *Grooving Tools*—Inspect grooving tools for wear on a frequent and regular basis. The rapidity of wear depends on the material from which the tool is made and the types of soils being tested. Sandy soils cause rapid wear of grooving tools; therefore, when testing these materials, tools should be inspected more frequently than for other soils. Any tool with a tip width greater than 2.1 mm must not be used. The depth

of the tip of the grooving tool must be 7.9 to 8.1 mm.

NOTE 6—The width of the tip of grooving tools is conveniently checked using a pocket-sized measuring magnifier equipped with a millimetre scale. Magnifiers of this type are available from most laboratory supply companies. The depth of the tip of grooving tools can be checked using the depth measuring feature of vernier calipers.

9.2 *Adjustment of Height of Drop*—Adjust the height of drop of the cup so that the point on the cup that comes in contact with the base rises to a height of 10 ±0.2 mm. See Fig. 4 for proper location of the gage relative to the cup during adjustment.

NOTE 7—A convenient procedure for adjusting the height of drop is as follows: place a piece of masking tape across the outside bottom of the cup parallel with the axis of the cup hanger pivot. The edge of the tape away from the cup hanger should bisect the spot on the cup that contacts the base. For new cups, placing a piece of carbon paper on the base and allowing the cup to drop several times will mark the contact spot. Attach the cup to the device and turn the crank until the cup is raised to its maximum height. Slide the height gage under the cup from the front, and observe whether the gage contacts the cup or the tape. See Fig. 4. If the tape and cup are both contacted, the height of drop is approximately correct. If not, adjust the cup until simultaneous contact is made. Check adjustment by turning the crank at 2 revolutions per second while holding the gage in position against the tape and cup. If a ringing or clicking sound is heard without the cup rising from the gage, the adjustment is correct. If no ringing is heard or if the cup rises from the gage, readjust the height of drop. If the cup rocks on the gage during this checking operation, the cam follower pivot is excessively worn and the worn parts should be replaced. Always remove tape after completion of adjustment operation.

MULTIPOINT LIQUID LIMIT—PROCEDURES A AND B

10. Preparation of Test Specimens

10.1 *Wet Preparation*—Except where the dry method of specimen preparation is specified (10.2), prepare specimens for test as described in the following sections.

10.1.1 *Samples Passing the 425-μm (No. 40) Sieve*—When by visual and manual procedures it is determined that the sample has little or no material retained on a 425-μm (No. 40) sieve, prepare a specimen of 150 to 200 g by mixing thoroughly with distilled or demineralized water on the glass plate using the spatula. If desired, soak soil in a storage dish with small amount of water to soften the soil before the start of mixing. Adjust the water content of the soil to bring it to a consistency that would require 25 to 35 blows of the liquid limit device to close the groove (Note 8). If, during mixing, a small percentage of material is encountered that would be retained on a 425-μm (No. 40) sieve, remove these particles by hand, if possible. If it is impractical to remove the coarser material by hand, remove small percentages (less than about 15 %) of coarser material by working the specimen through a 425-μm (No. 40) sieve using a piece of rubber sheeting, rubber stopper, or other convenient device provided the operation does not distort the sieve or degrade material that would be retained if the washing method described in 10.1.2 were used. If larger percentages of coarse material are encountered during mixing, or it is considered impractical to remove the coarser material by the methods just described, wash the sample as described in 10.1.2. When the coarse particles found during mixing are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-μm (No. 40) sieve, but remove by hand or by washing. Place the mixed soil in the storage dish, cover to prevent loss of moisture, and allow to stand for at least 16 h (overnight). After the standing period and immediately before starting the test, thoroughly remix the soil.

NOTE 8—The time taken to adequately mix a soil will vary greatly, depending on the plasticity and initial water content. Initial mixing times of more than 30 — in may be needed for stiff, fat clays.

10.1.2 *Samples Containing Material Retained on a 425-μm (No. 40) Sieve:*

10.1.2.1 Select a sufficient quantity of soil at natural water content to provide 150 to 200 g of material passing the 425-μm (No. 40) sieve. Place in a pan or dish and add sufficient water to cover the soil. Allow to soak until all lumps have softened and the fines no longer adhere to the surfaces of the corase particles (Note 9).

NOTE 9—In some cases, the cations of salts present in tap water will exchange with the natural cations in the soil and significantly alter the test results should tap water be used in the soaking and washing operations. Unless it is known that such cations are not present in the tap water, distilled or demineralized water should be used. As a general rule, water containing more than 100 mg/L of dissolved solids should not be used for washing operations.

10.1.2.2 When the sample contains a large percentage of material retained on the 425-μm

(No. 40) sieve, perform the following washing operation in increments, washing no more than 0.5 kg (1 lb) of material at one time. Place the 425-μm (No. 40) sieve in the bottom of the clean pan. Pour the soil water mixture onto the sieve. If gravel or coarse sand particles are present, rinse as many of these as possible with small quantities of water from a wash bottle, and discard. Alternatively, pour the soil water mixture over a 2-mm (No. 10) sieve nested atop the 425-μm (No. 40) sieve, rinse the fine material through and remove the 2-mm (No. 10) sieve. After washing and removing as much of the coarser material as possible, add sufficient water to the pan to bring the level to about 13 mm (½ in.) above the surface of the 425-μm (No. 40) sieve. Agitate the slurry by stirring with the fingers while raising and lowering the sieve in the pan and swirling the suspension so that fine material is washed from the coarser particles. Disaggregate fine soil lumps that have not slaked by gently rubbing them over the sieve with the fingertips. Complete the washing operation by raising the sieve above the water surface and rinsing the material retained with a small amount of clean water. Discard material retained on the 425-μm (No. 40) sieve.

10.1.2.3 Reduce the water content of the material passing the 425-μm (No. 40) sieve until it approaches the liquid limit. Reduction of water content may be accomplished by one or a combination of the following methods: (*a*) exposing the air currents at ordinary room temperature, (*b*) exposing to warm air currents from a source such as an electric hair dryer, (*c*) filtering in a Buckner funnel or using filter candles, (*d*) decanting clear water from surface of suspension, or (*e*) draining in a colander or plaster of paris dish lined with high retentivity, high wet-strength filter paper.[7] If a plaster of paris dish is used, take care that the dish never becomes sufficiently saturated that it fails to actively absorb water into its surface. Thoroughly dry dishes between uses. During evaporation and cooling, stir the sample often enough to prevent overdrying of the fringes and soil pinnacles on the surface of the mixture. For soil samples containing soluble salts, use a method of water reduction such as *a* or *b* that will not eliminate the soluble salts from the test specimen.

10.1.2.4 Thoroughly mix the material passing the 425-μm (No. 40) sieve on the glass plate using the spatula. Adjust the water content of the mixture, if necessary, by adding small increments of distilled or demineralized water or by allowing the mixture to dry at room temperature while mixing on the glass plate. The soil should be at a water content that will result in closure of the groove in 25 to 35 blows. Return the mixed soil to the mixing dish, cover to prevent loss of moisture, and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, remix the soil thoroughly.

10.2 *Dry Preparation:*

10.2.1 Select sufficient soil to provide 150 to 200 g of material passing the 425-μm (No. 40) sieve after processing. Dry the sample at room temperature or in an oven at a temperature not exceeding 60°C until the soil clods will pulverize readily. Disaggregation is expedited if the sample is not allowed to completely dry. However, the soil should have a dry appearance when pulverized. Pulverize the sample in a mortar with a rubber tipped pestal or in some other way that does not cause breakdown of individual grains. When the coarse particles found during pulverization are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-μm (No. 40) sieve, but remove by hand or other suitable means, such as washing.

10.2.2 Separate the sample on a 425-μm (No. 40) sieve, shaking the sieve by hand to assure thorough separation of the finer fraction. Return the material retained on the 425-μm (No. 40) sieve to the pulverizing apparatus and repeat the pulverizing and sieving operations as many times as necessary to assure that all finer material has been disaggregated and material retained on the 425-μm (No. 40) sieve consists only of individual sand or gravel grains.

10.2.3 Place material remaining on the 425-μm (No. 40) sieve after the final pulverizing operations in a dish and soak in a small amount of water. Stir the soil water mixture and pour over the 425-μm (No. 40) sieve, catching the water and any suspended fines in the washing pan. Pour this suspension into a dish containing the dry soil previously sieved through the 425-μm (No. 40) sieve. Discard material retained on the 425-μm (No. 40) sieve.

10.2.4 Adjust the water content as necessary by drying as described in 10.1.2.3 or by mixing on the glass plate, using the spatula while adding increments of distilled or demineralized water,

[7] S and S 595 filter paper, available in 32-cm circles, has proven satisfactory.

until the soil is at a water content that will result in closure of the groove in 25 to 35 blows.

10.2.5 Put soil in the storage dish, cover to prevent loss of moisture and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, thoroughly remix the soil (Note 8).

11. Procedure

11.1 Place a portion of the prepared soil in the cup of the liquid limit device at the point where the cup rests on the base, squeeze it down, and spread it into the cup to a depth of about 10 mm at its deepest point, tapering to form an approximately horizontal surface. Take care to eliminate air bubbles from the soil pat but form the pat with as few strokes as possible. Heap the unused soil on the glass plate and cover with the inverted storage dish or a wet towel.

11.2 Form a groove in the soil pat by drawing the tool, beveled edge forward, through the soil on a line joining the highest point to the lowest point on the rim of the cup. When cutting the groove, hold the grooving tool against the surface of the cup and draw in an arc, maintaining the tool perpendicular to the surface of the cup throughout its movement. See Fig. 5. In soils where a groove cannot be made in one stroke without tearing the soil, cut the groove with several strokes of the grooving tool. Alternatively, cut the groove to slightly less than required dimensions with a spatula and use the grooving tool to bring the groove to final dimensions. Exercise extreme care to prevent sliding the soil pat relative to the surface of the cup.

11.3 Verify that no crumbs of soil are present on the base or the underside of the cup. Lift and drop the cup by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 13 mm (½ in.). See Fig. 6.

NOTE 10—Use the end of the grooving tool, Fig. 2, or a scale to verify that the groove has closed 13 mm (½ in.).

11.4 Verify that an air bubble has not caused premature closing of the groove by observing that both sides of the groove have flowed together with approximately the same shape. If a bubble has caused premature closing of the groove, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving operation and repeat 11.1 to 11.3. If the soil slides on the surface of the cup, repeat 11.1 through 11.3 at a higher water content. If, after several trials at successively higher water contents, the soil pat continues to slide in the cup or if the number of blows required to close the groove is always less than 25, record that the liquid limit could not be determined, and report the soil as nonplastic without performing the plastic limit test.

11.5 Record the number of drops, N, required to close the groove. Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at right angles to the groove and including that portion of the groove in which the soil flowed together, place in a weighed container, and cover.

11.6 Return the soil remaining in the cup to the glass plate. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.

11.7 Remix the entire soil specimen on the glass plate adding distilled water to increase the water content of the soil and decrease the number of blows required to close the groove. Repeat 11.1 through 11.6 for at least two additional trials producing successively lower numbers of blows to close the groove. One of the trials shall be for a closure requiring 25 to 35 blows, one for closure between 20 and 30 blows, and one trial for a closure requiring 15 to 25 blows.

11.8 Determine the water content, W_N, of the soil specimen from each trial in accordance with Method D 2216. Make all weighings on the same balance. Initial weighings should be performed immediately after completion of the test. If the test is to be interrupted for more than about 15 min, the specimens already obtained should be weighed at the time of the interruption.

12. Calculations

12.1 Plot the relationship between the water content, W_N, and the corresponding number of drops, N, of the cup on a semilogarithmic graph with the water content as ordinates on the arithmetical scale, and the number of drops as abscissas on the logarithmic scale. Draw the best straight line through the three or more plotted points.

12.2 Take the water content corresponding to the intersection of the line with the 25-drop abscissa as the liquid limit of the soil. Computa-

tional methods may be substituted for the graphical method for fitting a straight line to the data and determining the liquid limit.

ONE-POINT LIQUID LIMIT—PROCEDURES C AND D

13. Preparation of Test Specimens

13.1 Prepare the specimen in the same manner as described in Section 10, except that at mixing, adjust the water content to a consistency requiring 20 to 30 drops of the liquid limit cup to close the groove.

14. Procedure

14.1 Proceed as described in 11.1 through 11.5 except that the number of blows required to close the groove shall be 20 to 30. If less than 20 or more than 30 blows are required, adjust the water content of the soil and repeat the procedure.

14.2 Immediately after removing a water content specimen as described in 11.5, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving and water content sampling operations. Repeat 11.2 through 11.5, and, if the second closing of the groove requires the same number of drops or no more than two drops difference, secure another water content specimen. Otherwise, remix the entire specimen and repeat.

NOTE 11—Excessive drying or inadequate mixing will cause the number of blows to vary.

14.3 Determine water contents of specimens as described in 11.8.

15. Calculations

15.1 Determine the liquid limit for each water content specimen using one of the following equations:

$$LL = W_N\left(\frac{N}{25}\right)^{0.121} \quad \text{or}$$

$$LL = K(W_N)$$

where:

N = the number of blows causing closure of the groove at water content,

W_N = water content, and

K = a factor given in Table 1.

The liquid limit is the average of the two trial liquid limit values.

15.2 If the difference between the two trial liquid limit values is greater than one percentage point, repeat the test.

PLASTIC LIMIT

16. Preparation of Test Specimen

16.1 Select a 20-g portion of soil from the material prepared for the liquid limit test, either after the second mixing before the test, or from the soil remaining after completion of the test. Reduce the water content of the soil to a consistency at which it can be rolled without sticking to the hands by spreading and mixing continuously on the glass plate. The drying process may be accelerated by exposing the soil to the air current from an electric fan, or by blotting with paper that does not add any fiber to the soil, such as hard surface paper toweling or high wet strength filter paper.

17. Procedure

17.1 From the 20-g mass, select a portion of 1.5 to 2.0 g. Form the test specimen into an ellipsoidal mass. Roll this mass between the palm or fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length (Note 12). The thread shall be further deformed on each stroke so that its diameter is continuously reduced and its length extended until the diameter reaches 3.2 ±0.5 mm (0.125 ±.020 in.), taking no more than 2 min (Note 13). The amount of hand or finger pressure required will vary greatly, according to the soil. Fragile soils of low plasticity are best rolled under the outer edge of the palm or at the base of the thumb.

NOTE 12—A normal rate of rolling for most soils should be 80 to 90 strokes per minute, counting a stroke as one complete motion of the hand forward and back to the starting position. This rate of rolling may have to be decreased for very fragile soils.

NOTE 13—A 3.2-mm (⅛-in.) diameter rod or tube is useful for frequent comparison with the soil thread to ascertain when the thread has reached the proper diameter, especially for inexperienced operators.

17.1.1 When the diameter of the thread becomes 3.2 mm, break the thread into several pieces. Squeeze the pieces together, knead between the thumb and first finger of each hand, reform into an ellipsoidal mass, and reroll. Continue this alternate rolling to a thread 3.2 mm in diameter, gathering together, kneading and rerolling, until the thread crumbles under the pres-

sure required for rolling and the soil can no longer be rolled into a 3.2-mm diameter thread (See Fig. 7). It has no significance if the thread breaks into threads of shorter length. Roll each of these shorter threads to 3.2 mm in diameter. The only requirement for continuing the test is that they are able to be reformed into an ellipsoidal mass and rolled out again. The operator shall at no time attempt to produce failure at exactly 3.2 mm diameter by allowing the thread to reach 3.2 mm, then reducing the rate of rolling or the hand pressure, or both, while continuing the rolling without further deformation until the thread falls apart. It is permissible, however, to reduce the total amount of deformation for feebly plastic soils by making the initial diameter of the ellipsoidal mass nearer to the required 3.2-mm final diameter. If crumbling occurs when the thread has a diameter greater than 3.2 mm, this shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread 3.2 mm in diameter. Crumbling of the thread will manifest itself differently with the various types of soil. Some soils fall apart in numerous small aggregations of particles, others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally, the thread falls apart in many small platy particles. Fat clay soils require much pressure to deform the thread, particularly as they approach the plastic limit. With these soils, the thread breaks into a series of barrel-shaped segments about 3.2 to 9.5 mm (⅛ to ⅜ in.) in length.

17.2 Gather the portions of the crumbled thread together and place in a weighed container. Immediately cover the container.

17.3 Select another 1.5 to 2.0 g portion of soil from the original 20-g specimen and repeat the operations described in 17.1 and 17.2 until the container has at least 6 g of soil.

17.4 Repeat 17.1 through 17.3 to make another container holding at least 6 g of soil. Determine the water content, in percent, of the soil contained in the containers in accordance with Method D 2216. Make all weighings on the same balance.

NOTE 14—The intent of performing two plastic limit trials is to verify the consistency of the test results. It is acceptable practice to perform only one plastic limit trial when the consistency in the test results can be confirmed by other means.

18. Calculations

18.1 Compute the average of the two water contents. If the difference between the two water contents is greater than two percentage points, repeat the test. The plastic limit is the average of the two water contents.

PLASTICITY INDEX

19. Calculations

19.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:

LL = the liquid limit,

PL = the plastic limit.

Both LL and PL are whole numbers. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

20. Report

20.1 Report the following information:

20.1.1 Sample identifying information,

20.1.2 Any special specimen selection process used, such as removal of sand lenses from undisturbed sample,

20.1.3 Report sample as airdried if the sample was airdried before or during preparation,

20.1.4 Liquid limit, plastic limit, and plasticity index to the nearest whole number and omitting the percent designation. If the liquid limit or plastic limit tests could not be performed, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP,

20.1.5 An estimate of the percentage of sample retained on the 425-μm (No. 40) sieve, and

20.1.6 Procedure by which liquid limit was performed, if it differs from the multipoint method.

21. Precision and Bias

21.1 No interlaboratory testing program has as yet been conducted using this test method to determine multilaboratory precision.

21.2 The within laboratory precision of the results of tests performed by different operators at one laboratory on two soils using Procedure A for the liquid limit is shown in Table 2.

TABLE 1 Factors for Obtaining Liquid Limit from Water Content and Number of Drops Causing Closure of Groove

N (Number of Drops)	K (Factor for Liquid Limit)
20	0.974
21	0.979
22	0.985
23	0.990
24	0.995
25	1.000
26	1.005
27	1.009
28	1.014
29	1.018
30	1.022

TABLE 2 Within Laboratory Precision for Liquid Limit

	Average Value, $\bar{x}$	Standard Deviation, *s*
Soil A:		
PL	21.9	1.07
LL	27.9	1.07
Soil B:		
PL	20.1	1.21
LL	32.6	0.98

DIMENSIONS

LETTER	A △	B △	C △	E △	F	G	H	J △	K △	L △	M △
MM	54 ± 0.5	2 ± 0.1	27 ± 0.5	56 ± 2.0	32	10	16	60 ± 1.0	50 ± 2.0	150 ± 2.0	125 ± 2.0

LETTER	N	P	R	T	U △	V	W	Z
MM	24	28	24	45	47 ± 1.0	3.8	13	6.5

△ ESSENTIAL DIMENSIONS

CAM ANGLE DEGREES	CAM RADIUS
0	0.742 R
30	0.753 R
60	0.764 R
90	0.773 R
120	0.784 R
150	0.796 R
180	0.818 R
210	0.854 R
240	0.901 R
270	0.945R
300	0.974R
330	0.995R
360	1.000R

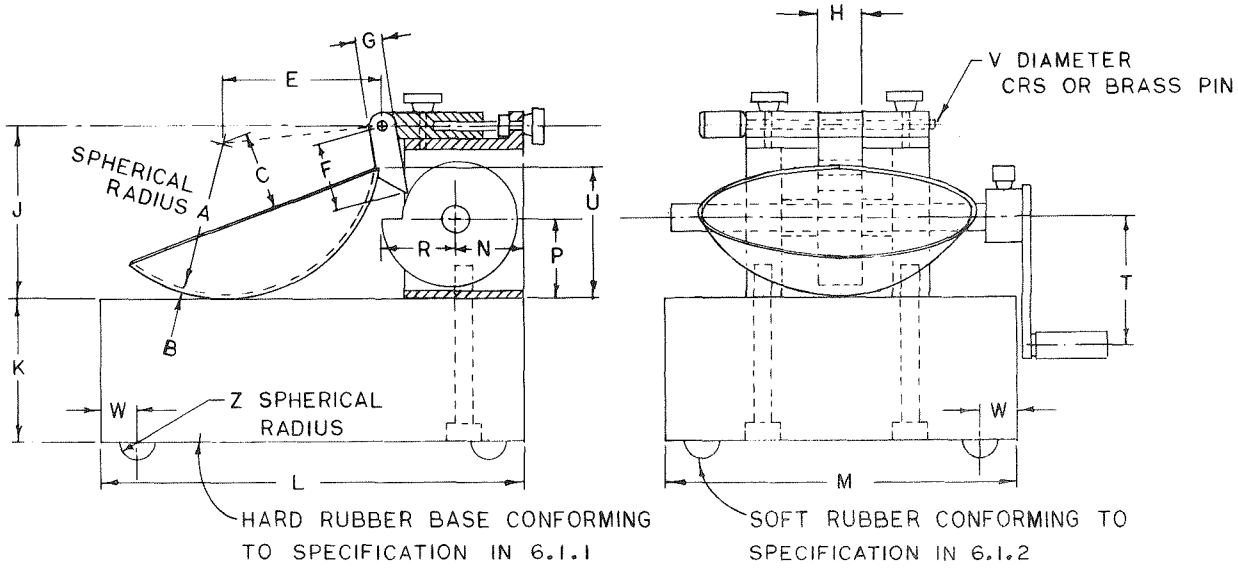


FIG. 1 Hand-Operated Liquid Limit Device

DIMENSIONS

LETTER	A △	B △	C △	D △	E △	F △
MM	2 ± 0.1	11 ± 0.2	40 ± 0.5	8 ± 0.1	50 ± 0.5	2 ± 0.1
LETTER	G	H	J	K △	L △	N
MM	10 MINIMUM	13	60	10 ±0.05	60 DEG ± 1 DEG	20

△ *ESSENTIAL DIMENSIONS*

□ *BACK AT LEAST 15 MM FROM TIP*

NOTE : DIMENSION A SHOULD BE 1.9–2.0 AND DIMENSION D SHOULD BE 8.0–8.1 WHEN NEW TO ALLOW FOR ADEQUATE SERVICE LIFE

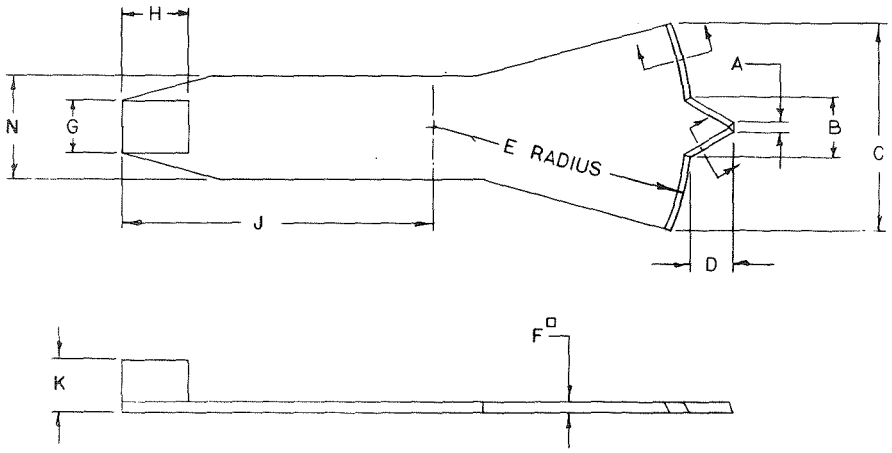
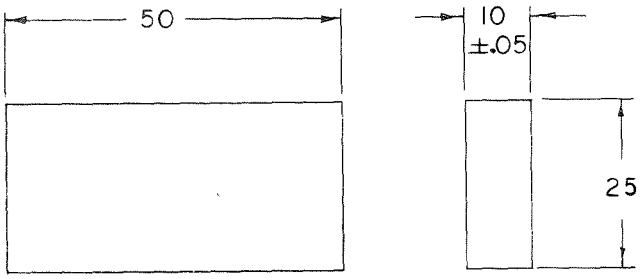


FIG. 2 Grooving Tool (Optional Height-of-Drop Gage Attached)



DIMENSIONS IN MILLIMETRES

FIG. 3 Height of Drop Gage

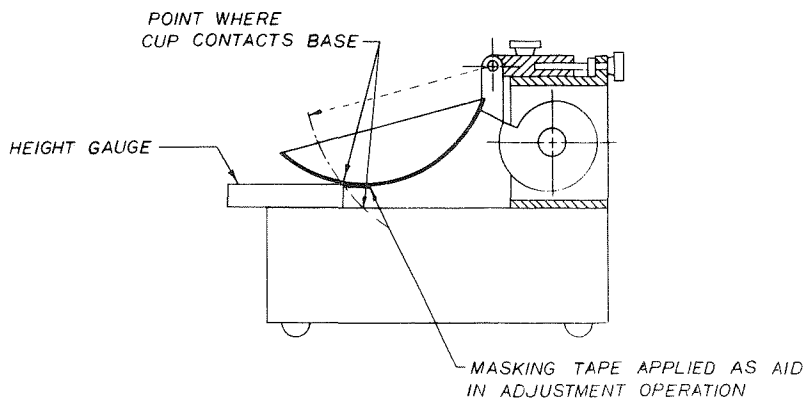


FIG. 4 Calibration for Height of Drop

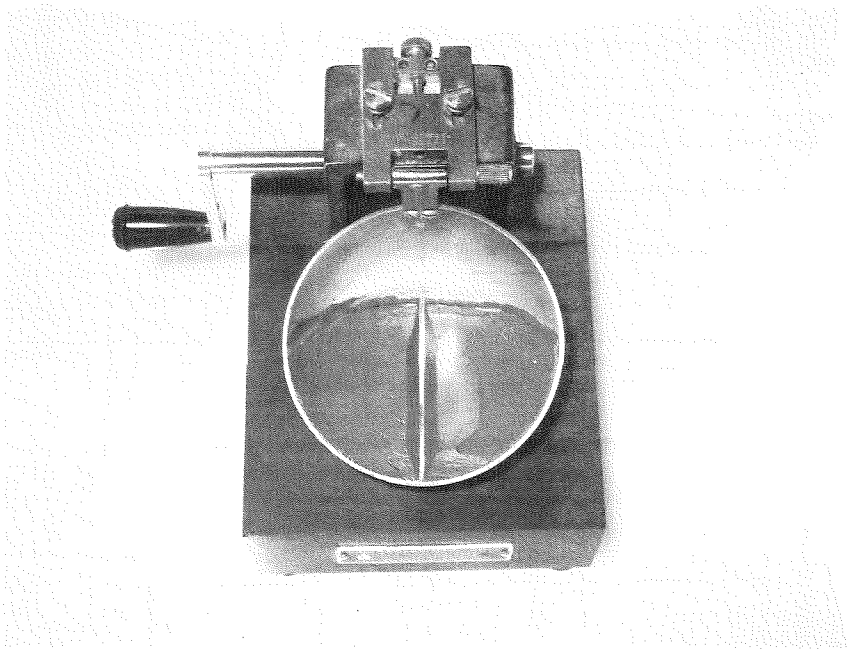


FIG. 5 Grooved Soil Pat in Liquid Limit Device

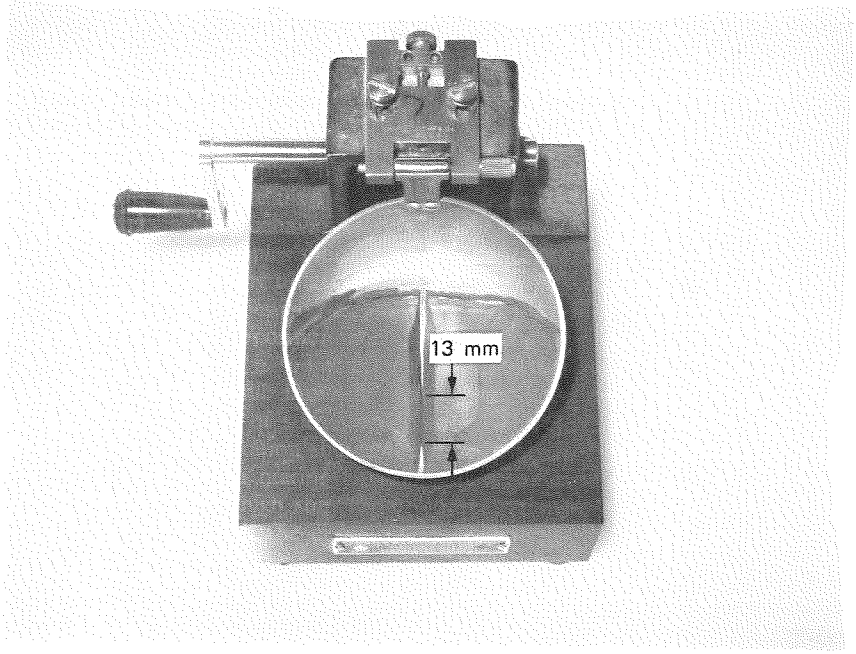


FIG. 6 Soil Pat After Groove Has Closed

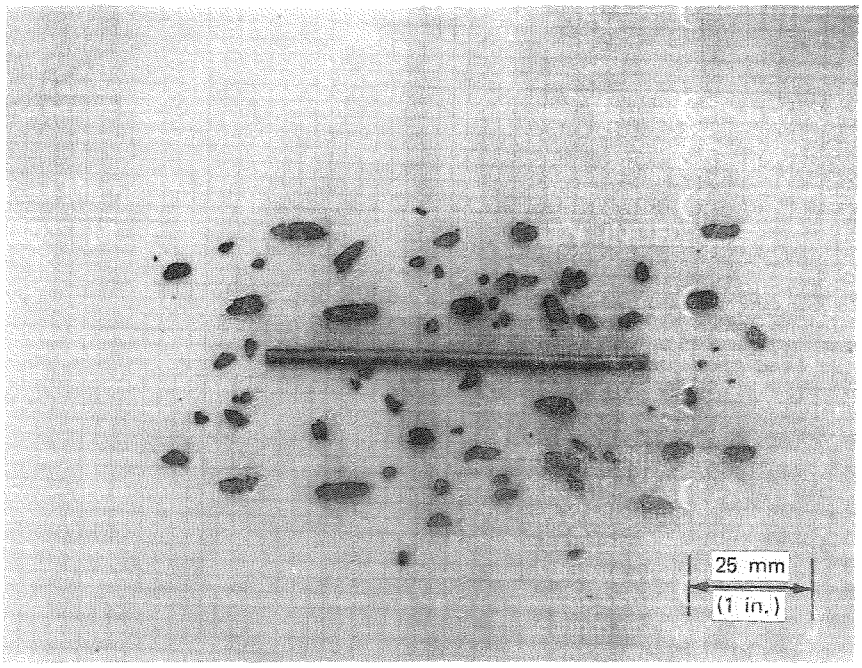


FIG. 7 Lean Clay Soil at the Plastic Limit

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Analyze soil samples for total chlorine (total halogen).

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 concentration):

Analyze 11 soil samples for total chlorine.

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: Daily by Overnight Carrier

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

Laboratory will report results within 40 days of sample receipt.

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

Total Chlorine ASTM D808

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

Prepare a 1:1 w/w slurry using white oil and soil for analysis. White oil standard will be procured from EPA. Prepare the spike sample so that it contains 0.5% chlorine. Analyze matrix spikes and matrix spike duplicates as specified by Section II of the QC Requirements.

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.

Report all QA/QC data, all instrument calibrations, printouts and other information associated with the analysis. Results will be reported on a dry weight basis.

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

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Total Chlorine	0.1%	+/- 25%

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Blank	1 per this project	detection limits
Duplicate	1 per this project	50%
Spike Sample	1 per this project	50 - 150%

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 886-1971)

CHEMICAL STANDARD USED FOR ASTM D808

STANDARD: White Oil of Camphor (White Oil)

CONSTITUENTS: Safrol, acetaldehyde, camphor, terpineol, eugenol, cineol, d-pinene, phellandrene, dipentene, cadinene.

PHYSICAL DATA: Colorless or yellowish liquid
Density @ 20C versus Water @ 20C = 0.875-0.900
Insoluble in Water
Soluble in Chloroform, Ether, and Oils

USE: As solvent in paint and lacquer industry; in perfuming of soaps and detergents; in technical odor masking.

Designation: D 808 – 87

An American National Standard

Standard Test Method for Chlorine in New and Used Petroleum Products (Bomb Method)[1]

This standard is issued under the fixed designation D 808; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been adopted for use by government agencies to replace Method 5651 of Federal Test Method Standard No. 791b.

Attention is called to Note 1 on Safety and to the specific precautionary directions incorporated in the test method.

1. Scope

1.1 This test method covers the determination of chlorine in lubricating oils and greases, including new and used lubricating oils and greases containing additives, and in additive concentrates. Its range of applicability is 0.1 to 50 % chlorine. The procedure assumes that compounds containing halogens other than chlorine will not be present.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The preferred units are mass percent.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1317 Test Method for Chlorine in New and Used Lubricants (Sodium Alcoholate Method)[3]

D 4057 API MPMS 8.1 Practice for Manual Sampling of Petroleum and Petroleum Products[4]

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure (**Caution**—See Note 1). The chlorine compounds thus liberated are absorbed in a sodium carbonate solution and the amount of chlorine present is determined gravimetrically by precipitation as silver chloride.

NOTE 1: **(Caution)—Safety**—Strict adherence to all of the provisions prescribed hereinafter insures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least ½ in. (12.7 mm) thick, or equivalent protection be provided against unforseeable contingencies.

4. Significance and Use

4.1 This test method may be used to measure the level of chlorine-containing compounds in petroleum products. This knowledge can be used to predict performance or handling characteristics of the product in question.

5. Apparatus

5.1 *Bomb*, having a capacity of not less than 300 mL, so constructed that it will not leak during the test, and that quantitative recovery of the liquids from the bomb may be readily achieved. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the chlorine content of the liquid in the bomb.

5.2 *Sample Cup*, platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 to 11 g.

5.3 *Firing Wire*, platinum, approximately No. 26 B & S gage.

5.4 *Ignition Circuit* (Note 2), capable of supplying sufficient current to ignite the nylon thread or cotton wicking without melting the wire.

NOTE 2—**Caution**—The switch in the ignition circuit shall be of a type that remains open, except when held in closed position by the operator.

5.5 *Nylon Sewing Thread, or Cotton Wicking*, white.

5.6 *Filter Crucible*, fritted-glass, 30-mL capacity, medium porosity.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on the Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D 02.03 on Elemental Analysis.

Current edition approved by Oct. 30, 1987. Published December 1987. Originally published as D 808 – 44. Last previous edition D 808 – 81.

In 1963, this test method is adopted as standard without revision.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

TABLE 1 Quantities of Sample and White Oil

Chlorine Content, %	Weight of Sample, g	Weight of White Oil, g
2 and under	0.8	0.0
Above 2 to 5, incl	0.4	0.4
Above 5 to 10, incl	0.2	0.6
Above 10 to 20, incl	0.1	0.7
Above 20 to 50, incl	0.05	0.7

reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or III of Specification D 1193.

6.3 *Nitric Acid* (1+1)—Mix equal volumes of concentrated nitric acid (HNO_3, sp gr 1.42) and water.

6.4 *Oxygen*, free of combustible material and halogen compounds, available at a pressure of 40 atmos. (**Warning**—See Note 3).

NOTE 3—**Warning**—Oxygen vigorously accelerates combustion.

6.5 *Silver Nitrate Solution* (50 g $AgNO_3$/L)—Dissolve 50 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

6.6 *Sodium Carbonate Solution* (50 g Na_2CO_3/L)—Dissolve 50 g of anhydrous Na_2CO_3, 58.5 g of $Na_2CO_3 \cdot H_2O$, or 135 g of $Na_2CO_3 \cdot 10\ H_2O$ in water and dilute to 1 L.

6.7 *White Oil*, refined.

7. Sampling

7.1 Take samples in accordance with the instructions in Practice D 4057.

7.2 Take care that the sample is thoroughly representative of the material to be tested and that the portion of the sample used for the test is thoroughly representative of the whole sample.

8. Procedure

8.1 *Preparation of Bomb and Sample*—Cut a piece of firing wire approximately 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert into the coil a nylon thread, or wisp of cotton, of such length that one end will extend into the sample cup. Place about 5 mL of Na_2CO_3 solution in the bomb (Note 4) and by means of a rubber policeman, wet the interior surface of the bomb, including the head, as thoroughly as possible. Introduce into the sample cup the quantities of sample and white oil (Notes 5 and 6) specified in Table 1 (**Caution**—Note 7), weighing the sample to the nearest 0.2 mg. (When white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion.)

NOTE 4—After repeated use of the bomb for chlorine determination, a film may be noticed on the inner surface. This dullness should be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with Grit No. 2/00 or equivalent paper[6] coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide[6] and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before using the bomb it should be washed with soap and water to remove oil or paste left from the polishing operation. Bombs with porous or pitted surfaces should never be used because of the tendency to retain chlorine from sample to sample.

NOTE 5—When the sample is not readily miscible with white oil, some other nonvolatile, chlorine-free combustible diluent may be employed in place of white oil. However, the combined weight of sample and nonvolatile diluent shall not exceed 1 g. Some solid additives are relatively insoluble, but may be satisfactorily burned when covered with a layer of white oil.

NOTE 6—The practice of running alternately samples high and low in chlorine content should be avoided whenever possible. It is difficult to rinse the last traces of chlorine from the walls of the bomb and the tendency for residual chlorine to carry over from sample to sample has been observed in a number of laboratories. When a sample high in chlorine has preceded one low in chlorine content, the test on the low-chlorine sample should be repeated and one or both of the low values thus obtained should be considered suspect if they do not agree within the limits of repeatability of this method.

NOTE 7—**Caution**—Do not use more than 1 g total of sample and white oil or other chlorine-free combustible material.

8.2 *Addition of Oxygen*—Place the sample cup in position and arrange the nylon thread, or wisp of cotton, so that the end dips into the sample. Assemble the bomb and tighten the cover securely. Admit oxygen (**Caution**—See Note 8) slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in Table 2.

NOTE 8—**Caution**—Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.

8.3 *Combustion*—Immerse the bomb in a cold water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination, and thoroughly clean the bomb before again putting it in use (Note 4).

8.4 *Collection of Chlorine Solution*—Rinse the interior of the bomb, the sample cup, and the inner surface of the bomb cover with a fine jet of water, and collect the washings in a 600-mL beaker. Scrub the interior of the bomb and the inner surface of the bomb cover with a rubber policeman. Wash the base of the terminals until the washings are neutral to the indicator methyl red. (The volume of the washings is normally in excess of 300 mL.) Take special care not to lose any wash water.

8.5 *Determination of Chlorine*—Acidify the solution by adding HNO_3 (1+1) drop by drop until acid to methyl red. Add an excess of 2 mL of the HNO_3 solution. Filter through a qualitative paper (if the solution is cloudy, the presence of

TABLE 2 Gage Pressures

Capacity of Bomb, mL	Minimum Gage Pressure,[A] atm	Maximum Gage Pressure,[A] atm
300 to 350	38	40
350 to 400	35	37
400 to 450	30	32
450 to 500	27	29

[A] The minimum pressures are specified to provide sufficient oxygen for complete combustion, and the *maximum pressures represent a safety requirement.*

[6] Emery Polishing Paper Grit No. 2/00 may be purchased from the Behr-Manning Co., Troy, NY. Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, NJ.

lead chloride ($PbCl_2$) is indicated and the solution should be brought to a boil before filtering) and collect in a second 600-mL beaker. Heat the solution to about 140°F (60°C) and, while protecting the solution from strong light, add gradually, while stirring, 5 mL of $AgNO_2$ solution. Heat to incipient boiling and retain at this temperature until the supernatant liquid becomes clear. Test to ensure complete precipitation by adding a few drops of the $AgNO_3$ solution. If more precipitation takes place, repeat the above steps which have involved heating, stirring, and addition of $AgNO_3$, as often as necessary, until the additional drops of $AgNO_3$ produce no turbidity in the clear, supernatant liquid. Allow the beaker and contents to stand in a dark place for at least an hour. Filter the precipitate by suction on a weighed fritted-glass filter crucible. Wash the precipitate with water containing 2 mL of HNO_3 (1+1)/L. Dry the crucible and precipitate at 110°C for 1 h. Cool in a desiccator, and weigh.

8.6 *Blank*—Make a blank determination with 0.7 to 0.8 g of white oil by following the normal procedure but omitting the sample (Notes 6 and 9). Repeat this blank whenever new batches of reagents or white oil are used. The blank must not exceed 0.03 % chlorine based upon the weight of the white oil.

NOTE 9—This procedure measures chlorine in the white oil and in the reagents used, as well as that introduced from contamination.

9. Calculation

9.1 Calculate the chlorine content of the sample as follows:

$$\text{Chlorine, weight \%} = [(P - B) \times 24.74]/W$$

where:

P = grams of AgCl obtained from the sample,
B = grams of AgCl obtained from the blank, and
W = grams of sample used.

10. Precision and Bias

10.1 The precision of this test method is not known to have been obtained in accordance with currently accepted guidelines (for example., in Committee D-2 Research Report RR:D2-1007, Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants).

10.2 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

10.2.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty:

Chlorine, %	Repeatability
0.1 to 1.9	0.07
2.0 to 5.0	0.15
Above 5.0	3 % of amount present

10.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty:

Chlorine, %	Reproducibility
0.1 to 1.9	0.10
2.0 to 5.0	0.30
Above 5.0	5 % of the amount present

10.3 Cooperative data indicate that deviations of test results from the true chlorine content are of the same order of magnitude as the reproducibility.

10.3.1 *Bias*—The bias of this test method cannot be determined since an appropriate standard reference material containing a known level of chlorine in liquid petroleum hydrocarbon is not available.

.S. Environmental Protection Agency
LP Sample Management Office
. O. Box 818, Alexandria, Virginia 22313
HONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

1. EPA Region/Client: Region V/ Onalaska Municipal Landfill
2. RSCC Representative: Jan Pels
3. Telephone Number: (312) 353-2720
4. Date of Request:
5. Site Name:

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 yo equest, please address the following considerations, if applicable. Incomplete or er eous information may result in delay in the processing of your request. Please continue re: se on additional sheets, or attach supplementary information as needed.

1. eral description of analytical service requested: Extraction Procedure (EP) Toxicity on soil samples

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 concentration):

Analyze 11 soil samples for Extraction Procedure Toxicity.

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

Superfund -

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30-45 days of receipt of samples.

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

Methods from SW-846, Second Edition Manual entitled " Test Methods for Evluating solid waste Physical/Chemical Methods." Extraction method 1310, digestion method(s) 3010,3020 or 3040 (as appropriate). Analysis methods as follows: Arsenic - 7060 or 7061;Barium-7080,7081 or 6010; Lead-7420,7421 or 6010;Mercury-7470;Selenium - 7740 or 7741; Silver-7760,7761 or 6010; Cadmium-7130,7131 or 6010;Chromium-7190,7191 or 6010

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

1). After extraction, the filtrates must be preserved with HNO_3 until analysis. 2). The method of standard additions must be used for all atomic absorption analyses(See Attachment I). 3). Minimum of 100 g of sample should be used for extraction. 4). During pH adjustment procedure in the extraction, add acid in small increments with constant stirring. Be sure that the pH never falls below 4.8 . The mixing time intervals specified in the method must be strictly adhered to .

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 II.

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

11. Name of sampling/shipping contact: _____

Phone: _____

Continued next page

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired[1] (± % or Conc.)
As	< 0.5 mg/l	± 10%
Se	< 0.1 mg/l	± 10%
Hg	< 0.02 mg/l	± 10%
Pb	< 0.5 mg/l	± 5%
Ba	< 10 mg/l	± 5%
Cd	< 0.1 mg/l	± 5%

1) Precision limits apply to control standards and calibration standards

II. QC REQUIREMENTS - As specified in the method plus the following:

Audits Required	Frequency of Audits	Limits* (% or Conc.)
extraction blank	once/batch	± detection limit
extraction duplicate	as requested	no limit
2) control standard	beginning and end of run plus once/10 samples	as listed in "precision"
2) method blank	once/10 samples	± detection limit
2) duplicates	once/10 samples	< RPD 10%
2) spikes	once/10 samples	100 ± 15% recovery
ICP interference check	beginning and end of run plus once/10 samples	as specified in method 6010 (8.5.3)

2) These are methods audits. They are to be carried through the digestion and the analysis but not the extraction.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

1. For the extraction blank, control standard, and method blank - discard all all associated data and reanalyze.

2. For duplicates and spikes - flag all associated data.

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
Cr	< 0.5 mg/l	± 5%
Ag	< 0.5 mg/l	± 5%

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Attachment I

Method of Standard Additions - Specifications

A four-point standard additions calibration is required. Acceptance criteria are described below.

A. Audits as well as samples must be analyzed using the method of standard additions. (Audits include blanks, duplicates, and control standards.)

B. Data from the standard additions analysis must be within the linear range of the test.

C. Recommended addition concentrations are five times the detection limit, 10 times the detection limit, and 15 times the detection limit.

D. The highest sample-plus-addition should not be more than 20% higher than the highest control standard-plus-addition. If the sample-plus-addition exceeds this value, the sample should be diluted and reanalyzed.

E. The correlation coefficient (r) for each standard addition analysis should be $\geq$ 0.995. The slope of the standard addition lines must be $\geq$ 1/3 of the slope of the blank-plus-addition line. If either of these criteria are not met, the sample should be reanalyzed (possibly at a lower dilution) until the criteria are met.

Data Reporting

All results are reported as mg/l.

C. Sample Data Package: A separate package is required for each dataset. The data package includes all analyses and associated QC. Only complete packages may be submitted. Each package must contain the following elements.

1. Tabulated results (concentrations) for the analyses as specified in this SAS.

2. Tabulated results for all QC audits as specified in this SAS.

3. The original or a legible photocopy of all raw data (sequential measurement readout record) clearly labeled with sufficient information to unequivocally identify:

   a. calibration standards
   b. method blanks (with acceptance limits)
   c. independent control standards (with acceptance limits)
   d. ~~Samples~~ all weights, dilutions, volumes, and calculations used to obtain the reported results
   e. duplicates (with control limits)
   f. spikes (including all calculations, theoretical results, percent recoveries, and control limits)
   g. for standard additions analyses, all information identified in attachment 1
   h. any instrument adjustment, data corrections, or other apparent anomalies on the measurement record
   i. date of analysis and original signature of analyst on all pages
   j. extraction log including observations, details, and comments on the preparation & analysis.

4. Report results for each sample as follows: Sample weight, initial pH of solution, amount of acetic acid (0.5N) added after 15 minutes, 30 minutes, 60 minutes, 2 hours, 4 hours, 6 hours, and 24 hours to maintain pH 5.0 $\pm$ 0.2; total amount of 0.5N acetic acid added; final pH; final volume of solution; and date and time extraction started and ended.

   NOTE: All "void" data or data not used to obtain reported values are identified on the reports and the raw data.

5. Indications of the method numbers used for digestion and analysis.

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Grain size analysis will be performed at a physical soil testing laboratory.

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 concentration):

Analyze 35 subsurface soil samples for grain size.

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: Daily by Overnight Carrier

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

Laboratory will report results within 30 days of sample receipt.

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

Particle Size Analysis of Soils, ASTM Method D422

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

Use only the method specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology.

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.

Submit all raw data including container tare weights. Report results as percent finer than the specified particle diameter and present data in the form of a grain size distribution curve on a semilogarithmic chart with percent finer by weight plotted on the arithmetic scale and grain size plotted on the logarithmic scale.

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

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Percentage Finer Than	Not Applicable	

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	One per 10 for sets >10 Two for sets <10	10% passing

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Designation: D 422 – 63 (Reapproved 1972)ε¹

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103

Standard Method for
PARTICLE-SIZE ANALYSIS OF SOILS[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε¹ NOTE—Section 2 was added editorially and subsequent sections renumbered in July 1984.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-μm), or No. 200 (75-μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm.

2. Applicable Documents

2.1 *ASTM Standards:*

D 421 Method for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.01.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.

results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1½-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
¾-in. (19.0-mm)	No. 140 (106-μm)
⅜-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-μm)
¾-in. (19.0-mm)	No. 50 (300-μm)
⅜-in. (9.5-mm)	No. 100 (150-μm)
No. 4 (4.75-mm)	No. 200 (75-μm)
No. 8 (2.36-mm)	

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Method D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Method D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Method D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cyclinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hy-

drometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/l). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continued the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. Accord-

ing to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,
n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)),
T = interval of time from beginning of sedimentation to the taking of the reading, min,
G = specific gravity of soil particles, and
G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(*1*) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(*2*) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
  (*a*) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
  (*b*) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
  (*c*) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(*3*) Silt size, 0.074 to 0.005 mm ..... %
(*4*) Clay size, smaller than 0.005 mm ..... %
  Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-μm)	.....
No. 200 (75-μm)	.....

HYDROMETER ANALYSIS

0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

TABLE 1 Values of Correction Factor, α, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}\,[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,
L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm^3, and
A = cross-sectional area of sedimentation cylinder, cm^2

Values used in calculating the values in Table 2 are as follows:

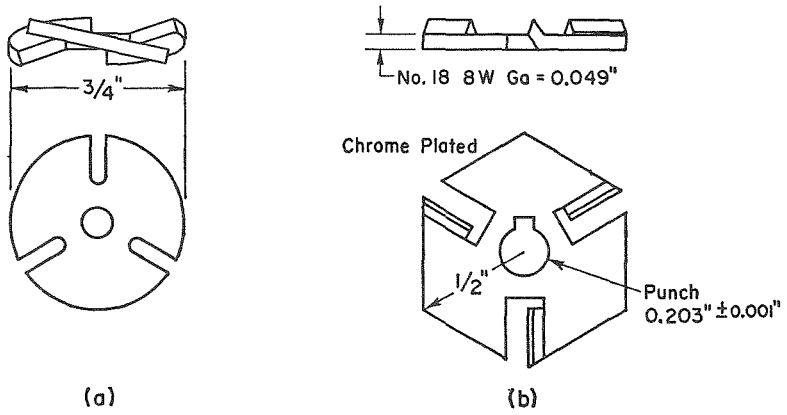
For both hydrometers, 151H and 152H:
L_2 = 14.0 cm
V_B = 67.0 cm^3
A = 27.8 cm^2

For hydrometer 151H:
L_1 = 10.5 cm for a reading of 1.000
= 2.3 cm for a reading of 1.031

For hydrometer 152H:
L_1 = 10.5 cm for a reading of 0 g/litre
= 2.3 cm for a reading of 50 g/litre

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

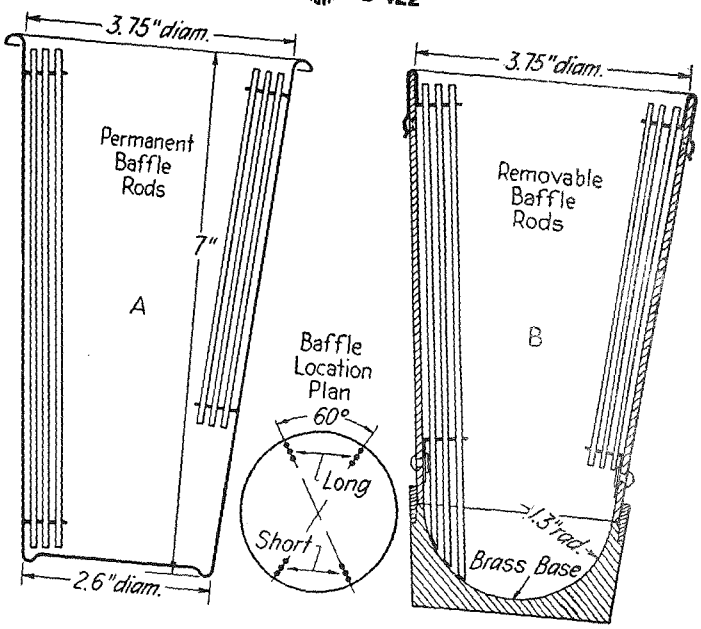
Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149



Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

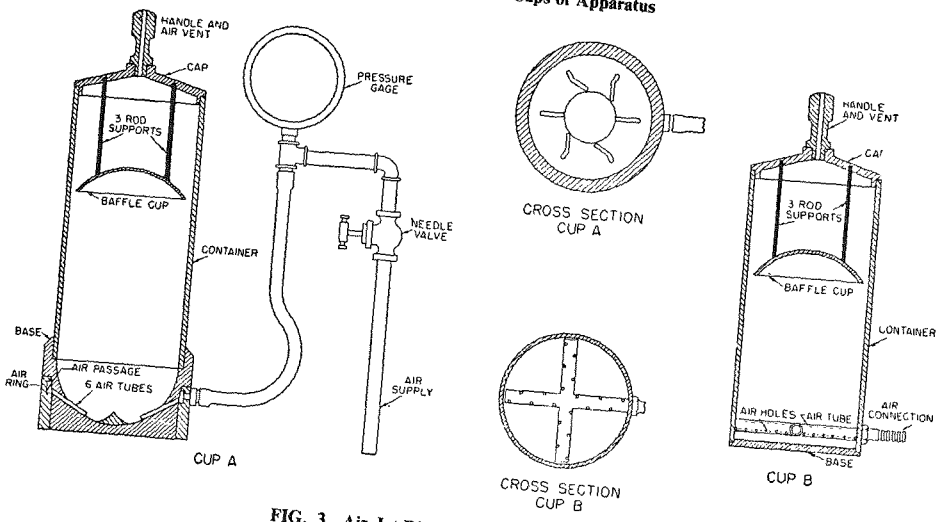
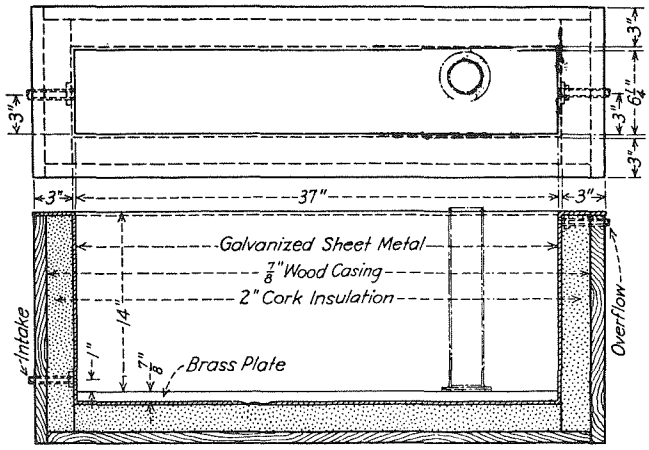


FIG. 3 Air-Jet Dispersion Cups of Apparatus B



Metric Equivalents

in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Analyze soil samples for heating value (Btu).

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 concentration):

Analyze 11 soil samples for heating value.

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: Daily by Overnight Carrier

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

Laboratory will report results within 30 days of sample receipt.

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

Heating Value: ASTM Method D2015-77

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

1. Exact compliance to the specific requirements of the method must be met. Particularly the restandardization requirements.
2. A reference standard of benzoic acid from the NBS for heating value must be run with each sample set. The acceptable range is +/- 6 Btu/C.

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.

1. Copies of Thermometer's Certificate of NBS Certification.
2. Logs of restandardization data.
3. Copies of all log pages particularly the calculations.

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

The correlation coefficient for standards must be calculated and supplied.

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Heat Value	Repeatability	<50 Btu/lb Dry Weight

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Blank	1 per 10 Samples	As specified in the method
Restandardization	As neceassary	As specified in the method
Duplicates	1 per 10 Samples	<50 Btu/lb Dry Weight

Correlation coeffiecient for standards must be > 0.98

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Designation: D 2015 – 85

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for GROSS CALORIFIC VALUE OF COAL AND COKE BY THE ADIABATIC BOMB CALORIMETER[1]

This standard is issued under the fixed designation D 2015; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the gross calorific value of coal and coke by the adiabatic bomb calorimeter.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 9.

2. Applicable Documents

2.1 *ASTM Standards:*

D 121 Definitions of Terms Relating to Coal and Coke[2]

D 346 Method of Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 1193 Specification for Reagent Water[3]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke[2]

D 3180 Method for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

D 4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods[2]

E 1 Specification for ASTM Thermometers[4]

E 144 Recommended Practice for Safe Use of Oxygen Combustion Bombs[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *calorific value*—the heat produced by combustion of a unit quantity of a substance under specified conditions. It is expressed in this test method in British thermal units per pound (Btu/lb). Calorific value may also be expressed in calories per gram (cal/g) or in the International System of Units (SI), joules per gram (J/g), when required. The unit equivalents are given in Table 1.

3.1.2 *gross calorific value (gross heat of combustion at constant volume) Q_v (gross)*—see Definitions D 121.

3.1.3 *net calorific value (net heat of combustion at constant pressure) Q_p (net)*—see Definitions D 121.

3.1.4 *calorimeter*—as used in this test method, consists of the bomb and its contents, the calorimeter vessel (bucket) with stirrer, the water in which the bomb is immersed, and the portions of the thermometer and the ignition leads within the calorimeter vessel.

3.2 *Description of Terms Specific to This Standard:*

3.2.1 *corrected temperature rise*—the temperature change of the calorimeter caused by the process that occurs inside the bomb, that is, the observed temperature change corrected for various effects as noted in 10.4.1.

NOTE 1—Temperature is measured in either degrees

[1] This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved Aug. 23, 1985. Published October 1985. Originally published as D 2015 – 62 T. Last previous edition D 2015 – 77 (1978).

[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

Celsius or degrees Fahrenheit. Thermometer corrections should be applied. Temperatures may be recorded in ohms or other arbitrary units instead of degrees. Consistent units must be used in standardization and the actual calorific value determination. If arbitrary units other than degrees Celsius or Fahrenheit are used, the temperature interval over which all tests are made, must not vary so much that an error greater than 0.001°C would be caused.

3.2.2 *energy equivalent, heat capacity, or water equivalent*—the energy required to raise the temperature of the calorimeter one arbitrary unit. This is the quantity that, when multiplied by the corrected temperature rise, then adjusted for extraneous heat effects, and divided by the mass of the sample, gives the gross calorific value.

NOTE 2—Energy units for quantities listed throughout this test method are such that the number of energy units per gram of sample corresponds exactly to the number of Btu's per pound of sample. For brevity these are referred to as Btu's. The actual energies are smaller than those stated by the ratio of the number of pounds per gram (1/453.59). The energy equivalent of the calorimeter has the units (Btu/lb) times (g/degree). Conversion to other units is discussed in Appendix X1.2. Time is expressed in minutes. Mass is expressed in grams.

4. Summary of Method

4.1 Calorific value is determined in this test method by burning a weighed sample, in oxygen, in a calibrated adiabatic bomb calorimeter under controlled conditions. The calorimeter is standardized by burning benzoic acid. The calorific value of the sample is computed from temperature observations made before, during and after combustion, making proper allowances for heat contributed by other processes, and for thermometer and thermochemical corrections.

NOTE 3—Oxidation after sampling of susceptible low-rank coal or lignite may result in a reduction of calorific value. Unnecessary exposure of the sample to air from the time of sampling or delay in analysis shall be avoided.

5. Significance and Use

5.1 The gross calorific value is used to compute the total calorific content of the quantity of coal represented by the sample for payment purposes, provided the buyer and the seller mutually agree upon this.

5.2 The gross calorific value is used in computing the calorific value versus sulfur content to determine if the coal meets regulatory requirements for industrial fuels.

5.3 The gross calorific value may be used for evaluating the effectiveness of beneficiation processes, or for research purposes.

6. Apparatus and Facilities

6.1 *Test Space,* shall be a room or area free from drafts and that can be kept at a reasonably uniform temperature for the time required for the determination. The apparatus should be shielded from direct sunlight and radiation from other heat sources. Thermostatic control of room temperature and controlled relative humidity are desirable.

6.2 *Combustion Bomb,* shall be constructed of materials that are not affected by the combustion process or products sufficiently to introduce measureable heat input or alteration of end products. The bomb must be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There must be no gas leakage during a test. The bomb must be capable of withstanding a hydrostatic pressure test of 20 MPa (3000 psig) at room temperature without stressing any part beyond its elastic limit.

6.3 *Balance,* shall be a laboratory balance having capability to weigh the sample to the nearest 0.0001 g. The balance should be checked periodically to determine is accuracy.

6.4 *Calorimeter Vessel,* shall be made of metal with a tarnish-resistant coating, and with all outer surfaces highly polished. Its size shall be such that the bomb will be completely immersed in water when the calorimeter is assembled. It shall have a device for stirring the water thoroughly and at a uniform rate, but with minimum heat input. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01°C (0.02°F) starting with identical temperatures in the calorimeter, room, and jacket. The immersed portion of the stirrer shall be coupled to the outside through a material of low-heat conductivity.

6.5 *Jacket,* shall be a double-walled, water-filled jacket fully enclosing the calorimeter. The sides, top, and bottom of the calorimeter vessel shall be approximately 10 mm from the inner wall of the jacket to minimize convection currents. Mechanical supports for the calorimeter vessel shall provide as little thermal conduction as possible. The jacket shall have a device for stirring the water thoroughly and at a uniform rate with minimum heat input.

6.6 *Thermometers,* used to measure temperature in the calorimeter and jacket shall be any of

the following types or combinations thereof:

6.6.1 *Liquid-in-Glass Thermometers,* conforming to the requirements for ASTM Thermometers 56C, 56F, 116C, or 117C as prescribed in Specification E 1. The thermometers shall be tested for accuracy against a known standard (preferably by the National Bureau of Standards). For Thermometers 56C and 56F the calibration should be at intervals no larger than 2.0°C or 2.5°F over the entire graduated scale. The maximum difference in correction between any two test points shall be no more than 0.02°C or 0.05°F. For Thermometers 116C and 117C, the calibration should be at intervals no larger than 0.5°C over the entire calibrated range. The maximum difference in correction between any two test points shall not be more than 0.02°C.

6.6.2 *Beckman Differential Thermometer,* (glass enclosed scale, adjustable), having a range of approximately 6°C in 0.01°C subdivisions reading upward and conforming to the requirements for Thermometer 115C, as prescribed in Specification E 1, may be used. Each of these thermometers shall be tested for accuracy against a known standard (preferably by the National Bureau of Standards) at intervals no larger than 1°C over the entire graduated scale. The maximum difference in the correction between any two test points shall not be more than 0.02°C.

6.6.3 *Other Thermometers,* of an accuracy equal to or better than 0.001°C, such as platinum resistance or linear thermistor thermometers, are satisfactory and may be used if properly calibrated. A Wheatstone bridge and galvanometer capable of measuring resistance to 0.0001 Ω are necessary for use with 25 Ω platinum resistance thermometers.

6.7 *Thermometer Accessories*—A magnifier is required for reading liquid-in-glass thermometers to one-tenth of the smallest scale division. This shall have a lens and holder designed so as to introduce no significant errors due to parallax.

6.8 *Sample Holder,* shall be an open crucible of platinum, quartz, or acceptable base-metal alloy. Base-metal alloy crucibles are acceptable, if after a few preliminary firings, the weight does not change significantly between tests.

6.9 *Ignition Wire,* shall be 100 mm of 0.16 mm diameter (No. 34 B & S gage) nickel-chromium (Chromel C) alloy or iron wire. Platinum or palladium wire, 0.10 mm diameter (No. 38 B & S gage), may be used, provided constant ignition energy is supplied. The length, or mass, of the ignition wire shall remain constant for all calibrations and calorific value determinations.

6.10 *Ignition Circuit,* for ignition purposes shall provide 6 to 16 V alternating or direct current to the ignition wire. An ammeter or pilot light is required in the circuit to indicate when current is flowing. A step-down transformer, connected to an alternating current lighting circuit or batteries, may be used.

6.11 *Buret,* used for the acid titration shall have 0.1-mL divisions.

6.12 *Automated Controller and Temperature Measuring Accessories,* may be used.

7. Reagents

7.1 *Reagent Water,* conforming to Type II of Specification D 1193, shall be used for preparation of reagents and washing of the bomb interior.

7.2 *Purity of Reagents,* reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[6] 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.

7.3 *Benzoic Acid,* (C_6H_5COOH), shall be the National Bureau of Standards benzoic acid. The crystals shall be pelleted before use. Commercially prepared pellets may be used provided they are made from National Bureau of Standards benzoic acid. The value of heat of combustion of benzoic acid for use in the calibration calculations shall be in accordance with the value listed in the National Bureau of Standards certificate issued with the standard.

7.4 *Methyl Orange, Methyl Red, or Methyl Purple Indicator,* may be used to titrate the acid formed during combustion. The indicator used shall be the same for both calibration and calorific value determinations.

7.5 *Oxygen,* shall be free of combustible matter. Only oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure,

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

should be used. Oxygen made by the electrolytic process may contain a small amount of hydrogen rendering it unfit without purification.

7.6 *Sodium Carbonate Standard Solution,* (Na_2CO_3), should be dried for 24 h at 105°C. Dissolve 20.9 g in water and dilute to 1 L. One millilitre of this solution is equivalent to 10.0 Btu in the nitric acid (HNO_3) titration.

8. Sample

8.1 The sample shall be the material pulverized to pass No. 60 (250-μm) sieve, prepared in accordance with either Mehod D 346 for coke, or Method D 2013 for coal.

8.2 A separate portion of the analysis sample should be analyzed simultaneously for moisture content in accordance with Method D 2013 and Test Method D 3173, so that calculation to other bases can be made.

8.3 Sulfur analysis shall be made in accordance with Test Methods D 3177.

9. Safety Precautions

9.1 The following precautions are recommended for safe calorimeter operation. Additional precautions are given in Recommended Practice E 144. Also consult the calorimeter manufacturer's installation and operating manuals before using the calorimeter.

9.2 The mass of coal or coke sample and the pressure of the oxygen admitted to the bomb must not exceed the manufacturer's recommendations.

9.3 Bomb parts should be inspected carefully after each use. Threads of the main closure should be checked frequently for wear. Cracked or significantly worn parts should be replaced. The bomb should be returned to the manufacturer occasionally for inspection and possible proof firing.

9.4 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety code. Suitable reducing valves and adaptors for 3 to 4-MPa (300 to 500-psi) discharge pressure are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

9.5 During ignition of a sample, the operator must not permit any portion of her/his body to extend over the calorimeter.

9.6 When combustion aids are employed, extreme caution must be exercised not to exceed the bomb manufacturer's recommendations and to avoid damage to the bomb. Do not fire loose fluffy material such as unpelleted benzoic acid, unless thoroughly mixed with the coal sample.

9.7 Do not fire the bomb if the bomb has been dropped or turned over after loading, or if there is evidence of a gas leak when the bomb is submerged in the calorimeter water.

9.8 For manually operated calorimeters, the ignition circuit switch shall be of the momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the charge.

10. Standardization

10.1 The calorimeter is standardized by combustion of benzoic acid.

10.2 Determine the energy equivalent of the calorimeter for a specific temperature rise as the average of a series of ten individual runs made over a period of not less than 3 days nor more than 5 days. To be acceptable, the standard deviation of the series shall be 6.5 Btu/°C (3.6 Btu/°F) or less (see Table 2). For this purpose, any individual test may be discarded only if there is evidence indicating incomplete combustion. If this limitation is not met, investigate for the source of the problem, correct it, then repeat the entire series to obtain a standard deviation within the acceptable limit.

10.3 *Procedure:*

10.3.1 Regulate the weights of the pellets of benzoic acid in each series to yield approximately the same temperature rise as that obtained with the coal tested in the same laboratory. The usual range of masses is 0.9 to 1.3/g. Weigh the pellet to the nearest 0.0001 g in the sample holder in which it is to be burned, and record the weight as the mass.

10.3.2 Rinse the bomb, invert to drain, and leave undried. Add 1.0 mL of water to the bomb prior to assembly for a determination.

10.3.3 Connect a measured length of ignition wire to the ignition terminals, with enough slack to allow the ignition wire to maintain contact with the sample.

10.3.4 Assemble the bomb and charge it with oxygen to a consistent pressure between 2 to 3

MPa (20 and 30 atm). This pressure must remain the same for each calibration and each calorific-value determination. Admit the oxygen slowly into the bomb so as not to blow powdered material from the sample holder. If the pressure exceeds the specified pressure, do not proceed with the combustion. Instead, detach the filling connection, exhaust the bomb in the usual manner, and discard the sample.

10.3.5 Fill the calorimeter vessel (bucket) with the measured (or weighed) quantity of water adjusted from 1.0 to 2.0°C (2.0 to 4.0°F) below room temperature, but not lower than 20°C (68°F). Use the same mass of water in each test weighed to +0.5 g. For 2000-mL calorimeters, the proper quantity can be obtained by use of a volumetric flask calibrated to deliver 2000 ± 0.5 mL. As the density of water varies with temperature, make suitable corrections if the water temperature varies from the temperature at which the flask was calibrated. Place the assembled bomb in the calorimeter vessel. Check that no oxygen bubbles are leaking from the bomb. Place the calorimeter vessel in the jacket; connect the electrodes; place the stirrers, thermometers, and cover in position. Start the stirrers and continue to operate them throughout the determination. Examine the thermometers for liquid separation and correct any separation before proceeding. The starting temperature should be within ±0.5°C (0.9°F) of that used in analysis of coal or coke samples.

NOTE 4—Check all liquid-in-glass thermometers at least daily for defects, for example, cracked glass, etc.

10.3.6 Allow 5 min for attainment of equilibrium. Adjust the jacket temperature to match the calorimeter temperature within 0.01°C (0.02°F) and maintain for 3 min. Use a magnifier when using ASTM Bomb Calorimeter Thermometers 56C or 56F, and estimate all readings (except those during the rapid-rise period) to the nearest 0.002°C or 0.005°F. Estimate ASTM Thermometers 115C, 116C, or 117C readings to 0.001°C, and 25 Ω resistance thermometer readings to the nearest 0.0001 Ω. Tap mercury thermometers (for instance, with a pencil) just before reading to avoid errors caused by mercury sticking to the walls of the capillary. Record the "initial temperature", t_i, 20°C (68°F) or higher, to within one-tenth of the smallest thermometer subdivision and ignite the charge. Adjust the jacket temperature to match that of the calorimeter temperature during the period of rise; keep the two temperatures as nearly equal as possible during the rapid rise and adjust to within 0.01°C (0.02°F) when approaching the final equilibrium temperature. Take calorimeter temperature readings at 1-min intervals until the same temperature, within one-tenth of the smallest thermometer subdivision, is observed in three successive readings. Record this as the "final temperature", t_f.

10.3.7 Open the cover and remove the bomb. Release the pressure at a uniform rate, such that the operation will require not less than 1 min. Open the bomb and examine the bomb interior. Discard the test if unburned sample or sooty deposits are found. Wash the interior of the bomb with distilled water containing the titration indicator, until the washings are free of acid, and titrate the washings with standard sodium carbonate solution.

10.3.8 Remove and measure, or weigh, the combined pieces of unburned ignition (firing) wire and subtract from the original length, or weigh to determine the wire consumed in firing. If the wire is weighed, remove the ball of oxidized metal from the end of each piece of wire before weighing.

10.4 *Calculations:*

10.4.1 *Temperature Rise*—Using data obtained as prescribed in 10.3.6, compute the corrected temperature rise, t, as follows:

$$t = t_f - t_i + C_e + C_s \quad (1)$$

where:

t = corrected temperature rise, °C or °F,
t_i = initial temperature reading at time of firing,
t_f = final temperature reading,
C_e = thermometer emergent stem correction, if required (see Note 5 and Annex A1.1.4, and
C_s = thermometer setting correction, if required (see Note 5 and Annex A1.1.3)

NOTE 5—With all mercury-in-glass thermometers, it is necessary to make corrections if the total calorific value is altered by 5.0 Btu or more. This represents a change of 0.001°C or 0.002°F in a calorimeter using approximately 2000 g of water. Beckmann thermometers also require a setting correction and an emergent stem correction (Annex A1.1.3 and A1.1.4). Solid-stem ASTM Thermometers 56C and 56F do not require emergent stem corrections if all tests, including standardization, are performed within the same 5.5°C (10°F) interval. If operating temperatures range beyond this limit, a differential emergent stem correction (Annex A1.1.4) must be applied to the corrected tempera-

ture rise, t, in all tests including standardization.

10.4.2 *Thermochemical Corrections* (see Appendix X1.1, X1.2, and X1.3)—Compute the following for each test:

e_1 = correction for the heat of formation of HNO_3, in Btu. Each millilitre of standard Na_2CO_3 is equivalent to 10.0 Btu, and

e_2 = correction for heat of combustion of firing wire, in Btu (Note 6).
0.41 Btu/mm or 2.6 Btu/mg for No. 34 B & S gage Chromel C.
0.49 Btu/mm or 3.2 Btu/mg for No. 34 B & S gage iron wire.

NOTE 6—There is no correction for platinum wire provided the ignition energy is constant.

10.4.3 Compute the calorimeter energy equivalent, E, by substituting in the following:

$$E = [(Hg) + e_1 + e_2]/t \quad (2)$$

where:

E = calorimeter energy equivalent (Note 8),
H = heat of combustion of benzoic acid, as stated in the National Bureau of Standards Certificate, Btu/lb in air,
g = mass (weight in air) of benzoic acid, g,
e_1 = titration correction (10.4.2),
e_2 = fuse wire correction (10.4.2), and
t = corrected temperature rise.

NOTE 7—Using the units and corrections as given in 10.4.1 and 10.4.2, the energy equivalent of the calorimeter is such that the calorific value of the coal sample will be obtained directly in British thermal units per pound when the mass of sample is taken in grams. The units of the energy equivalent are therefore: (Btu/lb) times (g/deg).

10.5 Repeat the procedure for a total of ten determinations. Compute the standard deviation as illustrated in Table 2.

11. Restandardization

11.1 Make checks on the energy equivalent value after changing the oxygen supply, after changing any part of the calorimeter, and at least once a month otherwise.

11.1.1 If a single new determination differs from the old value by 6 Btu/°C (4 Btu/°F), the old standard is suspect, thereby requiring a second test.

11.1.2 The difference between the two new determinations must not exceed 8 Btu/°C (5 Btu/°F), and the average of the two new determinations must not differ from the old standard by more than 4 Btu/°C (3 Btu/°F). If these requirements are met, do not change the calorimeter standard.

11.1.3 If the requirements given in 11.1.2 are not met, two more determinations must be run. The range of the four values must not exceed 14 Btu/°C (8 Btu/°F), and the average of the four new determinations must not differ from the old standard value by more than 3 Btu/°C (2 Btu/°F). If these requirements are met, do not change the calorimeter standard.

11.1.4 If the requirements given in 11.1.3 are not met, a fifth and sixth determination must be run. The range of the six new values must not exceed 17 Btu/°C (10 Btu/°F), and the average of the six new values must not differ from the old standard value by more than 2 Btu/°C (2 Btu/°F). If these requirements are met, do not change the calorimeter standard.

11.1.5 If the requirements given in 11.1.4 are not met, four more determinations must be run to complete a series of ten runs. The range of these ten results must not exceed 20 Btu/°C (12 Btu/°F), and the average of the ten new standards must not differ from the old standard by more than 1 Btu/°C (1 Btu/°F). If these requirements are met, do not change the calorimeter standard.

11.1.6 If the requirements given in 11.1.5 are not met, the average value from the ten new values must be used for the new standard energy equivalent, provided that the standard deviation of the series does not exceed 6.5 Btu/°C (3.6 Btu/°F).

11.2 The summary of the numerical requirements at each stage of restandardization is given in Table 3.

12. Procedure for Coal and Coke Samples (Note 8)

12.1 Thoroughly mix the analysis sample of coal or coke in the sample bottle and carefully weigh approximately 1 g of it into the sample holder. The sample shall be weighed to the nearest 0.0001 g. Make each determination in accordance with the procedure described in 10.3.2 through 10.3.8.

NOTE 8—For anthracite, coke, and coal of high ash content, that do not readily burn completely, one of the following procedures are recommended: (*1*) The inside of the sample holder is lined completely with ignited asbestos in a thin layer pressed well down in the angles, and the sample is then sprinkled evenly over the surface of the asbestos. (*2*) The mass of the sample may be varied to obtain good ignition. If the mass is varied, it will be necessary to recalibrate the calorimeter

so that the water equivalent will be based on the same temperature rise as that obtained with the sample weight. (*3*) A known amount of benzoic acid may be mixed with the sample. Proper allowance must be made for the heat of combustion of benzoic acid when determining the calorific value of the sample.

NOTE 9—For the calorific value of coke, it is necessary to use 3-MPa (30-atm) pressure for both standardization and analysis.

12.2 Determine the sulfur content of the sample by any of the procedures described in Test Methods D 3177.

13. Calculations (Note 2)

13.1 Compute the corrected temperature rise, t, as shown in 10.4.1.

13.2 *Thermochemical Corrections* (Appendix X1)—Compute the following for each test:

e_1 = correction for the heat of formation of HNO_3 in Btu. Each millilitre of standard sodium carbonate is equivalent to 10.0 Btu,

e_2 = correction for heat of combustion of ignition wire, Btu,
= 0.41 Btu/mm or 2.6 Btu/mg for No. 34 B & S gage Chromel C wire,
= 0.49 Btu/mm or 3.2 Btu/mg for No. 34 B & S gage iron wire, and

e_3 = correction for difference between heat of formation of H_2SO_4 from the heat of formation of HNO_3, in Btu,
= 23.7 times percent of sulfur in sample times mass of sample in g.

14. Calorific Value (Note 10)

14.1 *Gross Calorific Value*—Calculate the gross calorific value (gross heat of combustion at constant volume), Q_v (gross), as follows:

$$Q_v \text{ (gross)} = [(tE) - e_1 - e_2 - e_3]/g \quad (3)$$

where:

Q_v (gross) = gross calorific value, Btu/lb,
t = corrected temperature rise calculated in 13.1,
E = energy equivalent calculated in 10.4.3,
e_1, e_2, e_3 = corrections as prescribed in 13.2, and
g = mass of sample, g.

14.2 *Net Calorific Value*—Calculate the net calorific value (net heat of combustion at a constant pressure), Q_p, (net) as follows:

$$Q_p \text{ (net)} = Q_v \text{ (gross)} - 10.30\ (H \times 9)$$

where:

Q_p (net) = net calorific value, Btu/lb,
Q_v (gross) = gross calorific value, Btu/lb, and
H = total hydrogen, %.

NOTE 10—This calculation gives calorific value in Btu/lb. To obtain calorific value in Jg, see Appendix X2.

15. Report

15.1 The results of the calorific value may be reported on any of a number of bases, differing from each other in the manner that moisture is treated.

15.2 Use the percentage of moisture in the sample passing a No. 60 (250-μm), sieve (Test Method D 3173) to calculate the results of the analysis sample to a dry basis.

15.3 Procedures for converting the value obtained on the analysis sample to other bases are described in Method D 3180.

16. Precision and Bias

16.1 The following criteria should be used for judging the acceptability of results (95 % probability) on split 60-mesh (250-μm) sample.

16.1.1 *Repeatability*—Duplicate results by the same laboratory, using the same operator and equipment, should not be considered suspect unless they differ by more than 50 Btu/lb on a dry basis.

16.1.2 *Reproducibility*—The results submitted by two or more laboratories (different equipment, operators, date of test, and different portions of the same pulp) should not be considered suspect unless the two results differ by more than 100 Btu/lb on a dry basis.

16.2 *Bias*—There should be no bias because the equipment is standardized with a compound having a known heat of combustion.

TABLE 1 Calorific Value

1 Btu = 1055.06 J	1 Btu/lb = 2.326 J/g
1 Calorie[A] = 4.1868 J	1.8 Btu/lb = 1.0 cal/g

[A] International tables calorie.

TABLE 2 Standard Deviations for Calorimeter Standardization[A]

Standardization Number	Column A	Column B	Column C
	Energy Equivalent (Btu/lb) × (g/°C)	Code to 4400 (Column A − 4400)	(Column B)^2
1	4412	12	144
2	4407	7	49
3	4415	15	225
4	4408	8	64
5	4404	4	16
6	4406	6	36
7	4409	9	81
8	4410	10	100
9	4412	12	144
10	4409	9	81
SUM		92	940

Average = $\bar{X} = \Sigma X/10 = (92/10) + 4400 = 4409$

Variance = $s^2 = \dfrac{\Sigma \text{ Column C} - [(\Sigma \text{ Column B})^2/n]}{n-1} = \dfrac{940 - [(92)^2/10]}{9} = 10.4$

Standard deviation = $s = \sqrt{\text{variance}} = \sqrt{10.4} = 3.22$

[A] In this example the values of energy equivalent are typical for a calorimeter calibrated so that, if the energy equivalent is multiplied by the temperature rise in degrees Celsius per gram of sample, the calorific value of the sample will be obtained in British Thermal units per pound.

TABLE 3 Summary of Numerical Requirements

NOTE—Test values exceeding table limits require additional runs.[A]

Number of Runs	Maximum Range of Results		Maximum Difference between $\bar{X}_1$ and $\bar{X}_2$[B]	
	Btu/°C	Btu/°F	Btu/°C	Btu/°F
1	...	...	±6	±4
2	8	5	±4	±3
4	14	8	±3	±2
6	17	10	±2	±2
10	20	12	±1	±1

[A] Values in this table have been rounded off after statistical calculation, and are therefore not precisely in a ratio from 1.8 to 1.0.
[B] $\bar{X}_1$ = average of original standard. $\bar{X}_2$ = average of check runs.

ANNEX

(Mandatory Information)

A1. THERMOMETRIC CORRECTIONS

A1.1 Thermometer Corrections

A1.1.1 It is necessary to make the following individual corrections, if not making the correction would result in an equivalent change of 5.0 Btu or more.

A1.1.2 *Calibration Correction* shall be made in accordance with the calibration certificate furnished by the calibration authority.

A1.1.3 *Setting Correction* is necessary for the Beckmann thermometer. It shall be made in accordance with the directions furnished by the calibration authority.

A1.1.4 *Differential Emergent Stem Correction*—The calculation of differential stem correction depends upon the way the thermometer was calibrated and how it was used. Two conditions are possible:

A1.1.4.1 *Thermometers Calibrated in Total Immersion and Used in Partial Immersion*—This emergent stem correction is made as follows:

$$\text{Correction} = C_e = K\,(t_f - t_i)\,(t_f + t_i - L - T)$$

where:

C_e = emergent stem correction,
K = 0.00016 for thermometers calibrated in °C,
= 0.0009 for thermometers calibrated in °F,
L = scale reading to which the thermometer was immersed,
T = mean temperature of emergent stem,
t_i = initial temperature reading, and,
t_f = final temperature reading.

NOTE A1.1—*Example:* Assume the point L, to which the thermometer was immersed was 16°C; its initial reading, t_i, was 24.127°C, its final reading, t_f, was 27.876, the mean temperature of the emergent stem, T was 26°C; then:

Differential stem correction, C_e
= 0.00016 (28 − 24) (28 + 24 − 16 − 26)
= + 0.0064°C.

A1.1.4.2 *Thermometers Calibrated and Used in Partial Immersion, but at a Different Temperature than the Calibration Temperature*—This emergent stem correction is made as follows:

$$\text{Correction} = C_e = K\,(t_f - t_i)\,(t_c - t_o)$$

where:

C_e = emergent stem correction,
K = 0.00016 for thermometers calibrated in °C,
= 0.00009 for thermometers calibrated in °F,
t_i = initial temperature reading,
t_f = final temperature reading,
t_o = observed stem temperature, and
t_c = stem temperature at which the thermometer was calibrated.

NOTE A1.2—*Example:* Assume the initial reading, t_i, was 80°F, the final reading, t_f, was 86°F, and that the observed stem temperature, t_o, was 82°F, and calibration temperature, t_c, was 72°F then:

Differential stem correction:
= 0.00009 (86 − 80) (82 − 72)
= 0.005°F

APPENDIXES

(Nonmandatory Information)

X1. THERMOCHEMICAL CORRECTIONS

X1.1 *Energy of Formation of Nitric Acid*—A correction, e_1, (10.4.2 and 13.2), is applied for the acid titration. This correction is based on the assumptions (*1*) that all the acid titrated is HNO_3 formed by the following reaction: 1/2 N_2 (g) + 5/4 O_2 (g) + 1/2 H_2O (1) = HNO_3 (in 500 mol H_2O), and (*2*) that the energy of formation of HNO_3 in approximately 500 mol of water under bomb conditions is minus 59.0 kJ/mol.[7]

X1.1.1 A convenient concentration of Na_2CO_3 is 0.394 N (20.9 g Na_2CO_3/1000 mL) which gives e_1 = 10 times V, where V is the volume of Na_2CO_3 in millilitres. The factor 10.0 (0.394 × 59.0 = 2.326) is to be used for calculating calorific value in Btu/lb. For other units see Table X2.1. When H_2SO_4 is also present, a part of the correction for H_2SO_4 is contained in the e_1 correction and remainder in the e_3 correction.

[7] Calculated from data in National Bureau of Standards Technical Note 270-3.

X1.2 *Energy of Formation of Sulfuric Acid*—By definition (see Definitions D 121) the gross calorific value is obtained when the product of the combustion of sulfur in the sample is SO_4 (*g*). However, in actual bomb combustion process, all the sulfur is found as H_2SO_4 in the bomb washings. A correction, e_3 (see 13.2) is applied for the sulfur that is converted to H_2SO_4. This correction is based upon the energy of formation of H_2SO_4 in solutions, such as will be present in the bomb at the end of a combustion. This energy is taken as −295.0 kJ/mol.[8] A correction of 2 times 59.0 kJ/mol of sulfur was applied in the e_1 correction, so the additional correction necessary is 295.0 − (2 times 59.0) = 177 kJ/mol, or 5.52 kJ/g of sulfur in the sample (55.2 J times weight of sample in grams times percent sulfur in sample). This causes e_2 to be 23.7 times weight of sample in grams times percent sulfur in sample. The factor 23.7 (equals 55.2/2.326), for e_3 (see 13.2) is to be used for calculating calorific value in Btu/lb. For other units, see Appendix X2. The values above are based on a coal containing about 5 % sulfur and about 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in the water condensed during combustion of the sample.

X1.2.1 If a 1-g sample of such a fuel is burned, the resulting H_2SO_4 condensed with water formed on the walls of the bomb, will have a ratio of about 15 mol of water to 1 mol of H_2SO_4. For this concentration, the energy of the reaction SO_2 (*g*) + ½ O_2 + H_2O (1) = H_2SO_4 (in 15 moles of H_2O) under the conditions of the bomb process is −295.kJ/mol.[9] Basing the calculation upon a sample of comparatively large sulfur content reduces the possible overall errors, because, for small percentages of sulfur, the correction is smaller.

X1.3 *Fuse (Ignition) Wire*—Calculate the energy contributed by burning the fuse wire in accordance with the directions furnished by the supplier of the wire. For example, the energy of the combustion of No. 34 B & S gage Chromel C wire is 6.0 J/mg or approximately 0.95 J/mm. For calculating e_2 for use in Eqs 2 and 3, these give e_2 = 0.41 times length (mm) of wire or e_2 = 2.6 times weight (mg) of wire. The energy required to melt a platinum wire is constant for each experiment if the same amount of platinum wire is used. As the energy is small, its effect is essentially cancelled out in the relationship between the standardization experiments and the calorific value determinations, and it can be neglected. The factors listed above for e_2 (10.4.2 and 13.2) are suitable for calculating calorific value in Btu/lb. For other units, see Appendix X2.

[8] Calculated from data in National Bureau of Standards Circular 500.

[9] Mott, R. A. and Parker, C. "Studies in Bomb Calorimetry IX—Formation of Sulfuric Acid", *Fuel*, FUELB, Vol. 37, 1958, p. 371.

X2. REPORTING RESULTS IN OTHER UNITS

X2.1 *Reporting Results in Joules per Gram:*

X2.1.1 The gross calorific value can be expressed in joules per gram, calories per gram, or British thermal units per pound. The relationships between these units are given in Table 1.

X2.1.2 Because the energy of combustion of the reference material is measured and certified by the National Bureau of Standards in joules per gram, the most straightforward usage of the reference material would lead to the calorific value of the fuel in joules per gram. To carry out this procedure, we make changes outlined in X2.1.3 through X2.1.5.

X2.1.3 For calculating energy equivalent, substitute Eq 2′ for Eq 2:

$$E = [(H'g) + e_1']/t \qquad (2')$$

where the meanings of the symbols in Eq 2′ are the same as in Eq 2 except that:

E' = energy equivalent in units of joules per temperature unit,

H' = the heat of combustion of reference material in units of joules per gram weight in air (J/g from the certificate for the NBS benzoic acid), and

e_1' and e_3' = corrections in units of joules, (see Table X2.1).

X2.1.4 For calculating gross calorific value, substitute Eq 3′ for Eq 3:

$$Q_v \text{ (gross)} = [(t_E') - e_1' - e_2']/g \qquad (3')$$

where the meanings of the symbols in Eq 3′ are the same as in Eq 3 except that:

Q_v (gross) = gross calorific value with units of joules per gram (weight in air),

E' = energy equivalent units, of joules per temperature unit, and

e_1', e_2', and e_3' = corrections in units of joules (see Table X2.1).

X2.1.5 *Precision:*

X2.1.5.1 *Repeatability*—Duplicate results by the same laboratory, using the same operator and equipment, should not be considered suspect unless they differ by more than 120 J/g.

X2.1.5.2 *Reproducibility*—The results submitted by two or more laboratories (different equipment, operators, date of test, and different portions of the same sample) should not be considered suspect unless the results differ by more than 240 J/g.

TABLE X2.1 Alternative Thermochemical Correction Factors (Units in Joules)[A]

Correction	Multiplication Factor	Multiply By
e_1' (HNO_3)	20 J/mL	mL of 0.34 *N* Na_2CO_3
e_3' (H_2SO_4)	55.2 J/cgS	percentage of sulfur in sample times mass of sample in grams
e_2' (fuse wire)	0.95 J/mm	length (mm) of No. 34 B & S gage Chromel C wire
or		
e_2' (fuse wire)	1.14 J/mm	length (mm) of No. 34 B & S gage iron wire
e_2' (fuse wire)	6.0 J/mg	mass (mg) of Chromel C wire
or		
e_2' (fuse wire)	7.4 J/mg	mass (mg) of iron wire

[A] To be used in Eqs 2′ and 3′ only.

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Percent moisture analysis will be performed at a physical soil testing laboratory.

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 concentration):

Analyze 22 subsurface soil samples for percent moisture.

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: Daily by Overnight Carrier

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

Laboratory will report results within 30 days of sample receipt.

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

ASTM Method D2216-80: Standard Method for Laboratory Determination of Water Content of Soil, Rock, and Soil-aggregate Mixtures.

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

Use only the method specified above. Obtain approval of CPMS, CRL prior to use of any other method. Rewrite SAS request to reflect new methodology.

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.**

Report all raw data and parameter values used in making calculations of moisture content.

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

11. **Name of sampling/shipping contact:** Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Moisture Content	1%	Duplicates within 10%

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	One per 10 for sets >10 Two for sets <10	10%

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

AMERICAN NATIONAL STANDARD ANSI/ASTM D 2216 - 80

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for LABORATORY DETERMINATION OF WATER (MOISTURE) CONTENT OF SOIL, ROCK, AND SOIL-AGGREGATE MIXTURES[1]

This standard is issued under the fixed designation D 2216; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the laboratory determination of the water (moisture) content of soil, rock, and soil-aggregate mixtures by weight. For simplicity, the word "material" hereinafter refers to either soil, rock, or soil-aggregate mixtures, whichever is most applicable.

1.2 The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles.

1.3 This method does not give true representative results for: materials containing significant amounts of halloysite, montmorillonite, or gypsum minerals; highly organic soils; or, materials in which the pore water contains dissolved solids (such as salt in the case of marine deposits). For a material of the previously mentioned types, a modified method of testing or data calculation may be established to give results consistent with the purpose of the test.

2. Summary of Method

2.1 The practical application in determining the water content of a material is to determine the mass of water removed by drying the moist material (test specimen) to a constant mass in a drying oven controlled at 110 ± 5°C and to use this value as the mass of water in the test specimen. The mass of material remaining after oven-drying is used as the mass of the solid particles.

3. Significance and Use

3.1 For many soil types, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and an index property.

3.2 The water content of a soil is used in almost every equation expressing the phase relationships of air, water, and solids in a given volume of material.

3.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limit, is used to express its relative consistency or liquidity index.

3.4 The term "water" as used in geotechnical engineering, is typically assumed to be "pore" or "free" water and not that which is hydrated to the mineral surfaces. Therefore, the water content of materials containing significant amounts of hydrated water at in-situ temperatures or less than 110°C can be misleading.

3.5 The term "solid particles" as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, etc), water-soluble matter (such as salt) and highly organic

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock for Engineering Purposes and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

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matter typically require special treatment or a qualified definition of water content.

4. Apparatus

4.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, and maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

4.2 *Balances*, having a precision (repeatability) of ±0.01 g for specimens having a mass of 200 g or less, ±0.1 g for specimens having a mass of between 200 and 1000 g, or ± 1 g for specimens having a mass greater than 1000 g.

4.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and a change in mass upon repeated heating, cooling, and cleaning. Containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used (Note 1). One container is needed for each water content determination.

NOTE 1—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial weighing and to prevent absorption of moisture from the atmosphere following drying and before final weighing.

4.4 *Desiccator*—A desiccator of suitable size (a convenient size is 200 to 250-mm diameter) containing a hydrous silica gel. This equipment is only recommended for use when containers having close-fitting lids are not used. See 7.4.1.

5. Samples

5.1 Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight.

5.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as steel thin-walled tubes, paint cans, etc.) or sample bags are used.

6. Test Specimen

6.1 For water contents being determined in conjunction with another ASTM method, the method of specimen selection specified in that method controls.

6.2 The manner in which the test specimen is selected and its required mass is basically dependent on the purpose (application) of the test, type of material being tested, and the type of sample (specimen from another test, bag, tube, split-barrel, etc.). In all cases, however, a representative portion of the total sample shall be selected. If a layered soil or more than one soil type is encountered, select an average portion or individual portions or both, and note which portion(s) was tested in the report of the results.

6.2.1 For bulk samples, select the test specimen from the material after it has been thoroughly mixed. The mass of moist material selected shall be in accordance with the following table:

Sieve Retaining More Than About 10 % of Sample	Recommended Minimum Mass of Moist Specimen, g
2.0 mm (No. 10) sieve	100 to 200
4.75 mm (No. 4) sieve	300 to 500
19 mm	500 to 1000
38 mm	1500 to 3000
76 mm	5000 to 10 000

6.2.2 For small (jar) samples, select a representative portion in accordance with the following procedure:

6.2.2.1 For cohesionless soils, thoroughly mix the material, then select a test specimen having a mass of moist material in accordance with the table in 6.2.1. See Note 2.

6.2.2.2 For cohesive soils, remove about 3 mm of material from the exposed periphery of the sample and slice it in half (to check if the material is layered) prior to selecting the test specimen. If the soil is layered see 6.2. The mass of moist material selected should not be less than 25 g or should be in accordance with the table in 6.2.1 if coarse-grained particles are noted. (Note 2).

6.3 Using a test specimen smaller than the minimum mass indicated previously requires discretion, though it may be adequate for the purpose of the test. A specimen having a mass less than the previously indicated value shall be noted in the report of the results.

NOTE 2—In many cases, when working with a small sample containing a relatively large coarse-grained particle, it is appropriate not to include this particle in the test specimen. If this occurs, it should be noted in the report of the results.

7. Procedure

7.1 Select representative test specimens in accordance with Section 6.

7.2 Place the moist specimen in a clean, dry container of known mass (Note 3), set the lid securely in position, and determine the mass of the container and moist material using an appropriate balance (4.2). Record these values.

7.3 Remove the lid and place the container with moist material in a drying oven maintained at 110 ± 5°C and dry to a constant mass (Notes 4, 5, and 6).

NOTE 3—To assist in the oven-drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

NOTE 4–The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used. In most cases, drying a test specimen over night (about 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the mass after two successive periods (greater than ½ h) of drying indicate an insignificant change (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

NOTE 5—Oven-drying at 110 ± 5°C does not always result in water content values related to the intended use or the basic definition especially for materials containing gypsum or other minerals having significant amounts of hydrated water or for soil containing a significant amount of organic material. In many cases, and depending on the intended use for these types of materials, it might be more applicable to maintain the drying oven at 60 ± 5°C or use a vacuum desiccator at a vacuum of approximately 133 Pa (10 mm Hg) and at a temperature ranging between 23 and 60°C for drying. If either of these drying methods are used, it should be noted in the report of the results.

NOTE 6—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the oven. However, this requirement is not applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

7.4 After the material has dried to constant mass remove the container from the oven and replace the lid. Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents. Determine the mass of the container and oven-dried material using the same balance as used in 7.2. Record this value.

7.4.1 If the container does not have a lid, weigh the container and material right after their temperatures are such that the operation of the balance will not be affected by convection currents or after cooling in a desiccator.

NOTE 7—Cooling in a desiccator is recommended since it prevents absorption of moisture from the atmosphere during cooling.

8. Calculation

8.1 Calculate the water content of the material as follows:

$$w = [(W_1 - W_2)/(W_2 - W_c)] \times 100 = \frac{W_w}{W_s} \times 100$$

where:

w = water content, %,
W_1 = mass of container and moist specimen, g,
W_2 = mass of container and oven-dried specimen, g,
W_c = mass of container, g,
W_w = mass of water, g, and
W_s = mass of solid particles, g.

9. Report

9.1 The report (data sheet) shall include the following:

9.1.1 Identification of the sample (material) being tested, by boring number, sample number, test number, etc.

9.1.2 Water content of the specimen to the nearest 0.1 % or 1 %, depending on the purpose of the test.

9.1.3 Indication of test specimen having a mass less than the minimum indicated in Section 6.

9.1.4 Indication of test specimen containing more than one soil type (layered, etc).

9.1.5 Indication of the method of drying if different from oven-drying at 110 ± 5°C.

9.1.6 Indication of any material (size and amount) excluded from the test specimen.

10. Precision and Accuracy

10.1 Requirements for the precision and accuracy of this test method have not yet been developed.

U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Fels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Analyze pure product liquid samples by gas chromatography-flame ionization and flame photometric detection followed by comparison with analyzed naphtha reference compounds (ASTM Method D3328-78).

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 concentration):

Analyze 5 pure product liquid samples by gas chromatography-flame ionization and flame photometric detection followed by comparison with analyzed reference compounds (ASTM Method D3328-78).

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: Daily by Overnight Carrier

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

Laboratory will report results within 30 days of sample receipt.

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

ASTM Method D3328-78: Comparison of Waterborne Petroleum Oils By Gas Chromatography; from ASTM D3328-78, use Method B for capillary GC columns.

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

Please refer to the attached ASTM Method D3328-78:Method B-capillary GC columns. This is a qualitative method whereby identification (if possible) of the pure product liquid sample is determined by comparison with known oils or reference standards, selected because of their possible relationship to the pure product. The pure product liquid will be collected from suspected sources (5 samples) while the reference samples must be purchased and also submitted for the GC analysis. The known oils or reference standards shall be the following: VM & P naphtha, Stoddard solvent, and mineral spirits. These standards will be provided by the laboratory.

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.

Laboratory deliverables will include all raw data and reports (GC chromatograms, GC tuning data, QA/QC data, calculations, and all information generated by the method).

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

11. Name of sampling/shipping contact: Dave Shekoski
Phone: (414) 272-2426

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.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Analysis of Pure Product Liquid Samples (5) and Analysis of the Reference Standards: VM & P naphtha, Stoddard solvent, and Mineral spirits	Attached ASTM Method D3328-78	

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Blank	One per 10 samples	<+/- D.L.
Duplicate	One per 10 samples	<20% RPD
Lab Control Std.	One per 10 samples	90-110 %R

Please see attached ASTM Method D3328-78: Method B

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

***Action Required if Limits are Exceeded:**

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Designation: D 3328 – 78 (Reapproved 1982)[ε2]

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Methods for COMPARISON OF WATERBORNE PETROLEUM OILS BY GAS CHROMATOGRAPHY[1]

This standard is issued under the fixed designation D 3328; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[ε1] NOTE—Editorial changes were made in Sections 2 and 4 and footnotes were renumbered in January 1983.
[ε2] NOTE—Editorial changes were made throughout this standard in December 1986.

1. Scope

1.1 These methods cover the comparison of petroleum oils recovered from water or beaches with oils from suspect sources by means of gas chromatography **(1, 2, 3)**.[2] Such oils include distillate fuel, lubricating oil, and crude oil. Methods are described for packed and capillary column analyses using either single detection (flame ionization) or dual detection (flame ionization and flame photometric for sulfur).

	Sections
Method A—Packed Column	9 to 18
Method B—Capillary Column	19 to 27

1.2 Method A provides a low-resolution separation; Method B provides a higher resolution for more critical examination. The dual detection scheme should be employed whenever possible. The flame-photometric detection for sulfur components is an adjunct, not a substitute, for flame-ionization detection in the identification of waterborne petroleum oils (4 to 12). There are, however, certain circumstances where the sulfur chromatograms can distinguish two oils when the flame ionization chromatograms cannot.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Definitions of Terms Relating to Water[3]
D 1193 Specification for Reagent Water[3]
D 2549 Method for Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography[4]
D 3415 Practice for Identification of Waterborne Oils[5]
D 3325 Practice for Preservation of Waterborne Oil Samples[5]
D 3326 Practices for Preparation of Samples for Identification of Waterborne Oils[5]
D 3327 Method for Analysis of Selected Elements in Waterborne Oils[5]
E 260 Practice for Packed Column Gas Chromatography[6]
E 355 Practice for Gas Chromatography Terms and Relationships[6]

3. Definitions

3.1 For definitions of terms used in these methods, refer to Practice D 3415, Definitions D 1129, and Practice E 355.

4. Significance and Use

4.1 Identification of a recovered oil is determined by comparison with known oils, selected

[1] These methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.31 on Identification of Waterborne Oils.
Current edition approved Jan. 27, 1978. Published April 1978. Originally published as D 3328 – 74a T. Last previous edition D 3328 – 74a T.
[2] The boldface numbers in parentheses refer to the references at the end of these methods.
[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] *Annual Book of ASTM Standards*, Vol 05.02.
[5] *Annual Book of ASTM Standards*, Vol 11.02.
[6] *Annual Book of ASTM Standards*, Vol 14.01.

because of their possible relationship to the particular recovered oil. The known oils are collected from suspected sources. Samples of such known oils *must* be collected and submitted along with the unknown for analysis. At present, identification of the source of an unknown oil by itself cannot be made (for example, from a library of known oils).

4.2 The use of a flame-photometric detector in addition to the flame-ionization detector provides a second, independent profile of the same oil, that is, significantly more information is available from a single analysis with dual detection.

4.3 Many close similarities (within uncertainties of sampling and analysis) will be needed to establish identity beyond a reasonable doubt. The analyses described will distinguish many, but not all samples. For cases in which this method does not clearly identify a pair of samples, and for important cases where additional comparisons are needed to strengthen conclusions, other analyses will be required, such as Method B, and other appropriate methods (Practice D 3415).

4.4 For Method B, the "deasphalted" fraction of neat petroleum or petroleum residue is prepared to provide a sample free of asphaltenes in order to protect the capillary column.

5. Interferences

5.1 Compounds that have the same retention time as petroleum hydrocarbons will interfere in the comparison of the unknown with known oils. This is particularly true if animal fat or vegetable oil, naturally occurring hydrocarbons, or spill-treatment chemicals are present in relatively large amounts. Independent analysis, for example, infrared spectroscopy, will establish the presence of these contaminants if their presence is suspected. Animal or vegetable oils can be removed effectively by Method D 2549 or by Practices D 3326 (Method D).

NOTE 1—Method D 2549 will also remove the aromatic fraction.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society.[6]

6.2 Unless otherwise indicated references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

6.3 *Air*—For use with the flame-ionization and flame-photometric detectors; may be obtained using a laboratory pure air generator, or from a zero grade tank supply.

6.4 *Carrier Gas*—High-purity grade helium is used as carrier gas.

6.5 *Cyclohexane*—For use in reference standards.

6.6 *Hydrogen*—For use with the flame-ionization and flame-photometric detectors; may be obtained using a hydrogen generator, or from a prepurified grade tank supply.

6.7 *Methylene Chloride*—For use in reference standards and glassware cleaning.

6.8 *Normal Alkane Standards*—Normal alkanes, decane through hexatriacontane, for use as reference compounds.

6.9 *Normal Pentane*—Chromato-quality, normal pentane is used for sample deasphalting.

6.10 *Thiophene*—For use in optimization of flame-photometric detector.

7. Reference Standards

7.1 *Normal Paraffinic Hydrocarbons*—Prepared mixtures of approximately decane to hexatriacontane, or selected individual normal paraffins, are run under normal analysis conditions to determine retention times of compounds.

7.2 *Resolution Mixture*—Equal mixtures of *n*-hexadecane, *n*-octadecane and eicosane in cyclohexane solution (100 µl of each diluted to 10 mL with cyclohexane). See the annex for details (see A1.2.1).

8. Sampling

8.1 Collect a representative sample. The method used depends upon the quantity of sample available. If only a thin sheen is pres-

[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

ent on the water, the oil can be picked up by dipping TFE-fluorocarbon strips (50 by 75 by 0.25 mm). The adhering properties of the TFE-fluorocarbons can be enhanced by roughing (etching) the surface, or by perforating with 1.6-mm holes (5/cm²). The TFE-fluorocarbon strips are placed in a solvent-rinsed glass jar and sealed with TFE-fluorocarbon or aluminum-lined cap to avoid plasticizer contamination.

8.2 If the sample is not to be analyzed within 1 week, it should be preserved in accordance with Method D 3325 because of the possibility of bacterial decomposition of normal paraffins in the sample.

8.3 The sample should be prepared for analysis in accordance with Practices D 3326, because of the great variety of materials and circumstances associated with collecting petroleum oils from the environment. This preparation procedure removes water, particulate matter and asphaltenes (high molecular weight components that would build up on the column and effectively shorten its useful life).

METHOD A – PACKED COLUMN PROCEDURE

9. Scope

9.1 This method is applicable to samples of neat petroleum, or to prepared petroleum residue.

10. Summary of Method

10.1 This method uses a gas chromatographic packed column system for the separation of petroleum hydrocarbons. The effluent of the column may be detected with a flame-ionization detector, or it may be split (1+2) between a flame ionization and a flame-photometric detector. The flame photometric detector is equipped with a narrow bandpass interference filter for spectral isolation of the sulfur emission at 394 nm. The relative peak size of each component (as indicated by retention time) of recovered oil is compared visually with the relative peak size of each component (of like retention time) of the suspected source. A discussion of gas chromatography is presented in Practice E 260.

NOTE 2—This duel detector method is based on the early work done by Kahn **(13)**, Garza **(4)**, and Adlard **(7)**. Kahn and Garza currently use a method that does not employ an effluent split for simultaneous dual detection.

10.2 In this method, elution of characteristic hydrocarbons occurs generally in order of increasing boiling point.

11. Apparatus

11.1 *Chromatographic Column*—Columns may be purchased or prepared by the analyst.

11.1.1 *OV-101*[7]—A ⅛-in. by 10-ft (3-mm by 3-m) stainless steel column with 0.012-in. (0.3-mm) wall, packed with 60/80 mesh Chromosorb W[8] (acid washed and dimethylchlorosilane (AW-DMCS)-treated) coated with 10 weight % of OV-101.

11.2 *Gas Chromatograph*—A commercial or custom designed gas chromatograph with heated injection and detector zones and a column oven capable of being programmed from 75°C to at least 325°C for heavier oils (higher boiling than gasolines, jet fuels, etc.).

11.2.1 For light distillate fuels, the chromatograph must be capable of programming from 50°C and also be capable of maintaining isothermal control at 50°C.

11.2.2 *Injection Port*—The use of glass injector inserts that can be replaced or cleaned frequently, or both, will prolong the useful life of the column **(3)**.

11.2.3 *Detectors*—A hydrogen-flame ionization detector is always used for Method A, in addition a flame-photometric detector with a 394 nm bandpass filter is used for dual detection **(9, 10, 11, 12)**.[9, 10]

11.2.4 *Effluent Splitter*—An effluent splitter with a split ratio of 1+2 (FID/FPD) is required for dual detection.

11.2.5 *Bleeder for Reference Compound*—A device for in-line bleed of a reference compound (thiophene and cyclohexane) into the carrier flow for detector optimization is

[8] A registered trademark of Ohio Valley Specialty Chemical Co.

[9] A registered trademark of Johns-Manville Products Corp.

[10] The flame-photometric detector nearly universally used is that developed by Melpar and marketed under license by MicroTex Instrument Corp. of Austin, TX.

[11] Flame-photometric detector with fiber optics were developed by both Bendix and Perkin-Elmer. They are currently available from Perkin-Elmer, Norwalk, CT. Fiber optics permit removal of the photomultiplier tube from the heated flame zone which in turn permits operating temperatures of up to 450°C (versus the usual 250°C).

required, when using a flame-photometric detector.

11.2.6 *Strip-Chart Recorder*—A strip-chart recorder is required to measure detector response at full-scale range of 1 mV with a response time of 1 s (or less). A second recorder, or dual-pen recorder is required for dual detection.

11.3 *Syringe*—A microsyringe of 0.5 to 1 μL capacity.

11.4 *Gas Traps*—Any commerically available gas filter traps to be placed in line to remove trace hydrocarbon and water impurities from the helium, hydrogen, nitrogen, and air gas supplies.

11.5 *FPD Linearizer*—Optional accessory to facilitate comparison of FPD chromatograms.

12. Preparation of Chromatograph

12.1 Install the column in the chromatograph.

12.2 Shut off the downstream end of the system and pressurize the carrier gas supply to a gage pressure of approximately 15 psi (103 kPa) above the operating pressure. Shut off the cylinder valve and observe the pressure gage. Consider the system tight if no pressure drop is noted in 10 to 15 min. Use a small amount of aqueous soap solution to locate minor leaks. Do not use the soap solution near the ionization detector.

12.3 *Column Conditioning for New Columns:*

NOTE 3—For previously conditioned columns, proceed to 12.3.9.

12.3.1 Disconnect the column at the detector end to avoid deposition of volatiles on the detector(s) during conditioning.

12.3.2 For freshly prepared columns, pass helium through and cap the column with a brass or stainless steel plug. Program the oven to 325°C and hold for 4 h. Cool and remove the plug. For older columns, proceed directly with Step 12.3.9.

12.3.3 Adjust the carrier gas flow as indicated in Table 1.

12.3.4 Raise the column temperature to 275°C and hold at this temperature for 1 h with normal carrier flow rate.

12.3.5 Increase the column temperature to 300°C and hold at this temperature for at least 1 h with normal carrier flow.

12.3.6 Increase the column temperature to 325°C and hold overnight with the normal carrier flow rate.

12.3.7 Heat to 350°C and hold for ½ h.

12.3.8 After conditioning, cool the column and connect it to the detector(s).

12.3.9 Adjust the hydrogen and air flow, and the air/hydrogen flow ratio to the detector(s), as specified for the instrument being used. Ignite the flame(s) (see 12.4 for optimization).

12.3.10 Adjust the carrier gas flow as indicated in Table 1.

12.3.11 Program the column temperature as indicated in Table 1, and hold at the maximum temperature while monitoring the effluent. If there are no peaks in the chromatogram and there is minimal baseline shift at high temperatures, then the column is ready for use; otherwise recondition it.

12.3.12 Return the oven temperature to 75°C.

12.3.13 If the column is to be moved or stored, disconnect and seal the ends of the column. When the column is to be reused, even after conditioning, it is always necessary to cycle through the temperature program to remove any accumulated volatiles.

12.4 *Optimization of Detectors*—Adjust hydrogen and air flows to give optimal detector responses for a given background sample signal-provided by the reference compound bleeder (11.2.5). Use cyclohexane for FID optimization and thiophene for the FDP optimization.

13. Operating Conditions for Analysis (Notes 4, 5, 6)

NOTE 4—One of the problems frequently encountered with the flame photometric detector is "flameout" when large amounts of solvent are injected with the sample. The recommended sample preparation procedure avoids this problem at the same time that it permits the use of small samples. For those who may encounter this problem, a simple modification has been suggested **(8)** which consists of reversing the hydrogen gas and air/oxygen gas inlets to the detector.

NOTE 5—For oil identification under the recommended procedure, air has been found satisfactory for combustion for the FPD, that is, oxygen is not necessary.

NOTE 6—See the manufacturer's manual for maintenance information for the FPD. Present flame photometric units should not be heated above

250°C, unless the photometer is removed from the heated zone by fiber optics; older units cannot be heated above 170°C. Periodically, it may be necessary to remove the flame jet and clean it with solvent (cyclohexane) in an ultrasonic bath.

13.1 Operating conditions are summarized in Table 1; apparatus operated under these conditions should achieve partial resolution of two pairs of normal and isoprenoid hydrocarbons found in many, but not all, crude oils and certain petroleum products. In order of emergence from the column, these are heptadecane and pristane, and octadecane and phytane.

13.1.1 Each day, analyze the resolution mixture to test the column performance, monitor the instrument performance and thermally equilibrate the system the (see the Annex for details).

13.1.2 Apply annex procedure to ensure that column performance is acceptable. Repeated injection of samples containing asphaltenes will change the resolving power of the column until the column eventually will degrade to the point where its performance is no longer acceptable.

14. Component Identification

14.1 In most instances, it is unnecessary to identify individual components when comparing chromatograms of a spill with its source; it is sufficient to note their degree of match. Identification of the usually dominant normal paraffin hydrocarbons is readily achieved by comparing their retention times with those from known *n*-alkane standards.

14.1.1 Identification of peaks other than normal paraffins is not achieved, except in rare cases.

14.1.2 Comparison of peaks with the same retention times in the known and unknown oils is also made with respect to relative peak sizes of adjacent peaks.

14.2 To determine the retention time of normal paraffins, the following procedure is recommended:

14.2.1 With the column at the initial operating temperature, inject 0.2 µL of the known mixture of normal paraffins (11.1).

14.2.2 Turn on the recorder and mark the injection point on the recorder chart.

14.2.3 Adjust the instrument attenuation so that the maximum peak heights are on scale.

14.2.4 When the temperature program is complete and the baseline has stabilized, cool the oven to the initial temperature.

14.2.5 Measure the retention time in minutes to at least two significant figures for each normal paraffin in the known mixture.

15. Procedure for a Sample

15.1 First, cycle the instrument through its program to test the column and instrument performance (13.1) and thermally equilibrate the oven.

15.2 Zero the strip-chart recorder pen and make appropriate notations at the beginning of the chromatogram (sample name, reference number, date, amplifier attenuations).

15.3 For light distillate oils (such as gasoline, jet fuels, kerosines, and No. 2 fuel oils), inject 0.2 µL of sample directly into the injection port with the column at initial operating temperature. For heavier oils, deasphalt with 15 parts of pentane, before injecting the 0.2-µL sample (after pentane removal).

15.4 Start the recorder and the temperature program. Mark the injection point on the recorder chart.

15.5 Adjust the attenuations so that the highest peak is retained on scale and constant baselines are achieved after the analysis. Obtain a complete chromatogram at a single attenuation, repeating if necessary until a satisfactory chromatogram is obtained.

15.6 When the temperature program is complete and the baseline has stabilized, cool the oven to the initial temperature. (This is automatic for most instruments.) After resetting the initial conditions, another sample may be analyzed.

15.7 After completion of the analysis, record the following information for each set of chromatograms: column length and diameter; liquid phase and weight percent; support material and mesh size; initial and final column temperatures; programming rate; carrier gas flow rate; detector manifold and injection port temperatures; FPD heater temperature (if used); hydrogen and air flow rates; injection port split ratio and effluent split ratio (if employed); sample size and amplifier ranges. (Rubber stamps are commercially available which facilitate the recording of these data.)

15.8 Prepare chromatograms of samples of known origin, that is, from potential sources

(see 15.1 to 15.7).

16. Interpretation

16.1 *Basis of Matching*—The matching of oil samples is essentially a profiling technique based on the premise that identical oils give identical chromatograms. Normally, the matching of a spilled oil to a suspect oil can be accomplished by comparison of the chromatograms for each of the oils in a spill case.

16.2 *Chromatogram Features*—The major features of a chromatogram used for comparison are listed as follows and are illustrated in Figs. 1 and 2 (gas chromatograms of a Kuwait crude oil which depict FID as well as FPD curves).

16.2.1 The FID curve shows a typical separation with the features of a homologous series of normal paraffins, the isoprenoid hydrocarbons pristane and phytane, the unresolved envelope and other resolved peaks. All of these features are used to characterize an oil.

16.2.2 The FPD curve has fewer readily ascribed characteristics; rather it gives the overall sulfur profile generated by the detector. It is useful not only qualitatively, but semiquantitatively.

16.3 *Weathering Effects:*

16.3.1 When an oil is spilled on open water, or a relatively small amount of oil is widely dispersed in an area such as a bilge tank, weathering will progress rapidly. A thin slick on open water may lose significant amounts of its components up to *n*-C_{15} (271°C atmospheric boiling point) within 48 h of being spilled. It is important to be cognizant of the effects of weathering when analyzing spill samples more than a few hours old. It is advisable to compare only those portions of chromatograms boiling above pentadecane in order to minimize the difference resulting from changes due to weathering.

16.3.2 Light distillate fuels cannot survive heavy weathering and have few hydrocarbons above C_{15}. Comparison of the residues of these oils can only be done qualitatively—from about C_8–C_{15}.

16.4 *Comparison of Chromatograms:*

16.4.1 Normally a direct comparison of chromatograms, considering the features enumerated above, will suffice for establishing identity or nonidentity between samples. The comparison involves simply a peak-for-peak matching, noting differences or similarities in relative peak size. If the chromatograms are the same on the basis of peak-for-peak matching, there is a high degree of probability that the samples are from the same source. A mismatch is obtained when the curves are different. The differences may be due to the presence of one or more components in one sample relative to another or consistent differences in relative intensities of peak responses, or both. Spill samples may contain components such as bilge cleaning detergents, plasticizers, paint vehicles, etc. The presence of one or two components in a spill sample, which are absent in a suspect, is not intrinsically indicative of nonidentity.

17. Report

17.1 Based upon the visual comparison of chromatograms, and after considering 8.2, 16.3, and 1.64, report the sample of unknown origin as belonging to one of the categories below:

17.1.1 *Match*—Like one, or more, of the samples submitted for comparison.

17.1.2 *Probable Match*—Like one, or more, of the samples submitted for comparison, except: (*a*) for changes which could be attributed to weathering (specific low molecular weight peak losses), or (*b*) differences attributable to specific contamination.

17.1.3 *Indeterminate*—Like one, or more, of the samples submitted for comparison, except for certain differences as in 17.1.2 of such magnitude that it is impossible to ascertain whether the unknown is the same oil heavily weathered, or a totally different oil.

17.1.4 *Mismatch*—Unlike the samples submitted for comparison.

18. Precision and Bias

18.1 No statement is made about either the precision or bias of Method A since the result merely states whether there is conformance to the criteria for success specified in the procedure.

METHOD B—CAPILLARY COLUMN PROCEDURE

19. Scope

19.1 See Section 9, but using a capillary column procedure.

20. Summary of Method

20.1 This method makes use of a gas chromatographic capillary column system for separation of petroleum hydrocarbons and either FID or FID and FPD for their measurement. The relative peak size of each component (as indicated by retention time) from a spill sample is compared with the relative peak size of each component (of like retention time) of a similarly prepared sample from the suspected source. Thus, Method B is basically the same as Method A except for the use of a higher resolution column.

21. Apparatus

21.1 *Chromatographic Column* (see 11.1):

21.1.1 *OV-101*[9]—A 0.02-in. by 50-ft (0.5-mm by 16-m) support coated open tubular (SCOT) stainless steel column.

21.2 *Gas Chromatograph* (see 11.2)—The same gas chromatograph used in Method A may be used in Method B provided it has the necessary fittings to accomodate the capillary column and:

21.2.1 *Carrier Gas Pressure Regulator* is substituted pressure regulator for the mass flow controllers to give more precise flow rates in the low flow ranges (1 to 5 mL/mm).

21.2.2 *Effluent Splitter* is required for the column effluent, with a split ratio of 1 + 2 FID/FPD.

21.2.3 *Carrier Gas Makeup* is required at the effluent end of the column with a temperature-independent mass flow controller.

21.3 *Detectors*—A hydrogen flame-ionization detector and for dual detection a flame-photometric detector (see 11.2.3).

21.4 *Strip-Chart Recorder* (see 11.2.6).

21.5 *Microsyringe* (see 11.3).

21.6 *Bleeder for Reference Compound* (see 11.2.5).

22. Test Sample (Spilled oil)

22.1 Using 1 to 2 mL of neat petroleum, prepare a sample in accordance with Practices D 3326.

23. Preparation of Chromatograph

23.1 See Section 12.

24. Operating Conditions for Analysis

23.1 Operating conditions are summarized in Table 1.

NOTE 7—See A1.3.1 for the results expected on a new, properly conditioned SCOT column. A properly functioning column should provide 400 to 600 analyses, depending on the types of oil analyzed.

25. Method of Comparison

25.1 As instructed in Sections 14 and 16.

26. Procedure for a Sample

26.1 With the column at initial operating temperature, inject a 0.2-μL sample into the injection port.

26.2 See 15.4.

26.3 See 15.5.

26.4 See 15.6.

26.5 See 15.7.

26.6 Prepare chromatograms from samples of known origin in the manner described in 15.4 to 15.7.

27. Report

27.1 Based upon the visual comparison of chromatograms and after considering 3.2 and Section 16, report the sample of unknown origin as belonging to one of the categories below:

27.1.1 *Match* (see 17.1.1).

27.1.2 *Probable Match* (see 17.1.2).

27.1.3 *Indeterminate* (see 17.1.3).

27.1.4 *Mismatch* (see 17.1.4).

28. Precision and Bias

28.1 No statement is made about either the precision or bias of Method B since the result merely states whether there is conformance to the criteria for success specified in the procedure.

TABLE 1 Operating Conditions for Chromatographic Columns (11, 12, 13)

	Method A	Method B
Column	⅛-in. by 10-ft (3-mm by 3-m) stainless steel (see 11.1.1)	0.02-in. by 50-ft (0.5-mm by 16-m) stainless steel (see 21.1.1)
Packing	10 % OV-101[7], 60/80 mesh Chromosorb W[9] (AW-DMCS)	OV-101 SCOT
Carrier gas:	helium	helium
Flow, mL/min:		
Column	approximately 30	approximately 3
Makeup gas	none	approximately 30
Temperature, °C:		
Injection port	300	250
Column:		
Heavier oils:		
Initial	75	75
Final	325 (FID) 250 (FID/FPD)	250 (FID) 250 (FID/FPD)
Lighter oils:		
Initial	50 hold 2 min	50 hold 2 min
Final	250	250
Detector	350 (FID) 250 (FID/FPD)	275 (FID) 250 (FID/FPD)
Program Rate	8–10[4]	6–8[4]
Chart speed, in/min (mm/min)	2.5 (10)	2.5 (10)
Sensitivity, mV	1	1
Sample size, µL	0.2	0.2
Effluent split ratio (FPD procedures)	1+2 (FID/FPD)	1+2 (FID/FPD)

[4] The precise rate is dictated by the design of the gas chromatograph.

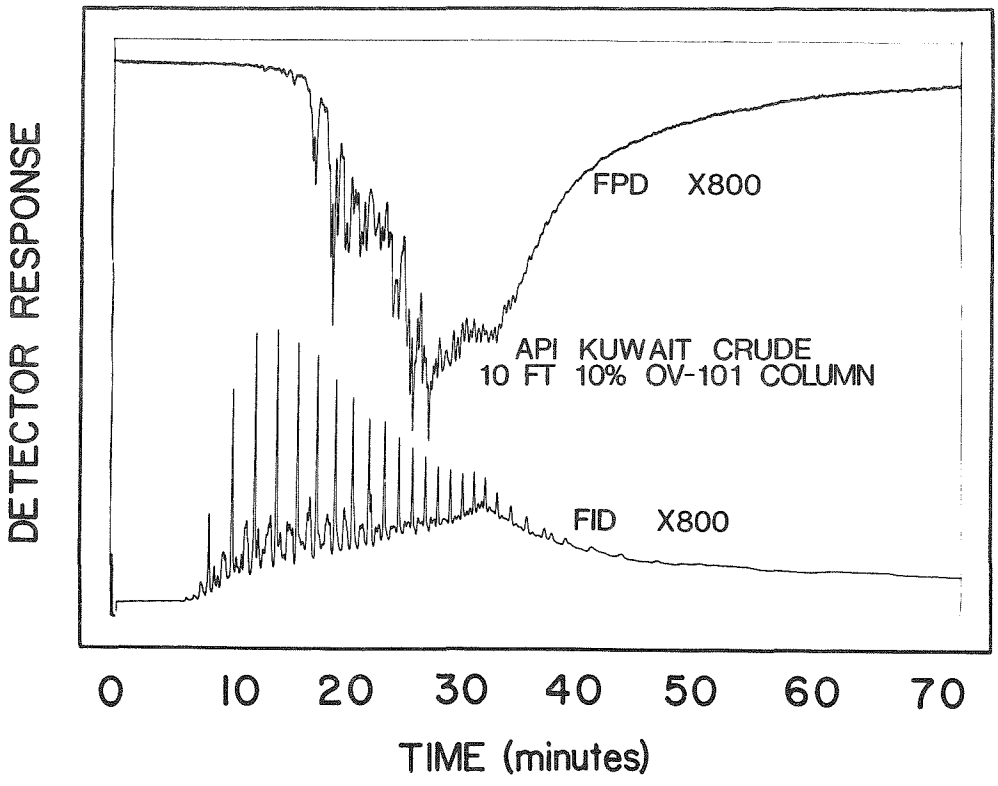

FIG. 1 Representative Chromatograms from Packed Column (Methods A1 and A2)

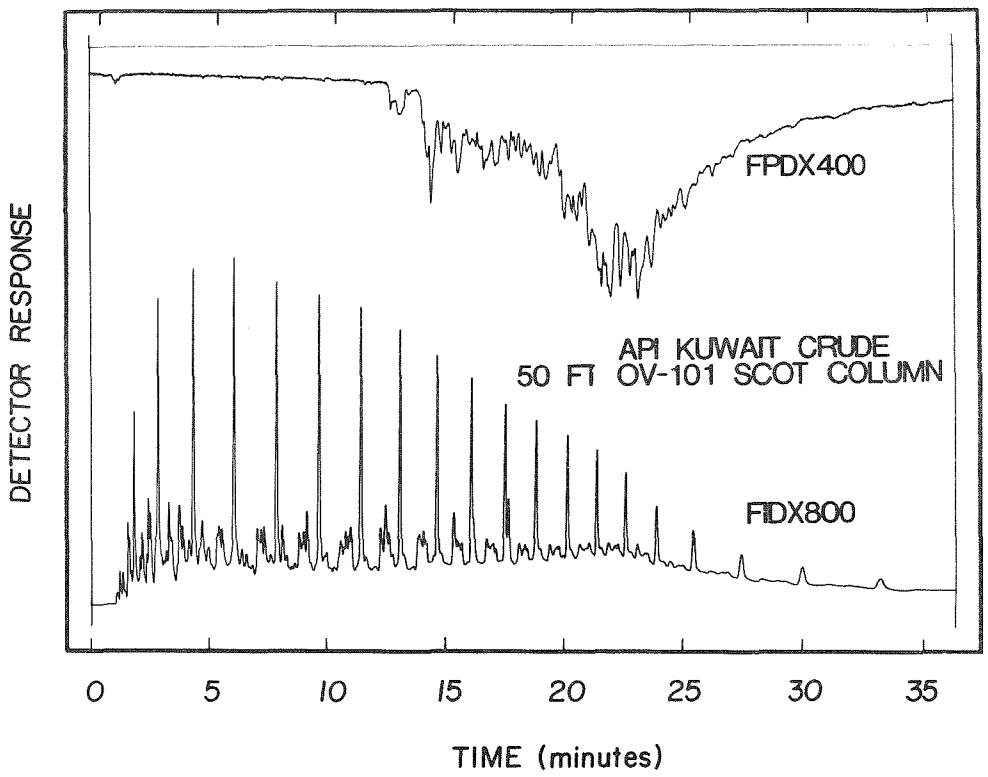

FIG. 2 Representative Chromatograms from Capillary Column (Methods B1 and B2)

ANNEX

(Mandatory Information)

A1. COLUMNS

A1.1 Column Performance

A1.1.1 The level of performance of the chromatographic system, in particular the gas chromatographic column, can be quantitated by calculation of the resolution of specific compounds. The term "resolution" is defined in Recommended Practice E 355. The resolution values for normal alkanes are used in defining column performance for this recommended practice.

A1.2 Procedure

A1.2.1 A resolution mixture is prepared consisting of 100 µL each of normal alkanes *n*-C16, *n*-C18, and *n*-C20; the alkanes are dissolved in cyclohexane to 10-ml volume. Gentle warming of the solutes may be necessary to carry out their transfer. The solution is prepared in a 10-mL volumetric flask.

A1.2.2 Instrumental conditions, gas flows, and temperature programming are exactly the same as for the analysis of samples (see Section 13). A 0.2-µL injection volume is used for analysis and will give a 35 to 60 % full-scale recorder response at normal amplifier settings used for oil samples. The resultant peaks are of the approximate size of the same peaks that will be found for many oil samples. Examples of the resolution mixture for packed and SCOT column operation are shown in Fig. A1.1 and A1.2, respectively.

A1.2.3 The resolution for the peak pairs *n*-C16 and *n*-C18 and for *n*-C18 and *n*-C20 are determined using Practice E 355.

NOTE A1 – A faster chart speed of 100 to 200 mm/min will improve the measurement of peak width. The measurement of peak width at half height may be necessary when peak tailing occurs; this measurement should be doubled for use in resolution equations.

A1.3 Performance Standards

A1.3.1 The resolution of components for a well performing column will give resolution values for

packed columns of Sections 13 and 11, and for SCOT columns values of 23 and 20 for the peak pairs *n*-C16 and *n*-C18 and for *n*-C18 and *n*-C20.

A1.3.2 Columns should be replaced and operating conditions thoroughly checked should resolution values approach 75 % of the values in A1.3.1.

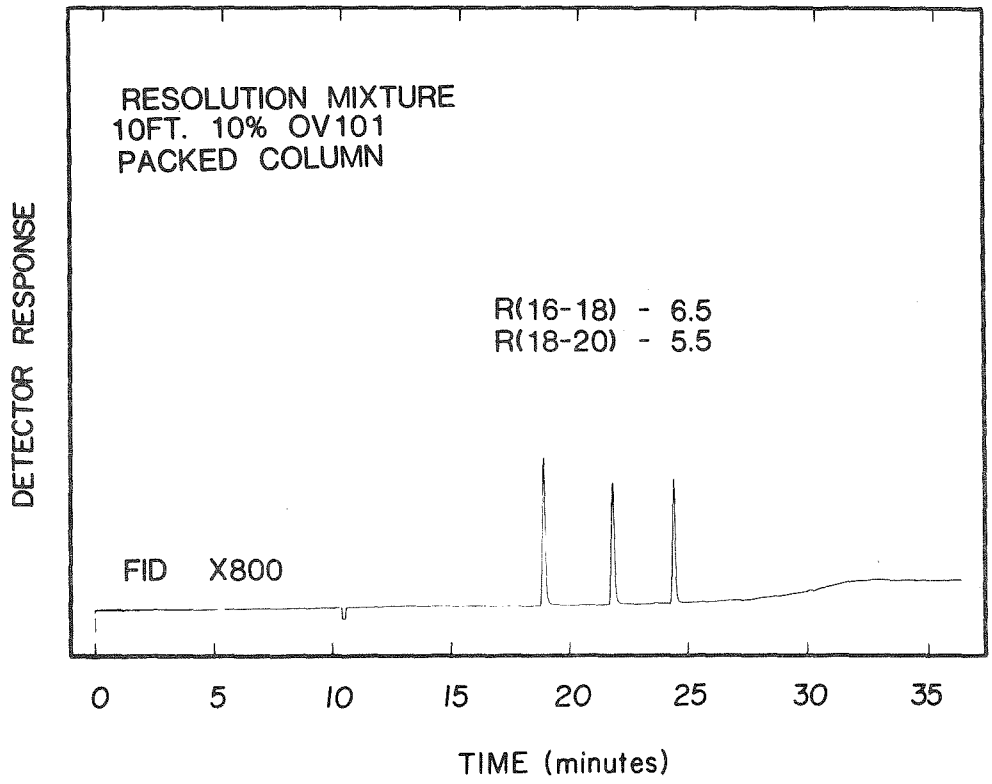

FIG. A1.1 Resolution on Packed Column

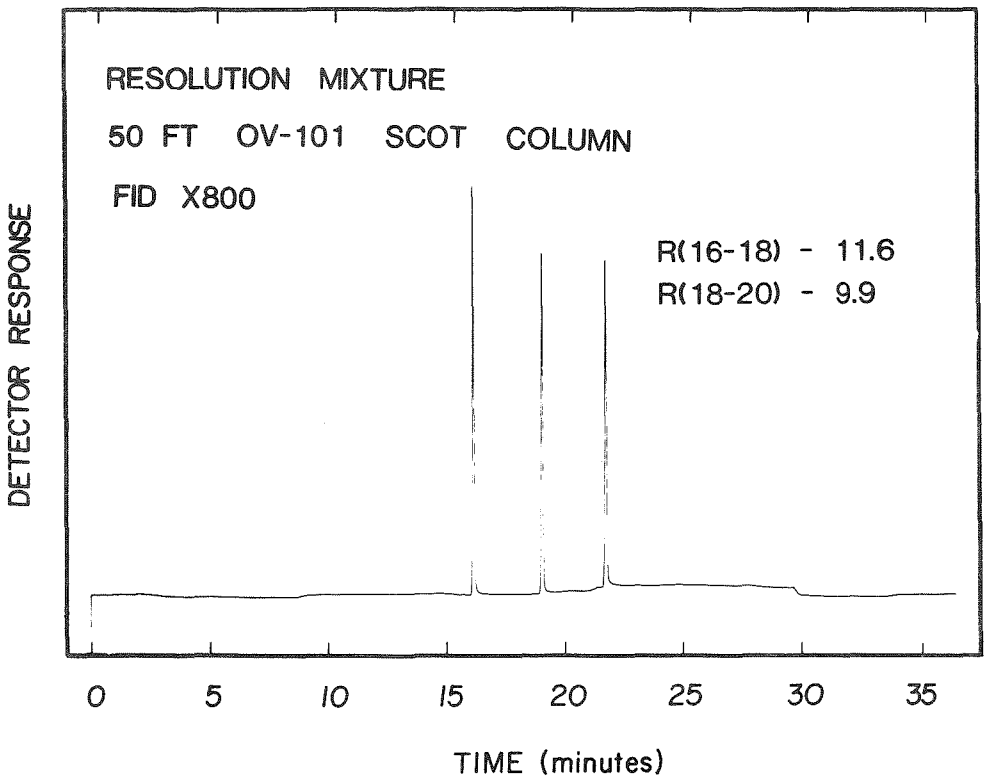

FIG. A1.2 Resolution on Capillary Column

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U.S. Environmental Protection Agency
HWI 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 Municipal Landfill

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite 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:

Analyze soil samples for sulfur content.

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 concentration):

Analyze 11 soil samples for percent sulfur.

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: Daily by Overnight Carrier

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Percent Sulfur	<1%	

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Blank	1 per 10 Samples	< D.L.
Duplicate	1 per 10 Samples	R % D < 10%
Reference Material (from EPA)	1 per 10 Sample	R % D < 10% % R 80-120% See Tables 2&3 from ASTM D3176

III. *Action Required if Limits are Exceeded:

Contact Jan Pels at EPA Region V (Phone (312) 353-2720)

Designation: D 3177 – 84

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103

Standard Test Methods for TOTAL SULFUR IN THE ANALYSIS SAMPLE OF COAL AND COKE[1]

This standard is issued under the fixed designation D 3177; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover two alternative procedures for the determination of total sulfur in samples of coal and coke. Sulfur is included in the ultimate analysis of coal and eoke.

1.2 The procedures appear in the following order.

	Sections
Method A—Eschka Method	6 to 9
Method B—Bomb Washing Method	10 to 11

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 11.1.1 to 11.1.1.7.

2. Applicable Documents

2.1 *ASTM Standards:*

D 346 Method of Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 1193 Specification for Reagent Water[3]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 2015 Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal[2]

D 3176 Methods for Ultimate Analysis of Coal and Coke[2]

D 3180 Methods for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

D 3286 Test Method for Gross Calorific Value of Solid Fuel by the Isothermal-Jacket Bomb Calorimeter[2]

E 144 Recommended Practice for Safe Use of Oxygen Combustion Bombs[4]

3. Summary of Methods

3.1 *Eschka Method*—A weighed sample and Eschka mixture are intimately mixed and ignited together. The sulfur is dissolved in hot water and then precipitated from the resulting solution as barium sulfate ($BaSO_4$). The precipitate is filtered, ashed, and weighed.

3.2 *Bomb Washing Method*—Sulfur is precipitated as $BaSO_4$ from oxygen-bomb calorimeter washings, and the precipitate is filtered, ashed, and weighed.

4. Significance and Use

4.1 Determination of sulfur is, by definition, part of the ultimate analysis of coal.

4.2 Sulfur analysis results obtained by these methods are used to serve a number of interests: evaluation of coal preparation, evaluation of potential sulfur emissions from coal combustion or conversion processes, evaluation of the coal quality in relation to contract specification, and other purposes of commercial or scientific interest.

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[1] These test methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved Jan. 27, 1984. Published March 1984. Originally published as D 3177 – 73. Last previous edition D 3177 – 82.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

5. Sample

5.1 The sample shall be the material pulverized to pass No. 60 (250-μm) sieve in accordance with Method D 2013 or Method D 346.

5.2 A separate portion of the analysis sample should be analyzed for moisture content in accordance with Test Method D 3173, so that calculation to other than the as-determined basis can be made.

5.3 Procedures for converting as-determined sulfur values obtained from the analysis sample to other bases are described in Method D 3176 and Method D 3180.

5.4 Standard Reference Material (SRM), such as SRM Nos. 2862 through 2685—Sulfur in Coal[5] which consist of four different coals that have been individually crushed and ground to pass a 60-mesh sieve, and bottled in 50-g units, or other commercially available reference material coals with a certified sulfur content of ±0.0xx precision can be used. Sulfur values obtained by analyzing these coals, using any of the methods described in this test method, may be used for checking the accuracy of analytical results.

ALTERNATIVE PROCEDURES

METHOD A—ESCHKA METHOD

6. Apparatus

6.1 *Gas* (Note 1) *or Electric Muffle Furnace, or Burners,* for igniting the sample with the Eschka mixture and for igniting the barium sulfate ($BaSO_4$).

NOTE 1—Gas may contain sulfur compounds.

6.2 *Crucibles or Capsules*—Porcelain capsules, 7/8 in. (22 mm) in depth and 1¾ in. (44 mm) in diameter, or porcelain crucibles of 30-mL capacity, high or low form, or platinum crucibles of similar size shall be used for igniting the sample with the Eschka mixture. Porcelain, platinum, alundum, or silica crucibles of 10 to 15-mL capacity, shall be used for igniting the $BaSO_4$.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Available Reagents of the American Chemical Society, where such specifications are available.[6] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type IV, conforming to Specification D 1193.

7.3 *Barium, Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride ($BaCl_2 \cdot 2H_2O$) and dilute to 1 L with water.

7.4 *Eschka Mixture*—Thoroughly mix 2 parts by weight of light calcined magnesium oxide (MgO) with 1 part of anhydrous sodium carbonate (Na_2CO_3). Both materials should be as free as possible from sulfur. Eschka mixture is also available commercially.

7.5 *Hydrochloric Acid* (1 + 1)—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

7.6 *Hydrochloric Acid* (1 + 9)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 9 volumes of water.

7.7 *Methyl Orange Indicator Solution* (0.2 g/L)—Dissolve 0.02 g of methyl orange in 100 mL of hot water and filter.

7.8 *Sodium Carbonate, Saturated Solution*—Dissolve approximately 60 g of crystallized sodium carbonate ($Na_2CO_3 \cdot 10H_2O$) or 22 g of anhydrous sodium carbonate (Na_2CO_3) in 100 mL of water, using a sufficient excess of Na_2CO_3 to ensure a saturated solution.

7.9 *Sodium Hydroxide Solution* (100 g/L)—Dissolve 100 g of sodium hydroxide (NaOH) in 1 L of water. This solution may be used in place of the Na_2CO_3 solution.

8. Procedure

8.1 *Preparation of Sample and Mixture*—Thoroughly mix on glazed paper approximately 1 g of the sample, weighed to nearest 0.1 mg and 3 g of Eschka mixture. The amount of sample to be taken will depend on the amount of $BaCl_2$ solution required in accordance with 8.3. Transfer to a porcelain capsule, or porcelain crucible, or a platinum crucible and cover with about 1 g

[5] Available from the Office of Standard Reference Materials, Room B314, Chemistry Bldg., National Bureau of Standards, Washington, D.C. 20234.

[6] "Reagent Chemicals, American Chemical Society Specification," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."

of Eschka mixture.

8.2 *Ignition*—Heat the crucible over an alcohol, gasoline, or gas flame as described in 8.2.1, or in a gas or electrically heated muffle as described in 8.2.2 for coal and in 8.2.3 for coke. The use of artificial gas for heating the sample and the Eschka mixture is permissible only when the crucibles are heated in a muffle.

8.2.1 *Open Flame*—Heat the crucible, placed in a slanting position on a triangle, over a very low flame to avoid rapid expulsion of the volatile matter that tends to prevent complete absorption of the products of combustion of the sulfur. Heat the crucible slowly for 30 min, gradually increase the temperature, and occasionally stir until all black particles have disappeared, which is an indication of the completeness of the procedure.

8.2.2 *Muffle (Coal)*—Place the crucible in a cold-vented muffle and gradually raise the temperature to 800 ± 25°C in about 1 h. Maintain this maximum temperature until, on stirring, all black particles have disappeared (about 1½ h).

8.2.3 *Muffle (Coke)*—Place the crucible in a warm-vented muffle (about 200°C) and gradually raise the temperature to 800 ± 25°C in about 30 min. Maintain this maximum temperature until, on stirring, all black particles have disappeared.

8.3 *Subsequent Treatment*—Remove the crucible and empty the contents into a 200-mL beaker and digest with 100 mL of hot water for ½ to ¾ h, while stirring occasionally. Decant the solution through filter paper, retaining as much insoluble material in beaker as possible. Thoroughly wash the insoluble matter in the beaker with hot water. After several washings in this manner, transfer the insoluble matter to the filter and wash five times with hot water, keeping the mixture well agitated. Make the filtrate, amounting to about 250 mL, just neutral to methyl orange with NaOH or Na_2CO_3 solution; then add 1 mL of HCL (1 + 9). Boil and add slowly from a pipet, while stirring constantly, 10 mL or more of $BaCl_2$ solution. The $BaCl_2$ solution must be in excess. If more than 10 mL of $BaCl_2$ solution is required, reduce the weight of sample to about 0.5 g and repeat the ignition and digestion. Continue boiling for 15 min and allow to stand for at least 2 h, or preferably overnight, at a temperature just below boiling. Filter through a fine ashless paper, such as Whatman No. 42 or similar, and wash with hot water until 1 drop of silver nitrate ($AgNO_3$) solution produces no more than a slight opalescence when added to 8 to 10 mL of filtrate.

8.3.1 Place the wet filter containing the precipitate of barium sulfate ($BaSO_4$) in a weighed platinum, porcelain, silica, or alundum crucible, fold the paper loosely over the precipitate to allow a free access of air but prevent spattering. Smoke the paper off gradually in a muffle furnace and at no time allow to burn with flame. After the paper is practically consumed, raise the temperature to approximately 800 ± 50°C and heat to constant weight. Weigh the barium sulfate to the nearest 0.1 mg.

8.4 *Blanks and Corrections*—In all cases, a correction must be applied. The preferred method of correction is by the analysis of a weighed portion of a standard sulfate using the prescribed reagents and operations in full compliance with the standard. It is acceptable but less accurate to make corrections by running a reagent blank in duplicate using procedures exactly as described in Section 9 of the standard, using the same amount of all reagents that were employed in the routine determination. If the standard sulfate analysis procedure is carried out once a week, or whenever a new supply of a reagent is used, for a series of solutions covering the approximate range of sulfur concentrations in the samples, it is only necessary to add to or subtract from the weight of $BaSO_4$ determined for the sample, the deficiency or excess found by the appropriate "check" determination. This is more accurate than the simple reagent blank because, for the amounts of sulfur in question and the conditions of precipitation prescribed, the solubility error for $BaSO_4$, is probably the largest one to be considered. Barium sulfate is soluble[7] in acids and pure water, and the solubility limit is reached almost immediately on contact with the solvent. Hence, if very high-purity reagents are used or extra precaution is exercised, there may be no sulfate apparent in the "blank." In other words, the solubility limit for $BaSO_4$ has not been reached or, at any rate, not exceeded; consequently, some sulfate in the sample may remain in solution or redissolve.

9. Calculation

9.1 Calculate the sulfur content as follows:

[7] *Journal of the American Chemical Society*, JACSA, Vol 32, 1910, p. 588; Vol 33, 1911, p. 829.

Sulfur, %, in the analysis sample

$$= \frac{(A - B) \times 13.738}{C}$$

where:

A = grams of $BaSO_4$ precipitated,
B = grams of $BaSO_4$ correction, and
C = grams of sample used.

METHOD B—BOMB WASHING METHOD[8]

10. Reagents

10.1 *Purity of Reagents*—(See 7.1.)

10.2 *Purity of Water*—(See 7.2.)

10.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

10.4 *Hydrochloric Acid* (1 + 1)—(See 7.5)

10.5 *Sodium Carbonate Solution*—Dissolve 20.90 g of anhydrous sodium carbonate (Na_2CO_3) in water and dilute to 1 L. The Na_2CO_3 should be previously dried for 24 h at 105°C.

10.6 *Wash Solution*—Dilute 1 mL of a saturated solution of methyl orange to 1 L with water.

11. Procedure

11.1 *Ignition*—Sulfur is determined in the washings from the oxygen-bomb calorimeter following the calorimetric determination (Test Method D 2015 or D 3286). The type of bomb, amount of water in the bomb, oxygen pressure, and amount of sample taken shall be the same as specified in the calorimetric determination (Test Method D 2015 or D 3286). The bomb shall stand in the calorimeter water for not less than 5 min after firing.

11.1.1 **Caution**—The following precautions are recommended for safe calorimeter operation. Additional precautions are given in Recommended Practice E 144.

11.1.1.1 The weight of coal or coke sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

11.1.1.2 Carefully inspect bomb parts after each use. Frequently check the threads on the main closure for wear. Replace cracked or significantly worn parts. Return the bomb to the manufacturer occasionally for inspection and possibly proof firing.

11.1.1.3 Equip the oxygen supply cylinder with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety code. Suitable reducing valves and adaptors for 300 to 500-psi (2070 to 3440 KPa) discharge pressure are obtainable from commercial sources of compressed gas equipment. Check the pressure gage periodically for accuracy.

11.1.1.4 During ignition of a sample, the operator must not permit any portion of his body to extend over the calorimeter.

11.1.1.5 Exercise extreme caution when combustion aids are employed so as not to exceed the bomb manufacturer's recommendations and to avoid damage to the bomb. Do not fire loose fluffy material, such as unpelleted benzoic acid, unless thoroughly mixed with the sample.

11.1.1.6 Admit oxygen slowly into the bomb so as not to blow powdered material from the crucible.

11.1.1.7 Do not fire the bomb if it has been filled to greater than 30 atm (3 MPa) pressure with oxygen, the bomb has been dropped or turned over after loading, or there is evidence of a gas leak when the bomb is submerged in the calorimeter water.

11.2 *Subsequent Treatment*—Remove the bomb from the calorimeter water and open the valve carefully so as to allow the gases to escape at an approximately even rate so the pressure is reduced to atmospheric in not less than 1 min. Bombs equipped with valves other than needle valves, such as compression valves, shall be provided with a device so the valve can be controlled to permit a slow and uniform release of the gases. Open the bomb and examine the inside for traces of unburned material or sooty deposit. If these are found, discard the determination. Wash carefully all parts of the interior of the bomb, including the capsule, with a fine jet of water containing methyl orange (10.6) until no acid reaction is observed. It is essential to wash through the valve opening in the case of bombs equipped with compression valves, or other types of valves with large openings, as considerable spray may collect in such valve openings.

11.3 Collect the washings in a 250-mL beaker and titrate with standard sodium carbonate solution (10.5) to obtain the "acid correction" for the heating value, as specified under the calori-

[8] Selvig, W. A., and Fieldner, A. C. "Check Determinations of Sulfur in Coal and Coke by the Eschka, Bomb-Washing and Sodium Peroxide Fusion Methods," *Industrial and Engineering Chemistry*, JECHA, Vol 29, 1927, pp. 729–733.

metric determination Test Method D 2015 or D 3286. Adjust the pH from 5.5 to 7.0 with dilute NH_4OH, heat the solution to boiling, and filter through a qualitative paper. Wash the residue and paper thoroughly five or six times with hot water. Adjust the acidity of the filtrate and washings, amounting to about 250 mL, precipitate, and determine the sulfur as specified under the Eschka method, Sections 6 through 9, inclusive.

NOTE 2—If the use of 1-g sample weight in the calorimetric determination produces an excess amount of sulfate that cannot be precipitated by the addition of 10 mL of barium chloride solution, either of the following alternatives may be used: (*1*) increase the amount of the baruim chloride solution from 10 mL in increments of 5 mL up to a maximum of 20 mL of solution, or (*2*) reduce the amount of sample from 1 to 0.5 g and add 0.5 g of benzoic acid in order to maintain appropriate temperature rise so the precision of the gross calorific value determination is not adversely affected.

12. Report

12.1 The percentage sulfur value obtained using any of the described methods is on an as-determined basis.

12.2 The results of the sulfur analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

12.3 Use the percentage of moisture as determined by Test Method D 3173 to calculate the as-determined results from the analysis basis to a dry basis.

12.4 Procedures for converting the value obtained on the analysis sample to other bases are described in Methods D 3176 and D 3180.

13. Precision and Bias

13.1 *Repeatability*—Results of two consecutive determinations carried out on the same sample in the same laboratory by the same operator using the same apparatus should not differ more than the following in more than 5 of 100 instances:

	%
Coal containing less than 2 % sulfur	0.05
Coal containing 2 % sulfur or more	0.10
Coke	0.03

13.2 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples taken from the same bulk sample after the last stage of reduction should not differ by more than the following in more than 5 of 100 instances:

	%
Coal containing less than 2 % sulfur	0.10
Coal containing 2 % sulfur or more	0.20
Coke	0.05

13.3 *Bias*—These are stoichiometric methods that agree with each other very well when known amounts of solutions or compounds containing predetermined quantities of sulfur (preferably as sulfate) are added to blanks determined as described in 8.4.

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SPECIAL ANALYTICAL SERVICES
Client Request

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A. EPA Region/Client: Region V/ Onalaska Municipal Landfill

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Onalaska Municipal Landfill

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 organic carbon(%) in soil (air-dried - all screened through 100 or 140 mesh). Applicable concentration 0.1% to 2.0% or more. Detailed information must be provided with Case Narrative for test procedures, instrumentation/ apparatus, and QC used. See Attachment I.

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 concentration):

Analyze 13 soil samples for total organic carbon (TOC).

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

Superfund, Remedial

4. Estimated date(s) of collections: _____

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

6. Number of days analysis and data required after laboratory receipt of samples:

_____

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

See Attachment 7

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

See Attachment 8

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 9

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

_____

11. Name of sampling/shipping contact: _____

Phone: _____

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+% or Conc.)
Organic Carbon % in soil	0.10% Report actual detection limit if smaller.	± 20% on duplicate sample results

II. QC REQUIREMENTS

	Audits Required	Frequency of Audits	Limits* (% or Conc.)
1.	Prep. Blank	1 in 10 samples, or at least twice.	≤ 0.1%
2.	Duplicate Samples	1 in 5 Samples.	< 20 % RPD in differences of duplicate sample results, or < 0.2% differences at small concentrations
3.	Positive Control (To be determined by lab)	1 in 10 samples	85-115% Recovery
4.	Instrument Calibration checks and Calibration Blanks (if appropriate	1 in 10 or fewer samples	90-110% Recovery for calibration check, and <0.1% total Carbon for assumed routine sample weight.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

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 I

Determination of organic carbon(%) in soil, using sub-aliquots of air-dried soil, passed through a 100 mesh to 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 2% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil shall be the 1) dry combustion (resistence furnace), 2) Dry combustion (induction furnace), 3) Dry combusiton (automated methods), or 4) Wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses," Part 2 - Chemical and Microbiological properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because no laboratory doing organic carbon analysis of soil should be without it.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd ed., as appropriate. Soils can be calcerous or noncalcerous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory, providing organic carbon analysis data, will provide information with the case narrative concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items #8, and #9 of this SAS.

ATTACHMENT 7
Analytical Methods - Organic Carbon in Soil

7a. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.

b. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (-5 min). If inorganic carbon is absent proceed with Total Carbon in items #7c, or 7d below. If inorganic carbon is present, or the test is not definitive, proceed with tiems #7e, or #7f prior to Total Carbon measurements of Item #7c or #7d.

c. Total Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:

1) Total Carbon (Dry Combustion - Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.

2) Total Carbon (Dry Combusiton - High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.

3) Total Carbon (Dry Combusiton - Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3, and 29-2.2.4.

d. Total Carbon (Wet Digestion), MSA, part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing K_2CrO_7). CO_2 evolved is absorbed and weighed, or absorbed in standard base and titrated.

1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.

e. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H_2SO_3). After several hours, remove the excess H_2SO_3 by leaving overnight in an evacuated dessicator. Read citation for further details.

f. Pretreatment prior to Wet Digestion, MSA, Part II, Seciton 29-3.3.2. Inorganic carbon is removed by sulfuric acid - ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

- (cont. next page) -

ATTACHMENT 7 (Cont.)

g. Use only the methods specified above or obtain approval of CPMS, CRL prior to use of other method. Test procedure description, and description of specific measurement principles including equivalency to each of the 10 items of Figure 29-1 of MSA, part II and sample pretreatmenst of Section 29-3, MSA, Part II.

h. Laboratory performing Total Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in sample.

A variety of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide calibration curve, including zero concentration standard and preparation blanks. Provide positive control (a test sample prepared independently from calibration standards) that provides a measure of accuracy of system. This should be done for all systems including grarmetric read-outs.

ATTACHMENT 3

Analytical Results Required

As part of Case Narrative, attach description of test procedure and instrumentation used for measurement of Total C and removal of any Inorganic C. Test procdure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and soultion taken to resolve any problems. A sample preparation log will be provided, as appropriate.

Bench record tabulating any order of any sample weights and tare weights of absorbed CO_2, instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be legible. Report results as % organic Carbon on a dry weight basis (103-105°).

Appendix C
CLOSE SUPPORT LABORATORY
STANDARD OPERATING PROCEDURES

CONTENTS

TABLES

FIGURES

*Follows page cited

INTRODUCTION

This report describes the standard operating procedures for the Close Support Laboratory (CSL) used for the Onalaska Landfill Remedial Investigation. The procedures are intended to provide guidance to persons working in the CSL and others using CSL data. Other site-specific information can be found in the Quality Assurance Project Plan, Sampling Plan, and Health and Safety Plan.

ORGANIZATION AND MANAGEMENT

CSL PROGRAM ORGANIZATION

The CSL program organization is shown in Figure 1. The CSL Program Manager is responsible for the overall CSL program, including facilities, staffing, and task management, and performs these duties by directing the CSL Operations Manager.

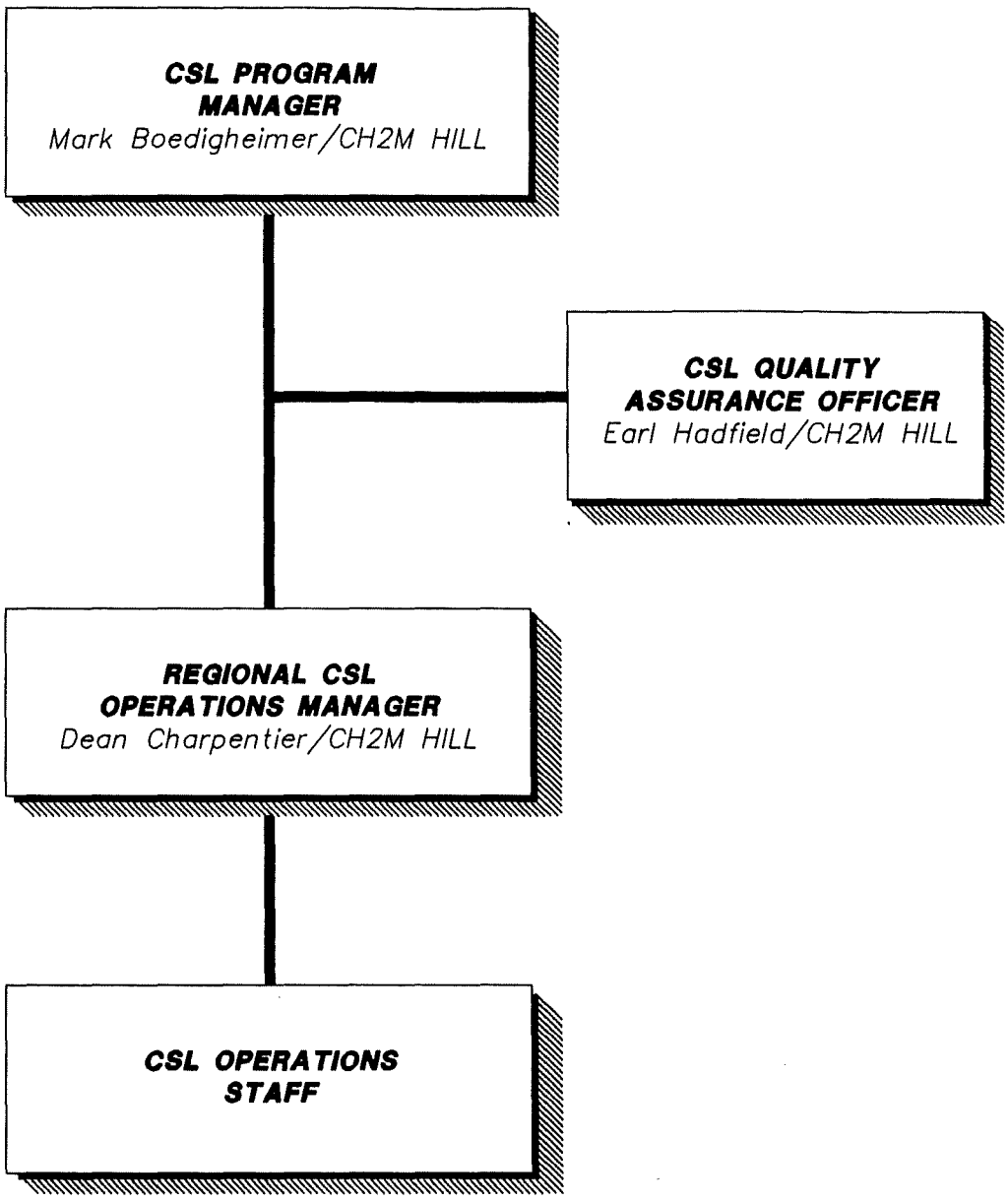
The Quality Assurance Officer is responsible for the overall program QA, ensuring that the data meet QA criteria.

The CSL Operations Manager is responsible for the financial and technical operations of the CSL, providing guidance specific to the site with respect to methods, QA/QC, analyst training, equipment and supply purchasing, maintenance, troubleshooting, sample and data management, health and safety, waste disposal, and demobilization.

SITE ORGANIZATION AND RESPONSIBILITIES

The field team organizational chart indicates channels of communication between project team members (Figure 2) to facilitate information flow, work scheduling, and problem solving. It is intended to promote direct communication between CSL team members, project task leaders, and the Site Manager so that minor, nontechnical issues can be resolved in the field.

The Site Manager has overall responsibility for the project, including budgeting, scheduling, and communication with the client. The Project Manager directs field investigation activities through the Field Team Leader, who is responsible for all field activities at the site. The Field Team Leader is responsible for providing field samples, requesting specific analyses, setting data priorities, and scheduling the analytical work. Either the Field Team Leader or Site Manager may report any problems to the CSL Operations Manager.



W65550.PP\FIG1 1/12/89

FIGURE 1
CSL ORGANIZATION
ONALASKA SOP

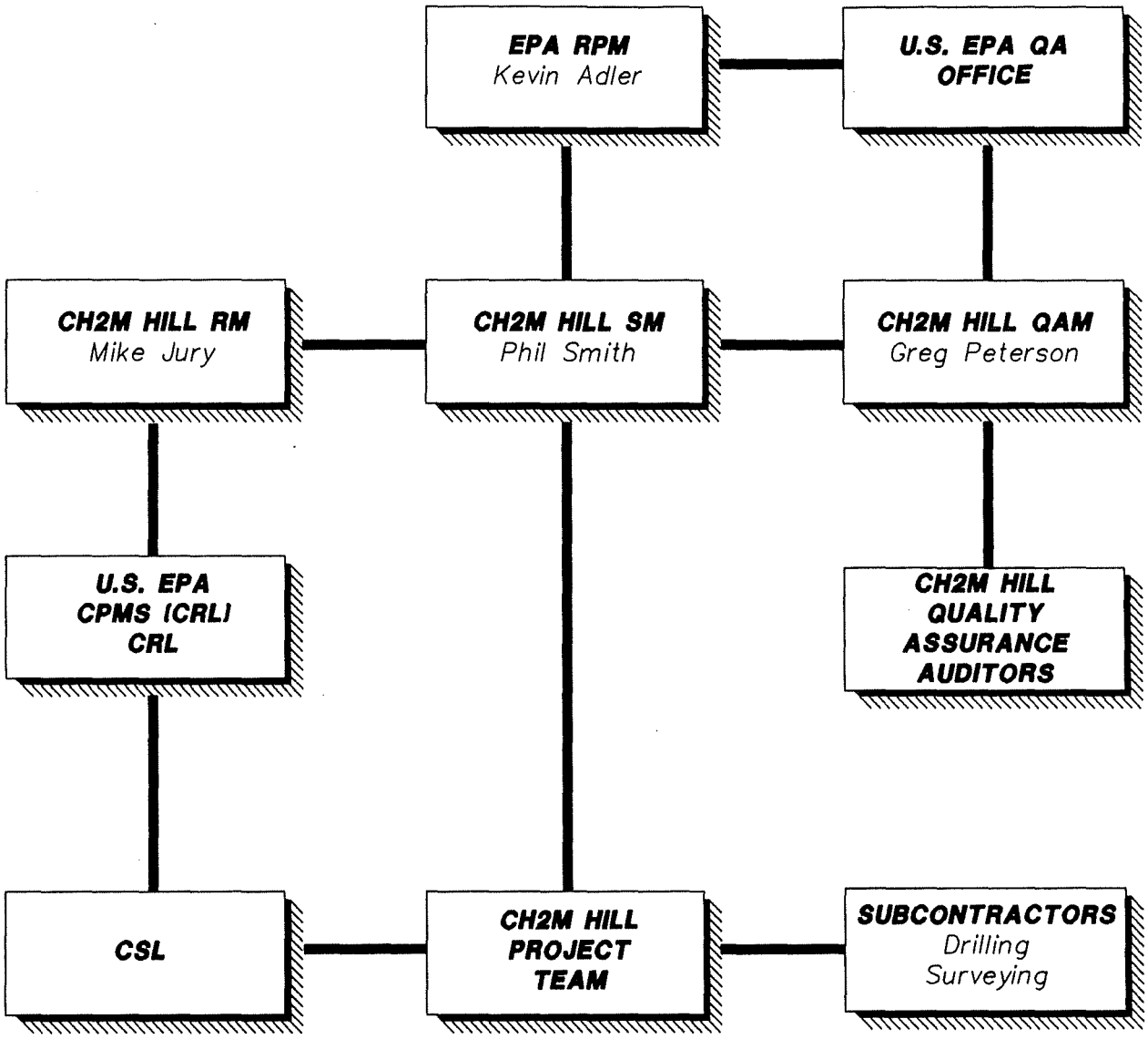


FIGURE 2
PROJECT ORGANIZATION
ONALASKA SOP

W655560.PP\FIG2 1/--/89

The CSL Operations staff is responsible for daily operation of the CSL, which includes providing timely analytical data within quality and budgetary guidelines.

DATA QUALITY OBJECTIVES

The CSL analytical performance objectives are not intended to equal those associated with offsite laboratories using EPA protocols or with CLP laboratories. Analyses performed by the CSL are intended to aid in the characterization of soils, water, and investigation derived materials. They are considered to be of Level II analytical quality/data use category as defined by EPA's Data Quality Objectives for Remedial Response Activities (Volume 1, 1987). Level II data are data from field analytical techniques characterized by tentative identification, presence or absence, screening, or determination of relative concentration.

The use of the CSL data will vary depending on the origin of the samples and the site activities that generate them. Data will be used for quickly identifying source areas and screening samples. CSL data typically improve the ability of the Field Team Leader and project task leaders to make informed and prompt decisions. For example, a task leader might use CSL data to determine the areal extent of contamination or to aid in the coordination of work activities.

The CSL data quality objectives summarized in Table 1 have been determined to be reasonable based on CH2M HILL's experience with similar complex matrices and project needs. However, they are only target values. Actual values will depend on the analyst, sample matrix, and CSL analytical conditions. The CSL data will be evaluated, in part, by comparison with the initial target values. If the target values cannot be achieved, then new ones will be specified.

LABORATORY OPERATIONS

ANALYTICAL METHODS

The analytical methods employed by the CSL have been developed and validated for a specific list of Onalaska Landfill indicator constituents that were selected on the basis of results obtained during previous site work. The analytical methods used are detailed in Attachment 1.

Table 1
DATA QUALITY OBJECTIVES

	MDL[a] Water (ug/l)	MDL Soil/ Sediment (ug/kg)	MDL Soil Gas (ug/l)	Precision[b] RPD (±%)	Accuracy[c] Recovery (%)
Total-1,2-Dichloroethylene	10.0	20.0	TBD[d]	30	38-155
1,1,1-Trichloroethane (111TCA)	10.0	20.0	TBD	30	41-138
Perchloroethylene (PCE)	10.0	20.0	TBD	30	26-162
Trichloroethylene (TCE)	10.0	20.0	TBD	30	35-146
Toluene	50.0	100.0	TBD	30	46-148
Xylene	50.0	100.0	TBD	30	60-140

[a] Method Detection Limit.
[b] Precision is relative percent difference of sample duplicates or matrix spike duplicates.
[c] Accuracy is range of spike recovery of a matrix spike analysis.
[d] To be determined

GLT824/66

SAMPLE MANAGEMENT

Samples to be analyzed by the CSL will be provided by the Field Team Leader or a designated field crew member. Samples will be assigned identifying numbers by the field crew in accordance with the Site Sampling Plan.

A sample logbook will be used to record the samples received by the CSL analyst. The logbook will contain the signature of the person relinquishing the sample, the sample designation number, date and time, initials of the sample recipient (analyst), sample type (composite or grab), sample description (clay, sandy, etc.), and sample disposition, location, and date (Figure 3).

The sample logbook contains essential information necessary for chain-of-custody purposes. The analyst will report deficiencies in the sample documentation or confusing requests for analyses to the sample Team Leader for resolution. The samples will normally be analyzed in the order in which they are submitted. Should the Team Leader request priority analysis, it will be noted in the sample logbook and analyzed in the specified priority.

Following the completion of the analysis and review process, the unused portion of the sample will be moved to and archived in a secured storage area within the confines of the CSL. The samples will be stored through the duration of the analysis. All sample aliquots used for analysis by the CSL will be discarded in a drum labeled "Lab Wastes Only."

Sample Preparation

Samples will be prepared to obtain homogenous, representative sub-samples for analysis. Preparation will take place in a fume hood for analyst protection. Details of sample preparation for the expected sample matrices are described in the analytical methods.

Sample Storage and Disposal

When the analysis is completed and the data reviewed by the analyst, a decision concerning final disposition of the sample will be made. Samples might be disposed of as described under "CSL Waste Management" or, at the request of the Field Team Leader, by archiving then or sending them to an offsite laboratory for further analysis. Sample disposal must be authorized by the Field Team Leader.

PREVENTIVE MAINTENANCE

Preventive maintenance will be performed in accordance with instrument manufacturers' manuals. Analytical components will be replaced as needed. Precision and accuracy data will be examined for trends and excursions beyond the limits in Table 1 and the analytical methods to determine evidence of equipment malfunction. Maintenance will be performed or other corrective actions made when shifts in calibration, decreased sensitivity, or failure to meet QC criteria are observed. Instrument logbooks will contain a history of routine and nonroutine maintenance. Instrument downtime can be minimized by having an inventory of expendable supplies and critical instrument components.

CSL WASTE MANAGEMENT

Three distinct types of wastes will be generated by the CSL. They will be classified and handled as follows:

CSL Waste Type	Handling Method
Sample Residues o Chemical extracts o Materials used in preparation of samples	Place in designated waste receptacles marked "Lab Wastes Only." Transfer to drum marked "Lab Wastes." Final disposition will be in accordance with the projects investigation derived materials.
Lab Waste Water o Sink drains o Rinse water o Instrument cooling water	Transfer contents to drums marked "Lab Waste Water." Final disposition will be in accordance with the project's investigation derived materials.
Lab Trash (non-hazardous) o Miscellaneous paper o Paper towels o Boxes and other shipping materials	Place in trash can marked "Trash Only." Dispose of at a landfill along with trash from other command post activities.

PURCHASING

After the CSL has been mobilized, the day-to-day laboratory operations will require the purchase of miscellaneous items

FIGURE 3: CSL LOGBOOK

DATE	TIME	RELINGUISHED BY	SAMPLE I.D.	SAMPLE TYPE	ANALYST	COMMENTS

(e.g., deionized water). Approval to purchase such items must be granted by the CSL Operations Manager.

DATA MANAGEMENT

All data will be recorded on a CSL data sheet (Figure 4). Data sheets and chromatograms will be kept in the CSL throughout the duration of the site investigation. Copies of these data will be made available for validation purposes in accordance with the section "Data Validation." The actual analytical values produced by the CSL will be entered into a database by the attending analyst. The database will include such information as sample identity, sample type, date received and analyzed, analytical method used, and data validation status. All data will be stored and updated on a floppy diskette. A backup and hard copy of the diskette will be made and continually updated.

The CSL data will be reported in accordance with the procedure described under "Data Management--Reporting." The data will be stored and archived along with the project files at the CH2M HILL Milwaukee office.

RECORDING

All data will be recorded in a clear and comprehensive manner. "Data" is defined as all analytical information in numerical and narrative form. This information will be recorded in ink in bound books having consecutively numbered pages. Entries in the books will be dated and signed on each line, page, or book as appropriate. Errors will be lined out with a single line and corrections initialed and dated. The bound books may include:

- Sample Logbook
- Analysis Data Book containing lists of sample parameters, sample designation, raw data, calculations, results, comments, and qualifiers
- Daily Journal containing descriptions of significant events, trailer and analytical equipment breakdown/repair comments, problems and solutions, requests for help and information from task leaders, etc.
- Any other instrument specific logbooks

HANDLING AND STORAGE

All data and instrument readouts (e.g., chromatograms, digital readouts) will be kept in the CSL trailer in the possession of the analyst for the duration of the field investigation. Copies of the data will be made available to the Sample Team for decision and review purposes. The analyst will enter analytical values and qualifiers into the database.

REPORTING

On completion of each day's batch of samples, the data will be reported to the Field Team Leader. At that point the data will not have passed the data validation check, as described in the section "Data Validation," and thus will be designated "preliminary." Data will be validated by a qualified chemist who is not associated with the CSL at this site. Upon approval of the chemist, data will be designated in the database as "validated."

Upon completion of the project, a decision will be made as to whether the CSL data will be included as part of the project reports. If used, the CSL data should be qualified by its definition and intended use in accordance with the data quality objectives for the project.

HEALTH AND SAFETY

The CSL operations staff will follow the guidelines stated in the project Health and Safety Plan. Typically they will apply when the CSL operators are outside the confines of the laboratory, but they may also apply in certain situations to activities performed within the lab trailer. Specific health and safety guidelines for CSL operations are presented in Attachment 1.

As a part of the ARCS Health and Safety Program, it is a matter of policy that all CSL operations staff will have up-to-date medical physicals. In addition to the general site health and safety orientation given to all site personnel, the CSL operations personnel will be given a CSL health and safety orientation before they begin operations. It will be the duty of the Site Safety Officer to monitor CSL health and safety and report any deficiencies to the Site Manager/Field Team Leader and CSL Operations Manager.

The CSL staff will follow standard laboratory safety practices for eye protection (safety glasses), inhalation

FIGURE 4: ONALASKA CLOSE SUPPORT LABORATORY DATA SHEET

SAMPLE INFORMATION

SAMPLE IDENITY _____ DATE RECEIVED _____

ANALYST _____ DATE ANALYZED _____

TYPE OF ANALYSIS _____

_____ SAMPLE _____ LAB BLANK _____ MATRIX SP _____ MATRIX SPIKE DUP _____ DUPLICATE _____ CAL CHECK

MATRIX DESCRIPTION

ANALYSIS

WATER VOLUME, mls---Vs _____ SOIL WEIGHT, gms---Ws _____

INJECTION TIME, sec _____ DILUTION FACTOR ---DF _____

	CONC. OF CONSTITUENT FOUND BY THE INSTRUMENT ug/ml---A	RETENTION TIME CHECK IF RT > +/- 0.10 min FLAG IF RT > +/- 0.20 min REJECT	CONC. OF CONSTITUENT IN THE SAMPLE ug/kg or ug/l
1,2-DICHLOROETHYLENE			
1,1,1-TRICHLOROETHANE			
TRICHLOROETHYLENE			
PERCHLOROETHYLENE			
TOLUENE			
YLENES			

CALCULATIONS

CONC. IN SOIL (ug/kg) = [(A x Vs x DF)/Ws)] x1000

CONC. IN WATER (ug/l) = A x DF x 1000

QUALITY CONTROL

	BACKGRD RESULT ppb	SAMPLE RESULT ppb	SPIKE LEVEL ppb	PERCENT RECOVERY	QC LIMITS %	BACKGRD RESULT ppb	SAMPLE RESULT ppb	SPIKE LEVEL ppb	PERCENT RECOVERY	QC LIMITS %	RPD %	QC LIMIT %
DICHLOROETHYLENE					38-155					38-155		30
TRICHLOROETHANE					41-138					41-138		30
TRICHLOROETHYLENE					35-146					35-146		30
PERCHLOROETHYLENE					26-162					26-162		30
TOLUENE					46-148					46-148		30
XYLENE(S)					30-150					30-150		30

QUALITY CONTROL SUMMARY

_____ OF _____ RECOVERIES OUT OF QC LIMITS

_____ OF _____ DUPLICATES OUT OF QC LIMITS

Examples of corrective actions include reanalyzing samples, recalibration with fresh standards, replacement of reagents that may be sources of blank contamination, examination of calculation procedures, additional training in sample preparation and analysis, or reassignment of analytical responsibilities.

Documentation of corrective action steps must include problem identification, action to eliminate the problem, increased monitoring of the effectiveness of the corrective action, and verification of problem elimination.

DATA VALIDATION

The CSL is supposed to provide data in a timely manner at the appropriate level of quality. To satisfy that objective, the CSL data will undergo two levels of review and validation.

The first level of review will be performed by the CSL staff, which generated the data, before the data are released to the Sample Team. This is the customary "double checking" of the data for conformance with the protocols detailed in these procedures and in Attachment 1.

The second level of review is performed outside the CSL. Each week a batch of data copies along with copies of the CSL diary will be submitted to a qualified chemist who is not associated with the CSL for data validation and checking of data reduction and reporting. Data validation consists of the steps taken so that the reported results correctly represent the samples and the analyses performed. There are two basic validation activities:

- Checking sample results and QC sample results to demonstrate that the analyses are within prescribed criteria for precision, accuracy, completeness, method detection limit, understandability, and legibility

- Checking the numerical computations for correct data and correct reporting of data

The data will be validated for consistency with the data quality objectives and items of importance in the analytical procedures used. In addition to numerical evaluation, the reviewer will evaluate the performance of the analytical system by reviewing the chromatograms for peak resolution, retention times, baseline noise and drift, and peak shape

and areas. Emphasis will also be placed on blank analysis, initial and continuing calibration, spike and matrix spike duplicate analysis, holding times, and reported results relative to additional cleanup or dilutions.

The following data qualifiers will be used to classify the data for conformance with QC requirements:

B = Blank contamination
N = Qualitatively suspect
J = Quantitatively suspect
R = Unable to calculate due to interference
U = Not detected at the specified detection limits

Retention time shift, instrument noise, or sample matrix interference are possible reasons for using the "N" qualifier. Calibration problems, blank contamination, or spike recoveries are possible reasons for using the "J" qualifier.

After the data validation process, the data reviewer will return the data to the Field Team Leader. Validated data will be considered final data. If operational problems become evident during validation, the CSL Operations Manager will determine the source of the problem, develop a plan, take corrective action, and report the results of his or her investigation to the Field Team Leader.

Attachment C-1
CLOSE SUPPORT LABORATORY METHOD FOR SCREENING ANALYSIS OF VOLATILE INDICATOR PARAMETERS IN SOIL AND WASTE SAMPLES

GLT809/55-2

CSL METHOD: VOA/SOIL/PENTANE EXT/GC-ECD/FID

CLOSE SUPPORT LABORATORY METHOD FOR ANALYSIS OF SELECTED ORGANIC COMPOUNDS

1. SCOPE AND APPLICATION

1.1 This method is used for field screening of soil and solid samples for organic compounds that are indicative of contamination at the Onalaska Landfill site. It is presented as a means to rapidly characterize contamination in soil samples. The method is semi-qualitative and semi-quantitative for the following target compounds:

Target Constituents

total-1,2-Dichloroethylene (DCE)
1,1,1-Trichloroethane (TCA)
Trichloroethylene (TCE)
Perchloroethylene (PCE)
Toluene
Xylene(s)

1.2 Application of this method is limited to the screening analysis of soil for the target constituents. The chromatographic record produced in the analyses allows the site investigation team to examine the relative degree of soil contamination associated with other nontargeted compounds in the sample extracts. Specifically, this method will be used to help locate source areas of contamination and to screen samples for selection and submittal to CLP laboratories. Positive identification and quantification of specific constituents, such as these constituents and other priority pollutants, will be supported by analyses of replicate and other composited samples at a remote CLP laboratory employing EPA approved testing protocols.

1.3 Preliminary method validation data indicates analyte recoveries of upwards of 80 percent.

1.4 The method detection limits (MDL) for the target constituents are estimated to be 20 ug/kg for the chlorinated compounds and 100 ug/kg for the aromatic target compounds. These estimates are the result of previous method development work. Interferences may affect method detection limits for each of the target compounds.

2. SUMMARY OF METHOD

2.1 The method presented here is loosely based on EPA Method 3550, *"Sonification Extraction,"* Method 8000, *"Gas Chromatography Analysis,"* found in EPA SW846, Test Methods for Evaluating Solid Waste, 3rd ed., November 1986. In brief, pentane is used in conjunction with sonification to affect extraction of the target parameters from the sample matrix. The extract is subsequently analyzed on a capillary gas chromatograph using both an electron capture detector for the chlorinated compounds and a flame ionization detector for the aromatic compounds.

3. SAFETY

3.1 The target constituents are either identified as or suspected of being carcinogens. All samples are assumed to be hazardous. All stock and working calibration standards, as well as all samples, shall be handled with the utmost care using good laboratory techniques in order to avoid harmful exposure.

3.2 Laboratory analysts shall wear lab coats, safety glasses, and surgical gloves at all times when preparing and handling standards and field and lab samples.

3.3 Standards and samples shall be prepared in a fume hood.

3.4 Pentane (C_5H_{12}) is regulated by NIOSH. The suggested permissible exposure level (PEL) is 120 ppm with a ceiling level of 610 ppm. Exposure pathways are oral, dermal, and airway. Effects of short-term exposure are drowsiness and irritation of eyes and nose, large doses may cause unconsciousness. Prolonged overexposure may cause irritation of skin. The odor threshold of n-pentane is reported as 2.2 ppm. Pentane is highly flammable and is incompatible with strong oxidizing agents.

3.5 Sample extracts and standards prepared in flammable solvents shall be stored in a refrigerator or in a cooler (outside the laboratory).

3.6 All of the target compounds are reported in the NIOSH manual as having good warning properties. Any situation which leads to or causes noticeable odors or produces any physical symptoms in the workers shall be investigated immediately followed by the appropriate corrective action.

3.7 The ultrasonic sonicator used for the sample extraction emits a high frequency sound. When in use, the sonicator horn shall be inside the sound chamber with the door closed.

3.8 Safety equipment including a fire extinguisher, first aid kit, eye wash, and chemical spill clean-up kit shall be available for use at all times.

3.9 Laboratory wastes shall be separated and properly disposed of. The wastes include: used sample aliquots, initial wash water, chemical wastes generated in the analysis, and disposables used in the preparation of the samples. These wastes shall be collected and deposited in a drum clearly marked as "CSL Lab Wastes Only--Hazardous". Water used for final rinsing of glassware will be considered nonhazardous and will be released into a 55-gallon drum. These wastes will then be disposed of in accordance with the appropriate and relevant disposal method.

4. SAMPLE SIZE REQUIREMENT AND SAMPLE COLLECTION

4.1 The purpose of soil sampling is to obtain a representative portion of soil that can be submitted for chemical analysis. In the case of surface soil, samples will be taken with a stainless-steel spoon or stainless steel garden trowel. The sample will then be transferred to a 120-ml widemouth glass bottle with a teflon-lined cap. The sample bottles should be filled to the extent possible. In some cases, it may be necessary to break or consolidate the sample matrix into a manageable size before placing the soil in the sample bottle.

4.2 Sampling equipment will be decontaminated after each sample has been collected. Refer to the Field Sampling Plan for details outlining sample collection and decontamination procedures.

4.3 Samples will be kept cool at 4 degrees Celsius until CSL analysis.

4.4 The EPA holding time for soil samples submitted for volatiles analysis is 14 days.

5. DETECTION LIMITS AND WORKING LINEAR RANGES

5.1 The method detection limit (MDL) for the target constituents is estimated to be 20 ug/kg for the chlorinated compounds, and the working linear range for these compounds is estimated to be 20 ug/kg to 2000 ug/kg. The method detection limit for the aromatic target compounds is estimated to be 100 ug/kg while the linear range for these compounds is estimated to be 100 ug/kg to 5000 ug/kg. These estimates are the result of previous method development work.

6. INTERFERENCE AND CORRECTIVE MEASUREMENTS

6.1 Samples containing compounds that co-elute with the target constituents may cause a positive bias in the results.

6.2 The presence of compounds that closely match the retention times of the target constituents may result in false identifications.

6.3 The MDLs for the target constituents may be suppressed by baseline noise associated with samples having high levels of background organics or other interferences.

6.4 The response factors for uncalibrated peaks that are significantly different than those of the target constituents may produce errors in the estimation of total target constituent contamination.

6.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for cross-contamination.

7. APPARATUS AND MATERIALS

7.1 Soil sampling equipment--described in Field Sampling Plan.

7.2 VOA soil sample bottles--120-ml capacity with Teflon-lined caps.

7.3 Gas chromatograph (GC)--Hewlett-Packard Model 5890A; temperature programming, electronic integration, report annotation, automatic sampler, 30-meter capillary column, flame ionization detector, electron capture detector.

7.4 Balance--Sartorius; top loading electronic with 1,500-gram capacity with 0.01-gram sensitivity.

7.5 Glassware--Class A volumetric pipets and flasks; beakers, vials, Pasteur pipets, and miscellaneous glassware as necessary for preparation and handling of samples and standards.

7.6 Labware--necessary for preparation and handling of samples and standards.

7.7 Syringes--Hamilton glass type as required for injection of sample extracts and standards, preparation of dilutions, and spiking of samples.

7.8 Sonifier--Heat Systems Ultrasonic Sonicator with variable control up to 375 watt output and water-cooled cup horn.

8. REAGENTS

8.1 Pentane (C_5H_{12})--spectrophotometric grade.

8.2 Sodium sulfate--reagent grade, anhydrous powder form.

8.3 Stock standards--prepare or purchase standard materials at approximately 1000 mg/l in methanol.

8.4 Working standards--prepared from stock standards by precise dilution in methanol for the chlorinated target compounds (20 ug/l, 200 ug/l, and 2000 ug/l); prepared for the aromatic compounds (100 ug/l, 1000 ug/l, and 5000 ug/l).

8.5 Hydrogen--carrier gas, grade 5.

8.6 Zero Grade Air (<0.1 ppm hydrocarbons)

8.7 Nitrogen makeup gas, prepurified grade.

8.8 Distilled water--adjusted to pH 12 with 5N NaOH.

9. CALIBRATION PROCEDURES

9.1 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels (20 ug/l, 200 ug/l, and 2000 ug/l for the chlorinated target compounds and 100 ug/l, 1000 ug/l, and 5000 ug/l for the aromatic compounds) by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with pentane.

9.2 Inject each calibration standard mix used into the gas chromatograph (i.e. 2-ul injections). Tabulate response factors for each calibration compound for each run. The units for the response factor are based on concentration per area.

Response factor = Std. Conc./Area for Std.

Where:

Std. Conc. = The concentration of the prepared standard (e.g., 1 ug/ml toluene)

Area for Std. = Integrated peak area as reported by the GC integrator (e.g., area for toluene)

9.3 Sample concentrations of unknowns can be calculated directly **if** the

response factor is the same for every compound at each calibration level. If not, the least squares line must be found using the calibration data, and sample concentrations for unknowns will be calculated using the least squares equation. In either case, the results must be used to prepare a calibration curve for each analyte.

9.4 The working calibration curve must be verified on each working day by the injection of the mid-range calibration standard.

10. SAMPLE PREPARATION

10.1 In a labeled VOA vial, add an aliquot of soil (approximately 5 grams to the vial) and accurately record the soil sample weight to the nearest 0.01 grams. Place the vial on the top loading balance and record its tare weight. Volumetrically pipet 5.0 ml of the pentane solvent into the sample vial.

10.2 Sample treatment: If the sample is wet or a highly consolidated material (e.g., clay), then add about 2 grams of sodium sulfate and mix. It may be necessary to integrate the sodium sulfate into the sample using a spatula or another appropriate utensil.

10.3 With the VOA vial cap tightly in place, sonicate at an output setting of 30 percent for approximately 5 minutes. The resultant sonified sample should be dispersed throughout the pentane solvent and have a grain-like appearance. If not, add an additional 1 gram of sodium sulfate and resonify. Repetitions of this process may be needed to properly extract some samples.

10.4 After sonification, let the VOA vial stand until the solids have settled. Using a Pastuer pipet, transfer a suitable aliquot of the pentane solvent (extract) from the vial into a labeled GC autosampler vial and cap immediately with septum crimp seals. Refrigerate the sample extracts until analyzed.

10.5 If the extract is noticeably colored, turbid, or otherwise indicates a dirty sample, it may be advantageous to do some cleanup by adding approximately 10 ml of pH 12 water and shaking vigorously. This will remove phenolic compounds and results in a cleaner extract. If the sample is obviously contaminated and past experience has shown similar extracts to contain high levels of contamination, it is recommended that a preliminary dilution be made to avoid grossly contaminating the gas chromatograph.

11. ANALYTICAL PROCEDURE

11.1 Perform GC analysis on the extract using the instrument conditions similar to those listed in Exhibit 1.

11.2 If the analysis indicates that the results are more than 50 percent above the calibration range, dilute the sample extract so that concentrations fall within the calibration range.

11.3 Check the retention values for each of the target constituents against the expected (calibration) value. Reject those results where the retention time does not fall within +/- 0.15 minute of the expected values.

11.4 Use the retention time marker as an indicator of the reliability of each sample injection and GC run. The retention time marker should fall within the same windows as the target constituents and should be within +/- 15 percent of the area counts of the initial calibration value. If these criteria are not met, re-evaluate the data using relative retention times. Reruns should occur to resolve data suspicions.

11.5 If a grossly contaminated sample is injected, it may be necessary to run a solvent blank several times afterward to clean residual contamination from the injection port and column. If this does not work, and blank runs still contain contamination, it will be necessary to change the injection port liner. Following the change of the liner, several blank runs will be needed to obtain a stable baseline.

12. DETAILS OF CALCULATIONS

12.1 Quantification of the target compounds is based on the integrated areas of the samples in comparison to the integrated areas of the calibration standards for each analysis. The integrator reports the concentrations in ug/ml in the extracts. Calculation of the concentration for each target constituent in the original sample on an as-received basis is as follows:

$$\text{Conc. in ug/kg} = \frac{A \times Vt \times DF}{Ws} \times 1{,}000$$

Where:

A = Amount of target constituent found in the extract in ug/gram

Vt = Volume of solvent added to the VOA vial, 5.0 ml

DF = Dilution factor, if required

1,000 = Dimensional correction factor

Ws = Weight of the sample added to the VOA vial in grams

13. QUALITY CONTROL REQUIREMENTS

13.1 Daily mid-range calibration checks performed prior to the analysis of each day's lot of samples or with each lot of 20 samples, whichever is more frequent.

13.2 Analysis of field blank samples at a frequency of 1 in 20 samples analyzed or 1/day, whichever is more frequent.

13.3 Analysis of laboratory blank samples at the same frequency. Should the results of the laboratory blanks show contamination, the cause of the contamination should be investigated and corrective action be taken.

13.4 Analysis of field replicate samples at a frequency of 1 in 20 or 1/day whichever is more frequent.

13.5 Analysis of a mid-range matrix spike samples and a matrix spike duplicate at a frequency of 1 in 20 samples analyzed or 1/day, whichever is more frequent.

13.6 Use of a retention time marker during the analysis of all samples and standards.

14. REPORT REQUIREMENTS

14.1 On completion of each day's batch of samples, the data will be reported to the Field Task Leader. This will take the form of copies of the pages from the Analysis Data Book and a summary table generated by a database program. The Field Task Leader will be responsible for disseminating the data to others as appropriate. At this point the data will not have passed the data validation process as described in the CSL QA document and will be considered preliminary. Ultimately, the CSL data will be archived along with other project records.

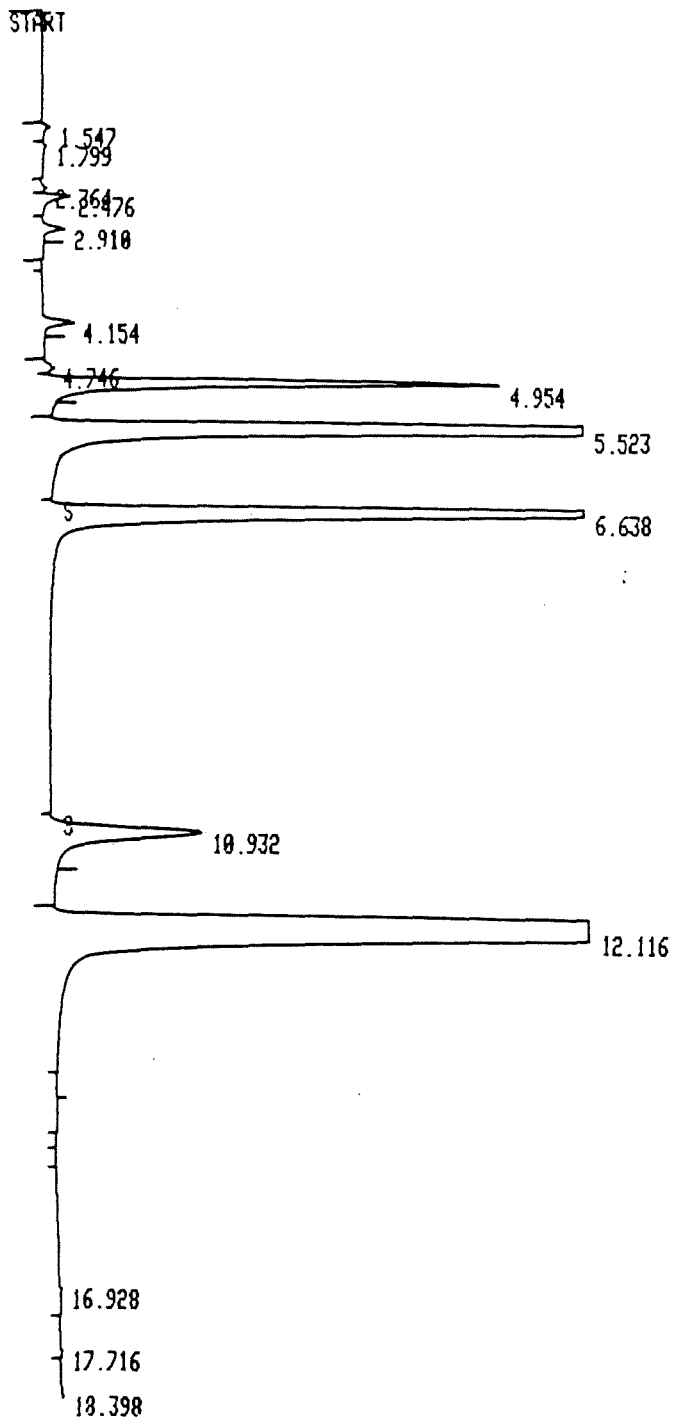
15. PREVENTATIVE MAINTENANCE

15.1 Preventative maintenance will be performed in accordance with instrument manufacturer's manuals. Expendable analytical components will be replaced as needed to attain analytical quality of the level previously described. Maintenance will be performed or other corrective

actions made when shifts in calibration, decreased sensitivity, or failure to meet QC criteria are observed. Instrument logbooks will contain a history of scheduled routine and nonroutine maintenance. Instrument downtime is minimized by having an inventory of expendable supplies and critical components.

CSL EXAMPLE #1: SAMPLE GAS CHROMATOGRAPH

WKFILE 3 @



RUN # 25
WORKFILE ID: A3
WORKFILE NAME:
SAMPLE # 3 1 ppm

ESTD

RT	AREA	TYPE	CAL #	AMOUNT
1.547	112690	BP		0.000
1.799	86703	PP		0.000
2.364	32360	PY		0.000
2.476	465940	YY		0.000
2.910	368900	YB		0.000
4.154	508830	PB	6R	0.931
4.746	105260	PP		0.000
4.954	9704300	PB	1	1.165
5.523	6.0173E+07	↑SPB	2	1.057
6.638	2.8504E+07	SPB	3	1.022
10.932	6013100	BB	4	0.572
12.116	1.3018E+08	↑SBH	5	1.014
16.928	104410	TPY		0.000
17.716	16165	TYP		0.000
18.398	10164	ITPB		0.000

TOTAL AREA= 2.3639E+08
MUL FACTOR= 1.0000E+00

RECALIBRATION
ESTD CALIB RUNS 1
REF % RTW= 5.00 % RTW= -0.10

CAL #	RT	AMT	AMT/AREA
1	4.95	1.1800E+00	1.2160E-07
2	5.53	1.1800E+00	1.9610E-08
3	6.63	1.1000E+00	3.8591E-08
4	10.92	1.0000E+00	1.6630E-07
5	12.14	1.0600E+00	8.1423E-09
6R	4.15	1.0000E+00	1.9653E-06

STOP

LIST: WKFILE EXAMPLE #2: GC OPERATING CONDITIONS

LAST EDITED: [illegible]

WORKFILE ID: 81
WORKFILE NAME:

MODEL #	ADDR	OBJECT	STATUS
3392A	0	I0P0	+
3392A	0	I1P0	-
3392A	0	C0C0	+
3392A	0	A0C0	-
3392A	0	S0C0	+
3392A	0	R0C0	-
3392A	0	B0C0	-

RUN PRMTRS
ZERO = 0
ATT 2↑ = 6
CHT SP = 1.0
PK WD = 0.04
THRSH = 1
AR REJ = 10000

RPRT OPTNS
2. RF UNC PKS= 0.0000E+00
3. MUL FACTOR= 1.0000E+00
4. PK HEIGHT MODE NO
5. EXTEND RT YES
6. RPRT UNC PKS YES

TIME TBL
EMPTY

CALIB TBL
ESTD CALIB RUNS 1
REF % RTW= 5.00 % RTW= 5.00

CAL #	RT	AMT	AMT/AREA
1	4.96	1.3000E-02	1.9056E-08
2	5.54	1.2000E-02	9.2421E-09
3	6.64	1.4000E-02	1.6031E-08
4	10.93	2.9000E-02	1.0484E-07
5	12.17	1.2000E-02	1.5634E-08
6	4.15	1.0000E+00	1.0000E+00

7. SUPPRESS RPRT NO
8. TRANSMIT RPRT NO
9. HDW RDY DELAY NO
10. AUTOMATE RUNS YES
11. SAMPLER PRMTRS
12. TRANSMIT PEAKS NO
13. TRANSMIT POINTS NO
14. SLICE WIDTH= 0.0000E+00
15. ID:
16. ISTD AMT=
SAMPLE AMT=
17. INSTR NETWORK CONFIG TBL
18. NEXT WORKFILE: 2
RUNS/SUBSEQ:
19. TIME SCALE NO
20. SAMPLE TBL

SAMPLE #	ISTD AMT	SAMPLE AMT	RCALB
1			1
2			1
3			1

MODEL #	ADDR		
7673A	2		
7673A	2	B0P0	-
7673A	2	S0P1	-
7673A	2	B0P1	-
7673A	2	K0C0	-
7673A	2	K1P0	-
7673A	2	K2C0	-
7673A	2	I0C0	+

REAR INJECTOR
INJ/BOTTLE 1
FIRST BOTTLE 1
LAST BOTTLE 1
OF SAMPLE WASHES 5
OF PUMPS 5
VISCOSITY 3
VOLUME 2
OF SOLVENT A WASHES 5
OF SOLVENT B WASHES 5
PRIORITY SAMPLE (1=YES) 0
CAPILLARY ON-COLUMN 0

MODEL #	ADDR	OBJECT	STATUS
5890A	1	C0P0	+
5890A	1	C0P1	-
5890A	1	C1P0	-
5890A	1	C1P1	-
5890A	1	K0C0	-

OVEN TEMP SETPT = 30
EQUIB TIME = 1.00 CRYO OFF
OVEN MAXIMUM = 300
INITIAL TEMP = 30
INITIAL TIME = 2.00

TEMP PRGM:	RATE	FINAL TEMP	FINAL TIME
	5.0	40	0.00
RAMP A	2.0	70	0.00
RAMP B	20.0	150	5.00

INJ A TEMP SETPT = 150 (OFF)
INJ B TEMP SETPT = 150
DET A TEMP SETPT = 220 (OFF)
DET B TEMP SETPT = 250

SIGNAL 1 = B
INET RANGED DATA ON
RANGE = 0
ZERO = 6.3
ATTN = 0

SIGNAL 2 = A
RANGE = 0
ZERO = 109.3
ATTN = 0

DETECTOR A = ON
DETECTOR B = ON

PURGE A = OFF
PURGE B = OFF

Attachment C-2
CLOSE SUPPORT LABORATORY METHOD FOR SCREENING ANALYSIS OF VOLATILE INDICATOR PARAMETERS IN WATER SAMPLES

CSL METHOD: VOA/WATER/PENTANE EXT/GC-ECD/FID

CLOSE SUPPORT LABORATORY METHOD FOR ANALYSIS OF SELECTED ORGANIC COMPOUNDS

1. SCOPE AND APPLICATION

1.1 This method is used for field screening of water samples for organic compounds that are indicative of contamination at the Onalaska Landfill site. It is presented as a means to rapidly characterize contamination in water samples. The method is semi-qualitative and semi-quantitative for the following target compounds:

Target Constituents

total-1,2-Dichloroethylene (DCE)
1,1,1-Trichloroethane (TCA)
Trichloroethylene (TCE)
Perchloroethylene (PCE)
Toluene
Xylene(s)

1.2 Application of this method is limited to the screening analysis of water for the target constituents. The chromatographic record produced in the analyses allows the site investigation team to examine the relative degree of water contamination associated with other nontargeted compounds in the sample extracts. Specifically, this method will be used to help locate source areas of contamination and to screen samples for selection and submittal to CLP laboratories. Positive identification and quantification of specific constituents, such as these constituents and other priority pollutants, will be supported by analyses of replicate and other composited samples at a remote CLP laboratory employing EPA approved testing protocols.

1.3 Preliminary method validation data indicates analyte recoveries of upwards of 80 percent.

1.4 The method detection limits (MDL) for the target constituents are estimated to be 10 ug/l for the chlorinated compounds and 50 ug/l for the aromatic target compounds. These estimates are the result of previous method development work. Interferences may affect method detection limits for each of the target compounds.

2. SUMMARY OF METHOD

2.1 The method presented here is loosely based on EPA Method 3550, *"Sonification Extraction,"* Method 8000, *"Gas Chromatography Analysis,"* found in EPA SW846, Test Methods for Evaluating Solid Waste, 3rd ed., November 1986. In brief, pentane is used in conjunction with sonification to effect extraction of the target parameters from the sample matrix. The extract is subsequently analyzed on a capillary gas chromatograph using both an electron capture detector for the chlorinated compounds and a flame ionization detector for the aromatic compounds.

3. SAFETY

3.1 The target constituents are either identified as or suspected of being carcinogens. All samples are assumed to be hazardous. All stock and working calibration standards, as well as all samples, shall be handled with the utmost care using good laboratory techniques in order to avoid harmful exposure.

3.2 Laboratory analysts shall wear lab coats, safety glasses, and surgical gloves at all times when preparing and handling standards and field and lab samples.

3.3 Standards and samples shall be prepared in a fume hood.

3.4 Pentane (C_5H_{12}) is regulated by NIOSH. The suggested permissible exposure level (PEL) is 120 ppm with a ceiling level of 610 ppm. Exposure pathways are oral, dermal, and airway. Effects of short-term exposure are drowsiness and irritation of eyes and nose, large doses may cause unconsciousness. Prolonged overexposure may cause irritation of skin. The odor threshold of n-pentane is reported as 2.2 ppm. Pentane is highly flammable and is incompatible with strong oxidizing agents.

3.5 Sample extracts and standards prepared in flammable solvents shall be stored in a refrigerator or in a cooler (outside the laboratory).

3.6 All of the target compounds are reported in the NIOSH manual as having good warning properties. Any situation which leads to or causes noticeable odors or produces any physical symptoms in the workers shall be investigated immediately followed by the appropriate corrective action.

3.7 The ultrasonic sonicator used for the sample extraction emits a high frequency sound. When in use, the sonicator horn shall be inside the sound chamber with the door closed.

3.8 Safety equipment including a fire extinguisher, first aid kit, eye wash, and chemical spill clean-up kit shall be available for use at all times.

3.9 Laboratory wastes shall be separated and properly disposed of. The wastes include: used sample aliquots, initial wash water, chemical wastes generated in the analysis, and disposables used in the preparation of the samples. These wastes shall be collected and deposited in a drum clearly marked as "CSL Lab Wastes Only--Hazardous." Water used for final rinsing of glassware will be considered nonhazardous and will be released into a 55 gallon drum. These wastes will then be disposed of in accordance with the appropriate and relevant disposal method.

4. SAMPLE SIZE REQUIREMENT AND SAMPLE COLLECTION

4.1 The purpose of water sampling is to obtain a representative portion of water that can be submitted for chemical analysis. In the case of surface water, samples will be taken with sample bottles as specified in the Field Sampling Plan.

4.2 Sampling equipment will be decontaminated after each sample has been collected. Please refer to the Field Sampling Plan for details outlining sample collection and decontamination procedures.

4.3 Samples will be kept cool at 4 degrees Celsius until CSL analysis.

4.4 The EPA holding time for water samples submitted for volatiles analysis is 10 days.

5. DETECTION LIMITS AND WORKING LINEAR RANGES

5.1 The method detection limit (MDL) for the target constituents is estimated to be 10 ug/l for the chlorinated compounds, and the working linear range for these compounds is estimated to be 10 ug/l to 1000 ug/l. The method detection limit for the aromatic target compounds is estimated to be 50 ug/l while the linear range for these compounds is estimated to be 50 ug/l to 2000 ug/l. These estimates are the result of previous method development work.

6. INTERFERENCE AND CORRECTIVE MEASUREMENTS

6.1 Samples containing compounds that co-elute with the target constituents may cause a positive bias in the results.

6.2 The presence of compounds that closely match the retention times of the target constituents may result in false identifications.

6.3 The MDLs for the target constituents may be suppressed by baseline noise associated with samples having high levels of background organics or other interferences.

6.4 The response factors for uncalibrated peaks that are significantly different than those of the target constituents may produce errors in the estimation of total target constituent contamination.

6.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for cross-contamination.

7. APPARATUS AND MATERIALS

7.1 Water sampling equipment--described in Field Sampling Plan.

7.2 VOA sample bottles--40-ml capacity with Teflon-lined caps.

7.3 Gas chromatograph (GC)--Hewlett-Packard Model 5890A; temperature programming, electronic integration, report annotation, automatic sampler, 30-meter capillary column, flame ionization detector, electron capture detector.

7.4 Glassware--Class A volumetric pipets and flasks; beakers, vials, Pasteur pipets, and miscellaneous glassware as necessary for preparation and handling of samples and standards.

7.5 Labware--necessary for preparation and handling of samples and standards.

7.6 Syringes--Hamilton glass type as required for injection of sample extracts and standards, preparation of dilutions, and spiking of samples.

7.7 Sonifier--Heat Systems Ultrasonic Sonicator with variable control up to 375 watt output and water-cooled cup horn.

8. REAGENTS

8.1 Pentane (C_5H_{12})--spectrophotometric grade.

8.2 Stock standards--prepare or purchase standard materials at approximately 1000 mg/l in methanol.

8.3 Working standards--prepared from stock standards by precise dilution in methanol for the chlorinated target compounds (10 ug/l, 100 ug/l, and 1000 ug/l); prepared for the aromatic compounds (50 ug/l, 500 ug/l, and 2000 ug/l).

8.4 Hydrogen--carrier gas, grade 5.

8.5 Zero Grade Air (<0.1 ppm hydrocarbons)

8.6 Nitrogen makeup gas, prepurified grade.

8.7 Distilled water--adjusted to pH 12 with 5N NaOH.

9. CALIBRATION PROCEDURES

9.1 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels (10 ug/l, 100 ug/l, and 1000 ug/l) for the chlorinated target compounds and 50 ug/l, 500 ug/l, and 2000 ug/l for the aromatic compounds) by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with pentane.

9.2 Inject each calibration standard mix used into the gas chromatograph (i.e. 2-ul injections). Tabulate response factors for each calibration compound for each run. The units for the response factor are based on concentration per area.

Response factor = Std. Conc./Area for Std.

Where:

Std. Conc. = The concentration of the prepared standard (e.g. 1 ug/ml toluene)

Area for Std. = Integrated peak area as reported by the GC integrator (e.g area for toluene)

9.3 Sample concentrations of unknowns can be calculated directly **if** the response factor is the same for every compound at each calibration level. If not, the least squares line must be found using the calibration data, and sample concentrations for unknowns will be calculated using the least squares equation. In either case, the results must be used to prepare a calibration curve for each analyte.

9.4 The working calibration curve must be verified on each working day by the injection of the mid-range calibration standard.

10. SAMPLE PREPARATION

10.1 In a labeled VOA vial, add an aliquot of sample water (approximately 5 milliliters to the vial) and record the volume in the lab notebook. Volumetrically pipet 5.0 ml of the pentane solvent into the sample vial.

10.2 With the VOA vial cap tightly in place, sonicate at an output setting of 30 percent for approximately 5 minutes. The resultant sonified sample should be dispersed throughout the pentane solvent.

10.3 After sonification, let the VOA vial stand. Using a pastuer pipet, transfer a suitable aliquot of the pentane solvent (extract) from the vial into a labeled GC autosampler vial and cap immediately with septum crimp seals. Refrigerate the sample extracts until analyzed.

10.4 If the extract is noticeably colored, turbid, or otherwise indicates a dirty sample, it may be advantageous to do some cleanup by adding approximately 10 ml of pH 12 water and shaking vigorously. This will remove phenolic compounds and result in a cleaner extract. If the sample is obviously contaminated and past experience has shown similar extracts to contain high levels of contamination, it is recommended that a preliminary dilution be made to avoid grossly contaminating the gas chromatograph.

11. ANALYTICAL PROCEDURE

11.1 Perform GC analysis on the extract using instrument conditions similar to those listed in Exhibit 1.

11.2 If the analysis indicates that the results are more than 50 percent above the calibration range, dilute the sample extract so that concentrations fall within the calibration range.

11.3 Check the retention values for each of the target constituents against the expected (calibration) value. Reject those results where the retention time does not fall within +/- 0.15 minute of the expected values.

11.4 Use the retention time marker as an indicator of the reliability of each sample injection and GC run. The retention time marker should fall within the same windows as the target constituents and should be within +/- 15 percent area counts of the initial calibration value. If these criteria are not met, re-evaluate the data using relative retention times. Reruns should occur to resolve data suspicions.

11.5 If a grossly contaminated sample is injected, it may be necessary to run a

solvent blank several times afterward to clean residual contamination from the injection port and column. If this does not work, and blank runs still contain contamination, it will be necessary to change the injection port liner. Following the change of the liner, several blank runs will be needed to obtain a stable baseline.

12. DETAILS OF CALCULATIONS

12.1 Quantification of the target compounds is based on the integrated areas of the samples in comparison to the integrated areas of the calibration standards for each analysis. The integrator reports the concentrations in ug/ml in the extracts. Calculation of the concentration for each target constituent in the original sample on an as-received basis is as follows:

$$\text{Conc. in ug/kg} = \frac{A \times Vt \times DF}{Vs} \times 1{,}000$$

Where:

A = Amount of target constituent found in the extract in ug/gram

Vt = Volume of solvent added to the VOA vial, 5.0 ml

DF = Dilution factor, if required

1,000 = Dimensional correction factor

Vs = Volume of the sample added to the VOA vial in milliliters

13. QUALITY CONTROL REQUIREMENTS

13.1 Daily mid-range calibration checks performed prior to the analysis of each day's lot of samples or with each lot of 20 samples, whichever is more frequent.

13.2 Analysis of field blank samples at a frequency of 1 in 20 samples analyzed or 1/day, whichever is more frequent.

13.3 Analysis of laboratory blank samples at the same frequency. Should the results of the laboratory blanks show contamination, the cause of the contamination should be investigated and corrective action be taken.

13.4 Analysis of field replicate samples at a frequency of 1 in 20 or 1/day whichever is more frequent.

13.5 Analysis of a mid-range matrix spike samples and a matrix spike duplicate at a frequency of 1 in 20 samples analyzed or 1/day, whichever is more frequent.

13.6 Use of a retention time marker during the analysis of all samples and standards.

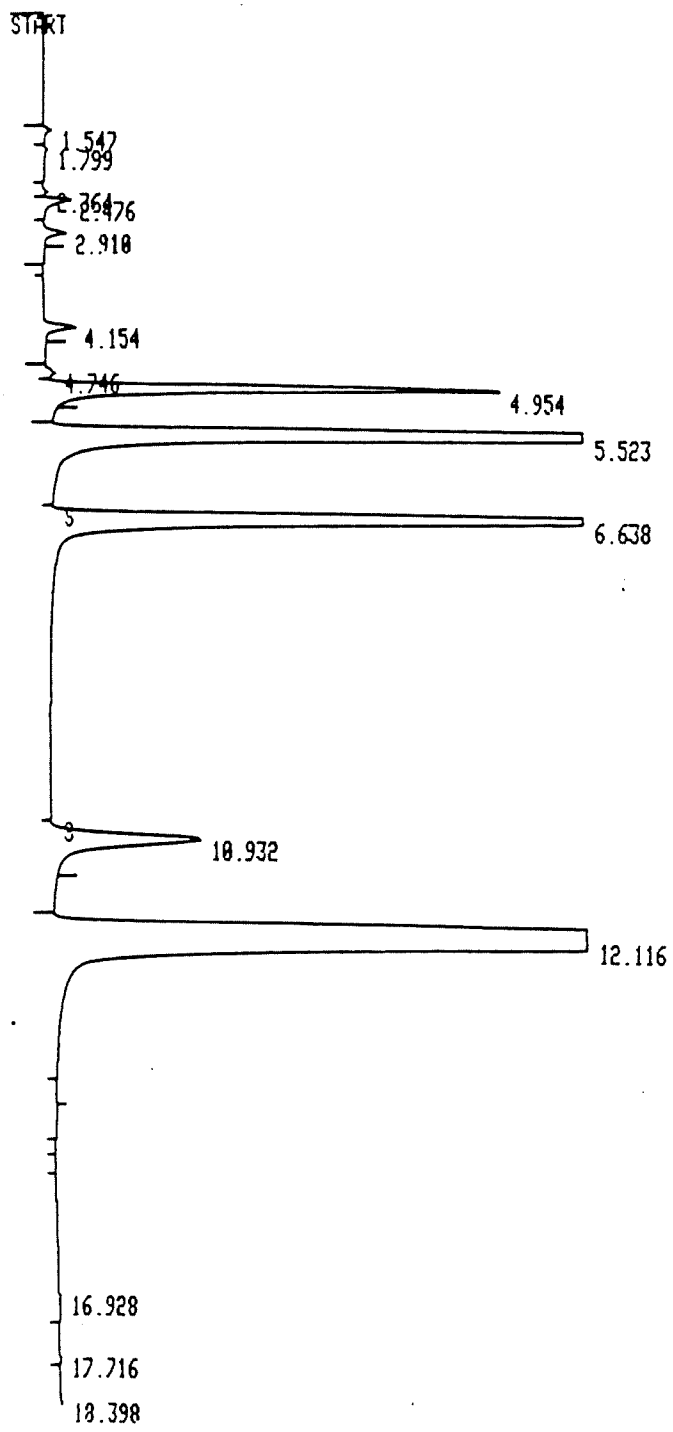
14. REPORT REQUIREMENTS

14.1 On completion of each day's batch of samples, the data will be reported to the Field Task Leader. This will take the form of copies of the pages from the Analysis Data Book and a summary table generated by a database program. The Field Task Leader will be responsible for disseminating the data to others as appropriate. At this point the data will not have passed the data validation process as described in the CSL QA document and will be considered preliminary. Ultimately the CSL data will be archived along with other project records.

15. PREVENTATIVE MAINTENANCE

15.1 Preventative maintenance will be performed in accordance with instrument manufacturer's manuals. Expendable analytical components will be replaced as needed to attain analytical quality of the level previously described. Maintenance will be performed or other corrective actions made when shifts in calibration, decreased sensitivity, or failure to meet QC criteria are observed. Instrument logbooks will contain a history of scheduled routine and nonroutine maintenance. Instrument downtime is minimized by having an inventory of expendable supplies and critical components.

Exhibit 1 (sheet 1 of 2) SAMPLE GAS CHROMATOGRAPH



RUN # 25
WORKFILE ID: A3
WORKFILE NAME:
SAMPLE # 3 1 ppm

ESTD

RT	AREA	TYPE	CAL #	AMOUNT
1.547	112690	BP		0.000
1.799	86703	PP		0.000
2.364	32360	PV		0.000
2.476	465940	VV		0.000
2.910	368900	VB		0.000
4.154	508830	PB	6R	0.931
4.746	105260	PP		0.000
4.954	9704300	PB	1	1.165
5.523	6.0173E+07	↑SPB	2	1.057
6.638	2.8504E+07	SPB	3	1.022
10.932	6013100	BB	4	0.572
12.116	1.3018E+08	↑SBH	5	1.014
16.928	104410	TPV		0.000
17.716	16165	TVP		0.000
18.398	10164	ITPB		0.000

TOTAL AREA= 2.3639E+08
MUL FACTOR= 1.0000E+00

RECALIBRATION
ESTD CALIB RUNS 1
REF % RTW= 5.00 % RTW= -0.10

CAL #	RT	AMT	AMT/AREA
1	4.95	1.1300E+00	1.2160E-07
2	5.53	1.1800E+00	1.9610E-08
3	6.63	1.1000E+00	3.8591E-08
4	10.92	1.0000E+00	1.6630E-07
5	12.14	1.0600E+00	8.1423E-09
6R	4.15	1.0000E+00	1.9653E-06

STOP

Exhibit 1 (sheet 2 of 2)
GC OPERATING CONDITIONS

```
LIST: WKFILE
LAST EDITED:

WORKFILE ID: B1
WORKFILE NAME:


MODEL #   ADDR   OBJECT   STATUS
3392A       0     I0P0       +
3392A       0     I1P0       -
3392A       0     C0C0       +
3392A       0     A0C0       -
3392A       0     S0C0       +
3392A       0     R0C0       -
3392A       0     B0C0       -

RUN PRMTRS
ZERO   =  0
ATT 2↑ =  6
CHT SP =  1.0
PK WD  =  0.04
THRSH  =  1
AR REJ =  10000

RPRT OPTNS
  2. RF UNC PKS=       0.0000E+00
  3. MUL FACTOR=       1.0000E+00
  4. PK HEIGHT MODE       NO
  5. EXTEND RT           YES
  6. RPRT UNC PKS        YES

TIME TBL
EMPTY

/CALIB TBL
 ESTD                  CALIB RUNS  1
 REF % RTW=   5.00     % RTW=   5.00

 CAL #      RT          AMT        AMT/AREA
   1       4.96     1.3000E-02   1.9056E-08
   2       5.54     1.2000E-02   9.2421E-09
   3       6.64     1.4000E-02   1.6031E-08
   4      10.93     2.9000E-02   1.0484E-07
   5      12.17     1.2000E-02   1.5634E-08
   6       4.15     1.0000E+00   1.0000E+00

   7. SUPPRESS RPRT        NO
   8. TRANSMIT RPRT        NO
   9. HDW RDY DELAY        NO
  10. AUTOMATE RUNS       YES
  11. SAMPLER PRMTRS
  12. TRANSMIT PEAKS       NO
  13. TRANSMIT POINTS      NO
  14. SLICE WIDTH=      0.0000E+00
  15. ID:
  16. ISTD AMT=
      SAMPLE AMT=
  17. INSTR NETWORK CONFIG TBL
  18. NEXT WORKFILE:    2
      RUNS/SUBSEQ:
  19. TIME SCALE           NO
  20. SAMPLE TBL

 SAMPLE #    ISTD AMT     SAMPLE AMT    RCALB
    1                                     1
    2                                     1
    3                                     1
```

```
MODEL #   ADDR
7673A       2
7673A       2      B0P0        -
7673A       2      S0P1        -
7673A       2      B0P1        -
7673A       2      K0C0        -
7673A       2      K1P0        -
7673A       2      K2C0        -
7673A       2      I0C0        +


 REAR INJECTOR
  INJ/BOTTLE                  1
  FIRST BOTTLE                1
  LAST BOTTLE                 1
  # OF SAMPLE WASHES          5
  # OF PUMPS                  5
  VISCOSITY                   3
  VOLUME                      2
  # OF SOLVENT A WASHES       5
  # OF SOLVENT B WASHES       5
  PRIORITY SAMPLE (1=YES)     0
  CAPILLARY ON-COLUMN         0

MODEL #   ADDR   OBJECT   STATUS
5890A       1     C0P0       +
5890A       1     C0P1       -
5890A       1     C1P0       -
5890A       1     C1P1       -
5890A       1     K0C0       -

 OVEN TEMP  SETPT =  30
  EQUIB TIME   = 1.00          CRYO OFF
  OVEN MAXIMUM = 300
  INITIAL TEMP =  30
  INITIAL TIME = 2.00

 TEMP PRGM: RATE   FINAL TEMP   FINAL TIME
             5.0        40          0.00
  RAMP A     2.0        70          0.00
  RAMP B    20.0       150          5.00

INJ A TEMP SETPT = 150  (OFF)
INJ B TEMP SETPT = 150
DET A TEMP SETPT = 220  (OFF)
DET B TEMP SETPT = 250

SIGNAL 1 = B
 INET RANGED DATA ON
 RANGE = 0
 ZERO  = 6.3
 ATTN  = 0

SIGNAL 2 = A
 RANGE = 0
 ZERO  = 109.3
 ATTN  = 0

DETECTOR A = ON
DETECTOR B = ON

PURGE A  = OFF
PURGE B  = OFF
```

Attachment C-3
CLOSE SUPPORT LABORATORY METHOD FOR SCREENING ANALYSIS OF VOLATILE INDICATOR PARAMETERS IN SOIL GAS SAMPLES

GLT809/55-4

NOTE

CH2M HILL will be performing method development as part of the Close Support Laboratory soil gas analysis. SW846 Method 5040 will serve as the framework for the CSL method excluding the purging device and the mass spectrometer.

GLT824/67

METHOD 5040

PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES FROM VOLATILE ORGANIC SAMPLING TRAIN

1.0 SCOPE AND APPLICATION

1.1 Method 5040 was formerly Method 3720 in the Second Edition of this manual.

1.2 This method covers the determination of volatile principal organic hazardous constituents (POHCs), collected on Tenax and Tenax/charcoal sorbent cartridges using a volatile organic sampling train, VOST (1). Much of the description for purge-and-trap GC/MS analysis is described in Method 8240 of this chapter. Because the majority of gas streams sampled using VOST will contain a high concentration of water, the analytical method is based on the quantitative thermal desorption of volatile POHCs from the Tenax and Tenax/charcoal traps and analysis by purge-and-trap GC/MS. For the purposes of definition, volatile POHCs are those POHCs with boiling points less than 100°C.

1.3 This method is applicable to the analysis of Tenax and Tenax/charcoal cartridges used to collect volatile POHCs from wet stack gas effluents from hazardous waste incinerators.

1.4 The sensitivity of the analytical method for a particular volatile POHC depends on the level of interferences and the presence of detectable levels of volatile POHCs in blanks. The desired target detection limit of the analytical method is 0.1 ng/L (20 ng on a single pair of traps) for a particular volatile POHC desorbed from either a single pair of Tenax and Tenax/charcoal cartridges or by thermal desorption of up to six pairs of traps onto a single pair of Tenax and Tenax/charcoal traps. The resulting single pair of traps is then thermally desorbed and analyzed by purge-and-trap GC/MS.

1.5 This method is recommended for use only by experienced mass spectroscopists or under the close supervision of such qualified persons.

2.0 SUMMARY OF METHOD

2.1 A schematic diagram of the analytical system is shown in Figure 1. The contents of the sorbent cartridges are spiked with an internal standard and thermally desorbed for 10 min at 180°C with organic-free nitrogen or helium gas (at a flow rate of 40 mL/min), bubbled through 5 mL of organic-free water, and trapped on an analytical adsorbent trap. After the 10-min desorption, the analytical adsorbent trap is rapidly heated to 180°C, with the carrier gas flow reversed so that the effluent flow from the analytical trap is directed into the GC/MS. The volatile POHCs are separated by temperature-programmed gas chromatography and detected by low-resolution mass spectrometry. The concentrations of volatile POHCs are calculated using the internal standard technique.

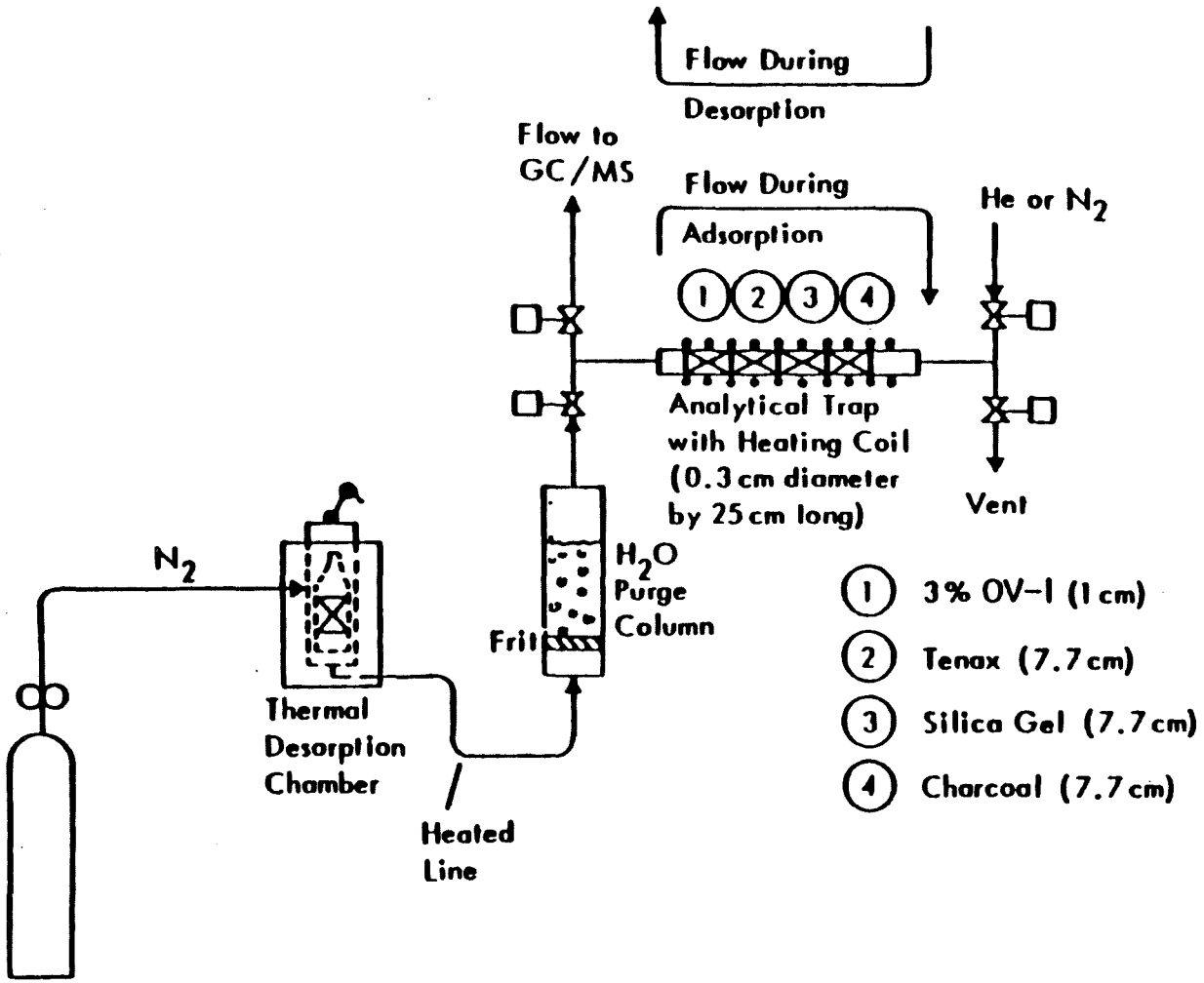


Figure 1. Schematic diagram of trap desorption/analysis system.

3.0 INTERFERENCES

3.1 Refer to Methods 3500 and 8240.

4.0 APPARATUS AND MATERIALS

4.1 Thermal desorption unit:

4.1.1 The thermal desorption unit (for Inside/Inside VOST cartridges, use Supelco "clamshell" heater; for Inside/Outside VOST cartridges, user fabricated unit is required) should be capable of thermally desorbing the sorbent resin tubes. It should also be capable of heating the tubes to 180 ± 10°C with flow of organic-free nitrogen or helium through the tubes.

4.2 Purge-and-trap unit:

4.2.1 The purge-and-trap unit consists of three separate pieces of equipment: the sample purger, trap, and the desorber. It should be capable of meeting all requirements of Method 5030 for analysis of purgeable organic compounds from water.

4.3 GC/MS system: As described in Method 8240.

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 parameters of interest.

5.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 450 g of activated carbon (Calgon Corporation, 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 distilled water for 15 min. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hr. Allow the water to cool to room temperature while continuing to bubble the inert gas through the water. This water should be transferred directly to the purge-and-trap apparatus for use.

5.1.4 Other methods that can be shown to produce organic-free water can be used.

5.2 Analytical trap reagents:

5.2.1 **2,6-Diphenylene oxide polymer:** Tenax (60/80 mesh), chromatographic grade or equivalent.

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

5.2.3 **Silica gel:** Davison Chemical (35/00 mesh), Grade 15, or equivalent.

5.2.4 **Charcoal:** Petroleum-based (SKC Lot 104 or equivalent).

5.3 Stock standard solution:

5.3.1 Stock standard solutions will be prepared from pure standard materials or purchased as certified solutions. The stock standards should be prepared in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA-approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

5.3.2 Fresh stock standards should be prepared weekly for volatile POHCs with boiling points of <35°C. All other standards must be replaced monthly, or sooner if comparison with check standards indicates a problem.

5.4 Secondary dilution standards:

5.4.1 Using stock standard solutions, prepare in methanol secondary dilution standards that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the desorbed calibration standards will bracket the working range of the analytical system.

5.5 4-Bromofluorobenzene (BFB) standard:

5.5.1 Prepare a 25 ng/uL solution of BFB in methanol.

5.6 Deuterated benzene:

5.6.1 Prepare a 25 ng/uL solution of benzene-d_6 in methanol.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to Method 0030, Chapter Ten.

6.2 Sample trains obtained from the VOST should be analyzed within 2-6 weeks of sample collection.

7.0 PROCEDURE

7.1 Assembly of PTD device:

7.1.1 Assemble a purge-and-trap desorption device (PTD) that meets all the requirements of Method 5030 (refer to Figure 1).

7.1.2 Connect the thermal desorption device to the PTD device. Calibrate the PTD-GC/MS system using the internal standard technique.

7.2 Internal standard calibration procedure:

7.2.1 This approach requires the use of deuterated benzene as the internal standard for these analyses. Other internal standards may be proposed for use in certain situations. The important criteria for choosing a particular compound as an internal standard are that it be similar in analytical behavior to the compounds of interest and that it can be demonstrated that the measurement of the internal standard be unaffected by method or matrix interferences. Other internal standards that have been used are d_{10}-ethylbenzene and d_4-1,2-dichloroethane. One adds 50 ng of BFB to all sorbent cartridges (in addition to one or more internal standards) to provide continuous monitoring of the GC/MS performance relative to BFB.

7.2.2 Prepare calibration standards at a minimum of three concentration levels for each analyte of interest.

7.2.3 The calibration standards are prepared by spiking a blank Tenax or Tenax/charcoal trap with a methanolic solution of the calibration standards (including 50 ng of the internal standard, such as deuterated benzene), using the flash evaporation technique. The flash evaporation technique requires filling the needle of a 5.0-uL syringe with clean methanol and drawing air into the syringe to the 1.0-uL mark. This is followed by drawing a methanolic solution of the calibration standards (containing 25 ug/uL of the internal standard) to the 2.0-uL mark. The glass traps should be attached to the injection port of a gas chromatograph while maintaining the injector temperature at 160°C. The carrier gas flow through the traps should be maintained at about 50 mL/min.

7.2.4 After directing the gas flow through the trap, the contents of the syringe should be slowly expelled through the gas chromatograph injection port over about 15 sec. After 25 sec have elapsed, the gas flow through the trap should be shut off, the syringe removed, and the trap analyzed by the PTD-GC/MS procedure outlined in Method 8240. The total flow of gas through the traps during addition of calibration standards to blank cartridges, or internal standards to sample cartridges, should be 25 mL or less.

7.2.5 Analyze each calibration standard for both Tenax and Tenax/charcoal cartridges according to Section 7.3. Tabulate the area response

of the characteristic ions of each analyte against the concentration of the internal standard and calculate response factor (RF) for each compound, using Equation 1.

$$RF = A_S C_{is} / A_{is} C_S \quad (1)$$

where:

A_S = Area of the characteristic ion for the analyte to be measured.

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

C_{is} = Amount (ng) of the internal standard.

C_S = Amount (ng) of the volatile POHC in calibration standard.

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

7.2.6 The working calibration curve or RF must be verified on each working day by the measurement of one or more of the calibration standards. If the response varies by more than ±25% for any analyte, a new calibration standard must be prepared and analyzed, for that analyte.

7.3 The schematic of the PTD-GC/MS system is shown in Figure 1. The sample cartridge is placed in the thermal desorption apparatus (for Inside/Inside VOST cartridges, use Supelco "clamshell" heater; for Inside/Outside VOST cartridges, user fabricated unit is required) and desorbed in the purge-and-trap system by heating to 180°C for 10 min at a flow rate of 40 mL/min. The desorbed components pass into the bottom of the water column, are purged from the water, and collected on the analytical adsorbent trap. After the 10-min desorption period, the compounds are desorbed from the analytical adsorbent trap into the GC/MS system according to the procedures described in Method 8240.

7.4 Qualitative identification:

7.4.1 The procedure for qualitative identification of volatile POHCs using this protocol is described in Method 8240.

7.5 Calculations:

7.5.1 When an analyte has been qualitatively identified, quantification should be based on the integrated abundance from the EICP of the primary characteristic ion chosen for that analyte. If the sample produces an interference for the primary characteristic ion, a secondary characteristic ion should be used.

7.5.1.1 Using the internal standard calibration procedure, the amount of analyte in the sample cartridge is calculated using the response factor (RF) determined in Paragraph 7.2.5 and Equation 2.

$$\text{Amount of POHC} = A_s C_{is} / A_{is} RF \quad (2)$$

where:

A_s = Area of the characteristic ion for the analyte to be measured.

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

C_{is} = Amount (ng) of internal standard.

7.5.1.2 The choice of methods for evaluating data collected using VOST for incinerator trial burns is a regulatory decision. The procedures used extensively by one user are outlined below.

7.5.1.3 The total amount of the POHCs of interest collected on a pair of traps should be summed. These values should then be blank corrected. Guidelines for blank correction of sample cartridges are outlined below.

7.5.1.3.1 After all blanks (field and trip) are analyzed, a paired t-test should be used to determine whether trip blanks are significantly different from field blanks. If no difference is found, then the mean and standard deviation of the combined field and trip blanks for each POHC of interest is calculated.

7.5.1.3.2 If, when using the paired t-test, the field and trip blanks are determined to be different, then the field blank (or the mean of multiple field blanks) associated with a particular run should be used as the blank value for that particular run.

7.5.1.4 Next, for each sample/POHC combination, a determination must be made as to whether a particular sample is significantly different from the associated blank. If the mean of the trip and field blanks is used, then a sample is different from the blank if:

$$(\text{measured sample value}) - (\text{mean blank value}) > (3 \times \text{blank standard deviation})$$

(If an individual field blank is used as the blank value, the above criteria do not apply.) If the sample is determined to be different from the blank according to the above criteria, then the emission value of a particular POHC is blank-corrected by subtracting the mean blank value from the measured sample value.

7.5.1.5 If, according to the above procedures, the sample cannot be distinguished from the blank (i.e., for a given POHC there is a high sample value and high blank value or there is a low sample value and low blank value), the measured sample value is not blank-corrected. In this case, the measured sample value is used to calculate a maximum emission value (and therefore a minimum DRE value) for that particular run.

7.5.1.6 The observation of high concentrations of POHCs of interest in blank cartridges indicates possible residual contamination of the sorbent cartridges prior to shipment to and use at the site. Data that fall in this category (especially data indicating high concentrations of POHCs in blank sorbent cartridges) should be qualified with regard to validity, and blank data should be reported separately. The applicability of data of this type to the determination of DRE is a regulatory decision. Continued observation of high concentrations of POHCs in blank sorbent cartridges indicates that procedures for cleanup, monitoring, shipment, and storage of sorbent cartridges by a particular user be investigated to eliminate this problem.

7.5.1.7 If any internal standard recoveries fall outside the control limits established in Section 8.4, data for all analytes determined for that cartridge(s) must be qualified with the observation.

8.0 QUALITY CONTROL

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

8.2 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of blank Tenax and Tenax/charcoal cartridges spiked with the analytes of interest. The laboratory is required to maintain performance records to define the quality of data that are generated. Ongoing performance checks must be compared with established performance criteria to determine if results are within the expected precision and accuracy limits of the method.

8.2.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable precision and accuracy with this method. This ability is established as described in Paragraph 7.2.

8.2.2 The laboratory must spike all Tenax and Tenax/charcoal cartridges with the internal standard(s) to monitor continuing laboratory performance. This procedure is described in Paragraph 7.2.

8.3 To establish the ability to generate acceptable accuracy and precision, the analyst must spike blank Tenax and Tenax/charcoal cartridges with the analytes of interest at two concentrations in the working range.

8.3.1 The average response factor (R) and the standard deviation (S) for each must be calculated.

8.3.2 The average recovery and standard deviation must fall within the expected range for determination of volatile POHCs using this method. The expected range for recovery of volatile POHCs using this method is 50-150%.

8.4 The analyst must calculate method performance criteria for the internal standard(s).

8.4.1 Calculate upper and lower control limits for method performances using the average area response (A) and standard deviation(s) for internal standard:

Upper Control Limit (UCL) = A + 3S.
Lower Control Limit (LCL) = A - 3S.

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

8.5 The laboratory is required to spike all sample cartridges (Tenax and Tenax/charcoal) with internal standard.

8.6 Each day, the analyst must demonstrate through analysis of blank Tenax and Tenax/charcoal cartridges and reagent water that interferences from the analytical system are under control.

8.7 The daily GC/MS performance tests required for this method are described in Method 8240.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

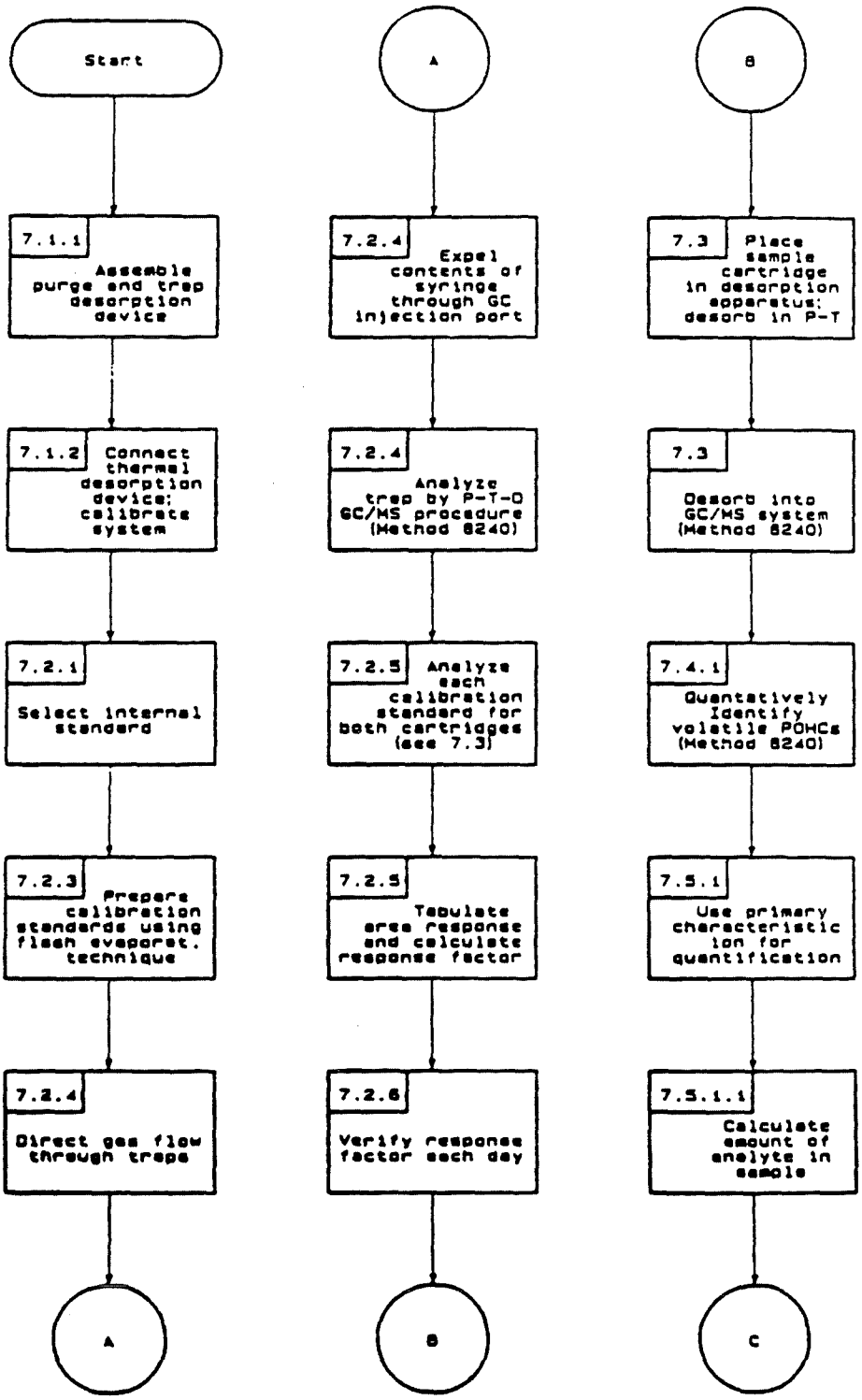
1. Protocol for Collection and Analysis of Volatile POHC's Using VOST. EPA/600/8-84-007, March 1984.

2. Validation of the Volatile Organic Sampling Train (VOST) Protocol. Volumes I and II. EPA/600/4-86-014a, January 1986.

METHOD 5040

PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES

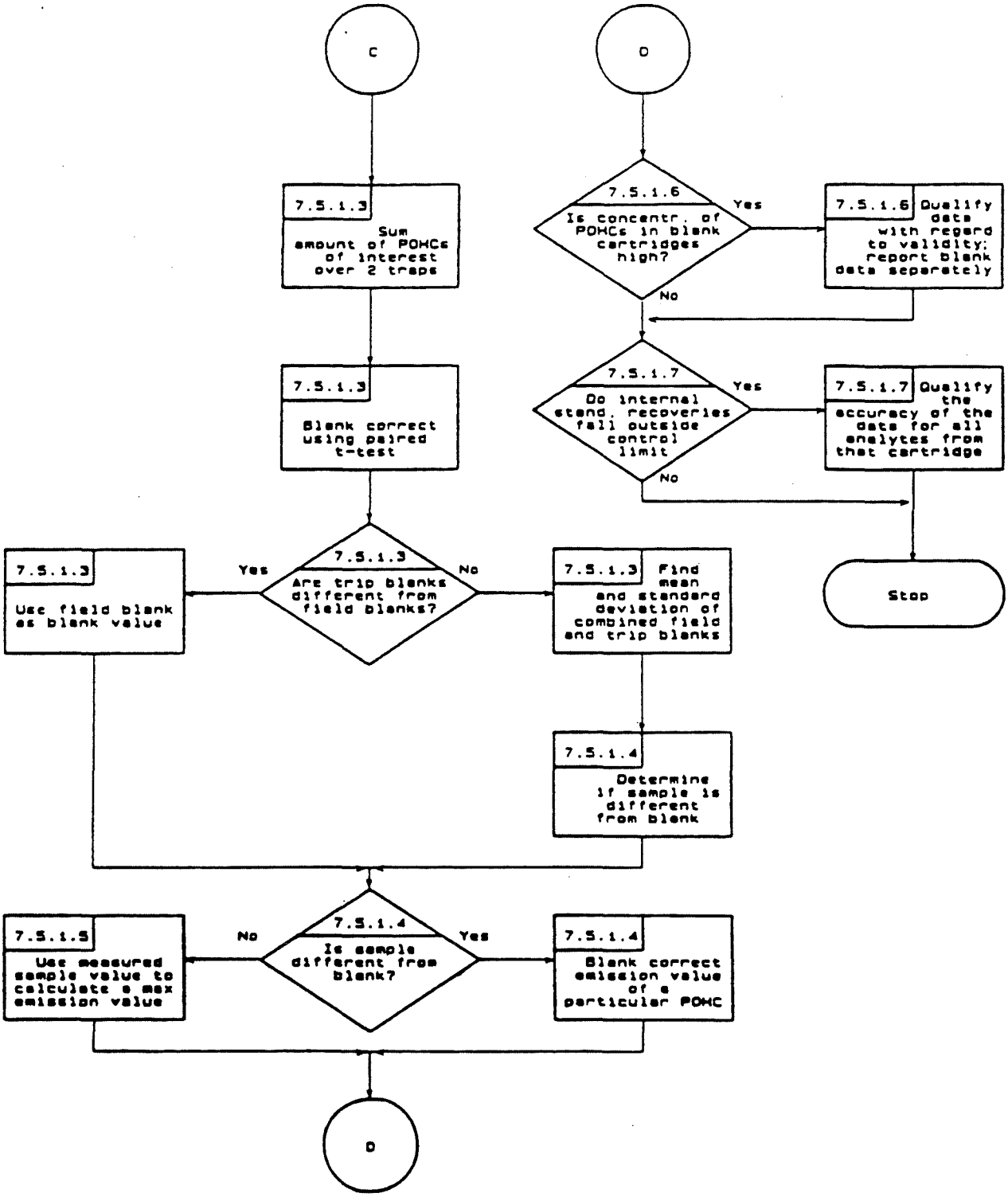
FROM VOLATILE ORGANIC SAMPLING TRAIN



METHOD 5040

PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES

FROM VOLATILE ORGANIC SAMPLING TRAIN
(Continued)



Appendix D
FIELD TESTING PROCEDURES

FIELD MEASUREMENT OF SPECIFIC CONDUCTIVITY AND TEMPERATURE

METHOD: Specific Conductivity

REFERENCE: Methods for Chemical Analysis of Water and Wastes, U.S. EPA Method 120.1, 1983

DETECTION LIMIT: 1 umho/cm @ 25°C

RANGE: 0.1 to 100,000 umho/cm

SAMPLE HOLDING TIME: Determine onsite or within 24 hours

REAGENTS: Distilled water in squirt bottle and standard potassium chloride solution

APPARATUS: Conductivity meter and electrode

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.

PROCEDURE

1. With mode switch at OFF position, check meter zero. If not zeroed, use meter screw to adjust to zero.

2. Plug probe into jack on side of meter.

3. Turn mode switch to red line and turn red line knob until needle aligns with red line on dial. If they cannot be aligned, change the batteries.

4. Immerse probe in 0.01 N standard potassium chloride solution. Do not allow the probe to touch the sample container.

5. Set the mode control to TEMPERATURE. Record the temperature on the bottom scale of the meter in degrees Celsius.

6. Turn MODE switch to appropriate conductivity scale, (i.e., X100, X10, or X1). Use a scale that will give a mid-range output on the meter.

7. Wait for needle to stabilize (about 15 seconds), multiply reading by scale setting, and record the conductivity. The conductivity reading must then be corrected for temperature.

   Calculate conductivity using the formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

where:

G_{25} = conductivity at 25°C, umho/cm

T = temperature of sample, °C

G_T = conductivity of sample at temperature T, umho/cm

Table 1 lists the values of conductivity this solution would have if the distilled water were nonconductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

8. Report results for the standard solution with each data set.

9. Record the type of meter and probe used on field sheet.

10. Rinse probe with deionized water.

11. Wipe meter clean as necessary.

12. Repeat steps 5 through 11 for water samples.

13. After returning to laboratory, compare results with previous data. Report problems to laboratory personnel.

Table 1
CONDUCTIVITY METER CALIBRATION TABLE

Temperature (°C)	Conductivity (umho/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

ACCURACY AND PRECISION

Accuracy and precision are dependent on the instrument used. Refer to the manufacturer's manual.

For the YSI Model 33 conductivity meter (per operating manual):

Conductivity

Expected accuracy ±2.5 percent at 500, 5,000, and 50,000 plus ±2 percent for probe.

Expected accuracy ±3 percent at 250, 2,500, and 25,000 plus ±2 percent for probe.

Temperature

Expected accuracy ±0.1°C at 2°C plus probe
±0.6°C at 45°C plus probe

Probe ±0.1°C at 0°C
±0.3°C at 40°C

PREVENTATIVE MAINTENANCE

Refer to manufacturer's manual.

FIELD MEASUREMENT OF pH

METHOD: Electrometric

REFERENCE: Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Method 150.1, 1983

SENSITIVITY: 0.1 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

APPARATUS

- pH meter
- Combination electrodes
- Beakers
- Glassware that has been washed with soap and water, rinsed twice with hot water, and rinsed twice with deionized water

CALIBRATION

1. Place electrode in pH 7 buffer solution.
2. Allow meter to stabilize and then turn calibration dial until a reading of 7.0 is obtained.
3. Rinse electrode with deionized water and place it in a pH 4 or pH 10 buffer solution.
4. Allow meter to stabilize again and then turn slope adjustment dial until a reading of 4.0 is obtained for the pH 4 buffer solution or 10.0 for the 10 pH solution.

5. Rinse electrode with deionized water and place in pH 7 buffer. If meter reading is not 7.0, repeat Steps 2 to 5.

PROCEDURE

1. Before going into the field:

   a) Check batteries.

   b) Do a quick calibration at pH 7 and 4 to check electrode.

   c) Obtain fresh solutions.

2. Calibrate meter using calibration procedure.

3. Pour the sample into a clean beaker.

4. Rinse electrode with deionized water between samples.

5. Immerse electrode in solution. Make sure the white KCl junction on side of electrode is in the solution. The level of electrode solution should be one inch above sample to be measured.

6. Recheck calibration with pH 7 buffer solution after every five samples.

When calibrating the meter, use pH buffers 4 and 7 for samples with pH < 8, and buffers 7 and 10 for samples with pH > 8. If meter will not read pH 4 or 10, something may be wrong with the electrode. Return it to the laboratory and explain the malfunction.

Measurement of pH is temperature dependent. Therefore, temperatures of buffers and samples should be within about 2°C. For refrigerated or cool samples, use refrigerated buffers to calibrate the pH meter.

Weak organic and inorganic salts and oil and grease interfere with pH measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water and rinse with a 10 percent solution of HCl. Then recalibrate meter.

Following field measurements:

a) Report any problems.
b) Compare with previous data.
c) Clean all dirt off meter and inside case.
d) Store electrode in pH 4 buffer.

ACCURACY AND PRECISION

Accuracy and precision are dependent on the instrument used; refer to manufacturer's manual

Expected accuracy and precision are ±0.1 pH unit.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's manual.

HNu MONITORING

OPERATION

For complete operating instructions, refer to the manufacturer's instruction manual.

CALIBRATION

By analyzing a gas of known concentration, the HNu is easily calibrated. Benzene is typically used as the calibration gas. When calibrating the HNu, always remember to:

- Calibrate in the range to be tested.
- Deliver the calibration gas at ambient temperature and pressure. Handle gas cylinders with care.
- Calibrate every day.

The calibration gas must be stable during the period of use and must be at a concentration that reflects field sample concentrations. All gas cylinders must have proper regulators.

CALIBRATION PROCEDURE

To calibrate an HNu:

1. Identify the probe by lamp label.
2. Connect the probe.
3. Affirm the ionization potential of the calibration gas.
4. Perform a battery check.
5. Zero the HNu.
6. Sample calibration gas and adjust to proper reading.
7. Repeat steps 5 and 6.
8. If calibration cannot be achieved, the lamp must be cleaned.
9. Replace lamp if the lamp output is too low or if the lamp has failed.

To obtain more than a two-point calibration, dilute the calibration gas to known concentrations and take additional readings.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's instruction manual.

OVA MONITORING

OPERATION

For complete operating instructions, refer to the manufacturer's instruction manual.

CALIBRATION

By analyzing a gas of known concentration, the OVA is easily calibrated. Methane in air 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.

CALIBRATION PROCEDURE

To calibrate an OVA in the field:

1. Zero the instrument.
2. Sample the calibration mixture and adjust to proper reading. Handle gas cylinders with care.
3. Next, set the calibration switch to a different range.
4. Sample another calibration mixture of different concentration and adjust to proper reading.
5. Zero the instrument.

PREVENTATIVE MAINTENANCE

Refer to manufacturer's instruction manual.

DOUBLE RING INFILTROMETER TESTING

METHOD: Double Ring Infiltrometer

REFERENCE: ASTM D3385-88

SAMPLE HANDLING: None--In Situ Test

APPARATUS:

1. Infiltrometer
2. Trowel
3. Water supply
4. Graduated container
5. Stopwatch

PROCEDURE:

1. Set outer ring to a depth of 6 inches, inner ring to 4 inches.
2. Fill rings with water to a height of 6 inches above the soil.
3. Start stopwatch.
4. Using the graduated container, maintain constant head in the rings. Record volume of water required. Head should be adjusted every 15 minutes for the first hour, every 30 minutes for the second, and every hour thereafter. Readings should continue until infiltration rate becomes constant.
5. Remove rings and excavate through center of test area to determine depth of wetting front.

NUCLEAR DENSITY TESTING

METHOD: Nuclear Direct Transmission

REFERENCE: ASTM D2922-81, Density of soil and soil-aggregate in place by nuclear methods (shallow depth)

SAMPLE HANDLING: None--In-Situ Test

APPARATUS

1. Nuclear density gauge
2. Drill and drill guide
3. Reference standard

PROCEDURE

1. Standardize instrument (instrument must be standardized every time it is turned off).
2. Select location and prepare site.
3. Drill hole.
4. Insert probe into hole and start instrument.
5. Secure readings and record.

GLT745/92

Appendix E
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

Appendix E
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

All samples collected at Superfund sites for laboratory analysis must follow established documentation protocol. Adherence to this protocol provides a network of valuable information for documenting sample identification, tracking, and chain of custody.

GENERAL DOCUMENTATION PROCEDURES

Organization and concentration are the keys to completing the required documents efficiently and without error. Make certain that a suitable work area has been set aside with ample table and floor space for the processing of forms and the packaging of samples. This is especially important for large projects.

Forms and tags can be filled out in any order; however, past experience has shown that paperwork can be completed most efficiently and accurately if the sample identification matrix is completed before or in conjunction with the completion of the rest of the documentation. The recommended procedure is as follows:

1. Make or obtain a list of the samples to be packaged and shipped on the same day and the laboratories to be used.

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 chain-of-custody 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. If the samples are being shipped for routine analytical service (RAS), 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. Assign tag numbers to each sample container for each sample and enter the numbers on the sample I.D. matrix.

12. Complete one traffic report per laboratory each day (or SAS packing lists or CRL basic data sheets) 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 custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form 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.

Following are step-by-step instructions for completing each form. Other items should be evident from the instructions.

SAMPLE IDENTIFICATION MATRIX (FIGURE 1)

1. Enter site name.

2. Enter project number.

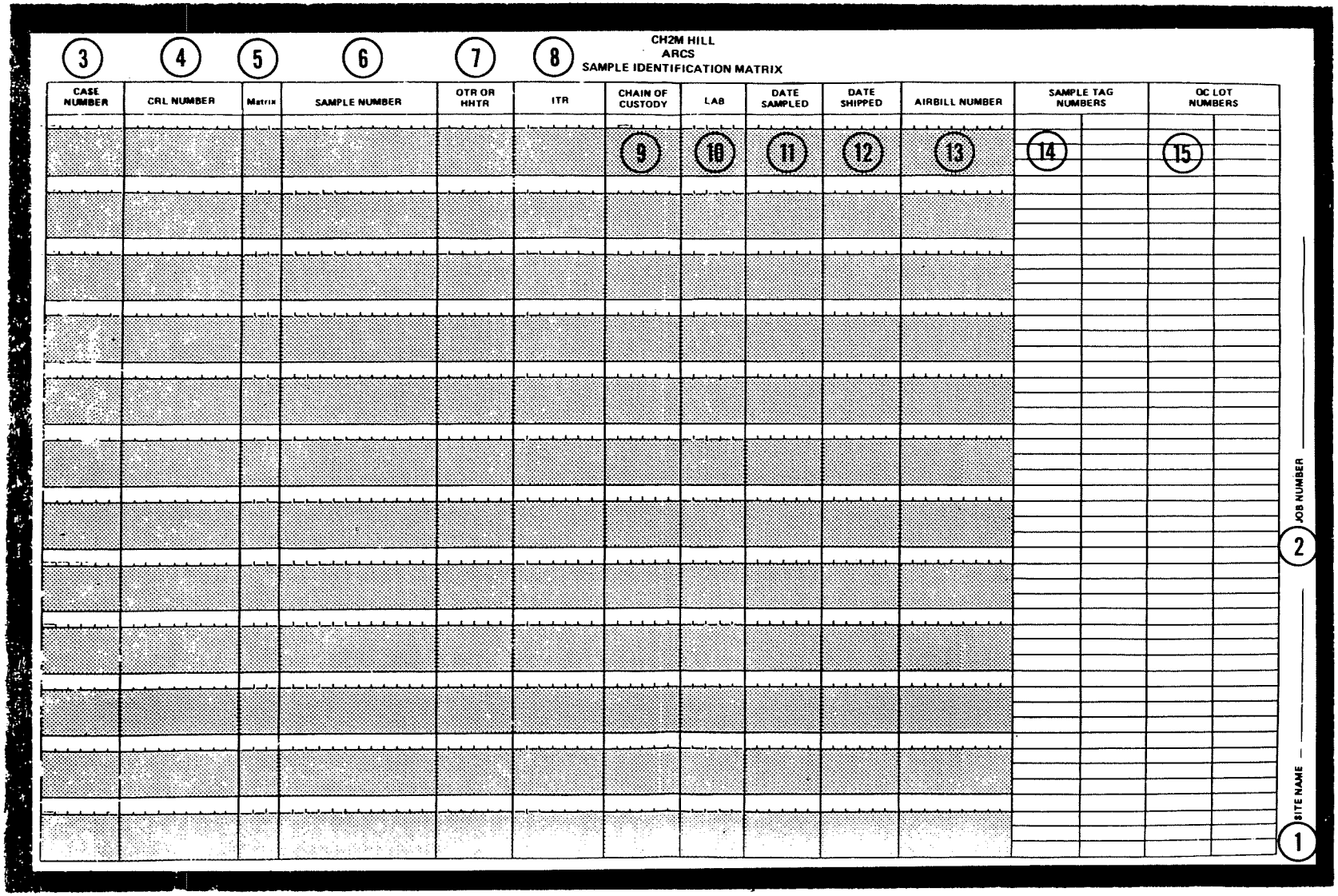
3. Enter the case number.

4. Enter the CRL number.

5. Specify the sample matrix using the two- or three-digit codes listed below followed by the letter L, M, or H to indicate low, medium, or high concentration:

    - SS--Surface Soil
    - SB--Subsurface Soil
    - SWO--Surface Water, Onsite
    - SWC--Surface Water, Creek
    - SDO--Sediment, Onsite
    - SDC--Sediment, Creek
    - GW--Groundwater

6. Enter the sample number.



NOTE: For purposes of illustration this form has been reproduced at 50% of original size.

FIGURE 1

7. Enter the organic traffic label 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., 051284).

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 the first six digits of the CRL sample identification.

2. Enter the last three digits of the CRL identification code.

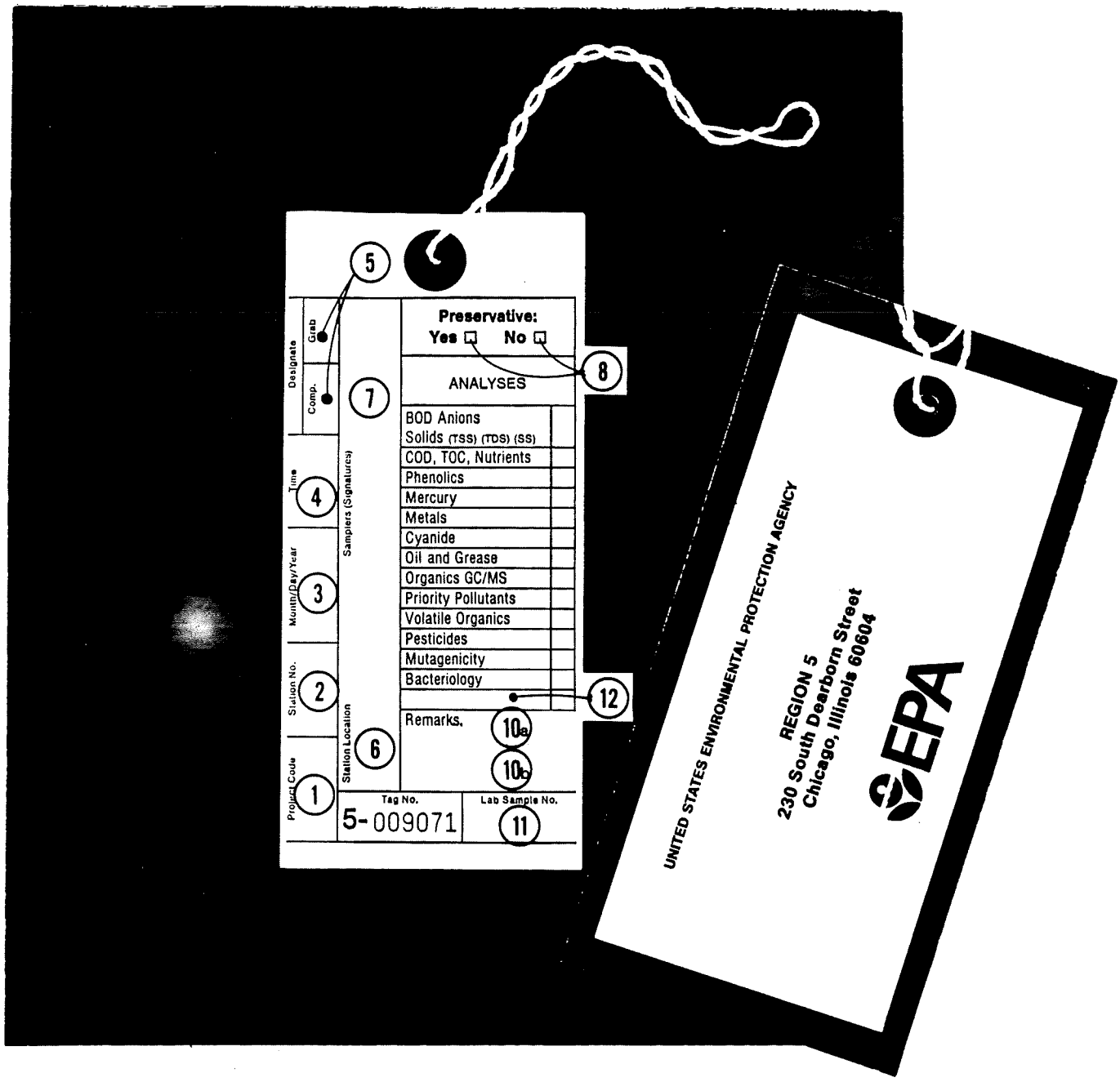
3. Enter date of sampling.

4. Enter time of sampling (military time only).

5. Specify "grab" or "composite" sample with an "X."

6. Enter CH2M HILL sample identification code.

7. Obtain signature of sample team leader.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 2

8. Indicate presence of preservative with an "X."

9. Specify all parameters for analysis with an "X" for each one.

10a. Indicate traffic label type and serial number (e.g., ITR number: MS 1534).

10b. Indicate case number and/or SAS number (e.g., Case No. 1234 and/or SAS No. 5678E).

11. Leave BLANK (for laboratory use only).

12. 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. Circle the code that describes the activity being performed.

4a. Enter site name.

4b. Enter city and state of site.

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.

9. Enter name of laboratory contact.

10. Enter date samples were taken.

11. Enter "F" for Federal Express, date of shipment, and airbill number corresponding with the sample shipment.

12. Write on form if shipment is complete or is not complete.

13. Indicate sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7).

★U. S. GOVERNMENT PRINTING OFFICE: 1987—196-712

USEPA CONTRACT LABORATORY PROGRAM
SAMPLE MANAGEMENT OFFICE
P.O. BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS-557-2490

CASE NO: (1) SAS NO: (2) (IF APPLICABLE)

INORGANIC TRAFFIC REPORT

FOR CLP USE ONLY

TYPE OF ACTIVITY (CIRCLE ONE) ①
SUPERFUND—PA SI ESI RIFS RD RA ER
NPLD O&M OTHER _____
NON-SUPERFUND—_____PROGRAM (3)

SITE NAME: (4a)

CITY, STATE: (4b) SITE SPILL ID:

REGION NO: (5) SAMPLING COMPANY (6) ②

SAMPLER: (NAME) (7)

SHIP TO: ③ (8)

ATTN: (9)

SAMPLING DATE: ④ (10)
BEGIN: _____ END: _____

DATE SHIPPED: _____ CARRIER: _____ ⑤
AIRBILL NO: (11)

SAMPLE DESCRIPTION ⑥
(ENTER IN BOX A)
1. SURFACE WATER 4. SOIL
2. GROUND WATER 5. SEDIMENT
3. LEACHATE 6. OIL (SAS)
7. WASTE (SAS)

DOUBLE VOLUME REQUIRED FOR MATRIX SPIKE/DUPLICATE AQUEOUS SAMPLE

SHIP MEDIUM AND HIGH CONCENTRATION SAMPLES IN PAINT CANS

SEE REVERSE FOR ADDITIONAL INSTRUCTIONS

(12) CLP SAMPLE NUMBER (FROM LABELS)	Ⓐ SAMPLE DESCRIPTION (FROM BOX 1) 1 2 3 4 5 6 7	Ⓑ CONCENTRATION L = LOW M = MED H = HIGH (SAS)	Ⓒ (15) RAS ANALYSIS: TOTAL METALS	CYANIDE	DISSOLVED METALS	HIGH ONLY (SAS): SULFIDE	pH	CONDUCTIVITY	CHLORIDE	Ⓓ SPECIAL HANDLING (16)	Ⓔ STATION LOCATION (17)
(13)											
(14)											

(18)

EPA Form 2075-6 (8-87)

WHITE — SMO COPY PINK — CLIENT COPY WHITE — LAB COPY FOR RETURN TO SMO YELLOW — LAB COPY

FIGURE 3

14. Specify sample concentration with an L, M, or H indicating contaminated concentration.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, M/S/D, field duplicate, or duplicate.

17. Enter CH2M HILL sample number.

18. Leave BLANK (for laboratory use only).

ORGANIC TRAFFIC REPORT (FIGURE 4)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Circle the code that describes the activity being performed.

4a. Enter site name.

4b. Enter site city and state.

5. Enter EPA region number (e.g., V).

6. Enter sample team leader's name.

7. Enter sample team leader's office.

8. Enter laboratory name and address.

9. Enter name of laboratory contact.

10. Enter date samples were taken.

11. Enter "F" for Federal Express, indicate date of shipment,
and indicate airbill number corresponding to sample shipment.

12. Write on form if shipment is complete or is not complete.

13. Specify sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7).

14. Specify the sample concentration with an L, M, or H.

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USEPA CONTRACT LABORATORY PROGRAM
SAMPLE MANAGEMENT OFFICE
P O BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS-557-2490

CASE NO: (1)

SAS NO: (IF APPLICABLE) (2)

ORGANIC TRAFFIC REPORT

FOR CLP USE ONLY

TYPE OF ACTIVITY (CIRCLE ONE) ①
SUPERFUND—PA SI ESI RIFS RD RA ER
NPLD O&M OTHER _____
NON-SUPERFUND—_____PROGRAM
(3)

SITE NAME: (4a)

CITY, STATE: (4b) SITE SPILL ID:

(5) REGION NO: SAMPLING COMPANY ②

SAMPLER: (NAME) (6) (7)

SHIP TO: ③
(8)
(9) ATTN:

SAMPLING DATE: (10) ④
BEGIN: END:

DATE SHIPPED: CARRIER: ⑤
(11) AIRBILL NO:

SAMPLE DESCRIPTION ⑥
(ENTER IN BOX A)
1. SURFACE WATER
2. GROUND WATER
3. LEACHATE
4. SOIL
5. SEDIMENT
6. OIL (SAS)
7. WASTE (SAS)

TRIPLE VOLUME REQUIRED FOR MATRIX SPIKE/DUPLICATE AQUEOUS SAMPLE

SHIP MEDIUM AND HIGH CONCENTRATION SAMPLES IN PAINT CANS

SEE REVERSE FOR ADDITIONAL INSTRUCTIONS

(12) CLP SAMPLE NUMBER (FROM LABELS)	Ⓐ SAMPLE DESCRIPTION (FROM BOX 1) 1 2 3 4 5 6 7	Ⓑ CONCENTRATION L - LOW/MED H - HIGH (SAS)	Ⓒ RAS ANALYSIS (15) VOLATILE	BASE/NEUT /ACID	PESTICIDE /PCB's		Ⓓ SPECIAL HANDLING (16)	Ⓔ STATION LOCATION (17)
(13)		(14)						

(18)

EPA Form 2075-7 (8-87)

WHITE — SMO COPY PINK — CLIENT COPY WHITE — LAB COPY FOR RETURN TO SMO YELLOW — LAB COPY

FIGURE 4

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, M/S/D, field duplicate, or duplicate.

17. Enter CH2M HILL sample number.

18. Leave BLANK (for laboratory use only).

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.

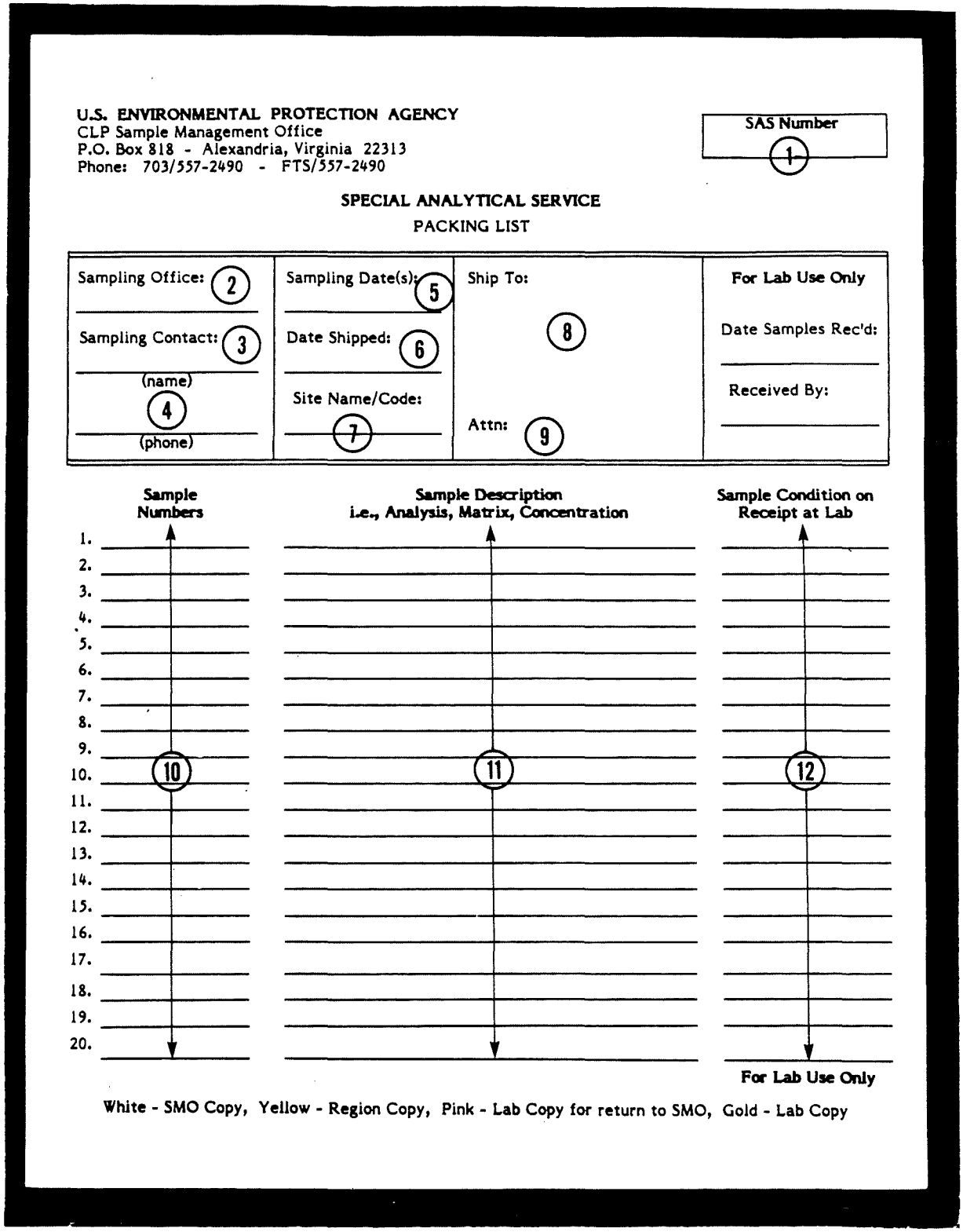
11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag number 5-48246).

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. Enter site name and CH2M HILL project number unless specifically required by EPA SM to omit.



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:

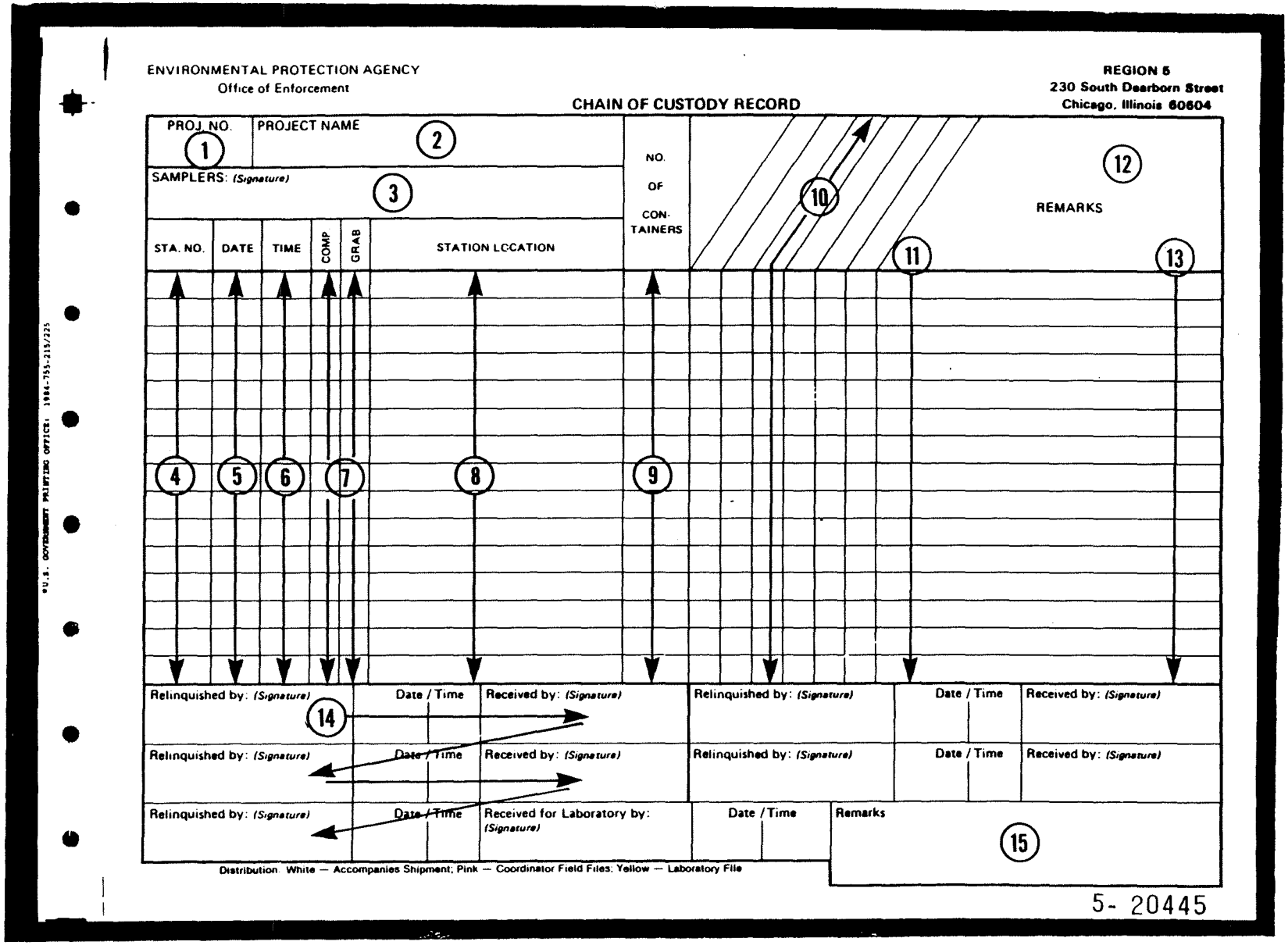
	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 5



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 6

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 and container volume (e.g., 2-40 ml).

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 #:" and enter the case 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.

NOTICE OF TRANSMITTAL (FIGURE 7)

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.

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 7

7. Enter matrix of samples.

8. Enter the site name in words.

9. Enter the location of the site (city, state).

FIELD LOG BOOK

All information pertinent to a field survey or sampling effort will be recorded in a log book or equivalent standard form. Each page or form will be consecutively numbered and will be at least 4-1/2 inches by 7 inches in size. All entries will be made in indelible ink or hard lead pencil and all corrections will consist of line-out deletions that are initialed and dated. As a minimum, entries in a log book will include the following:

- Purpose of sampling
- Location, description, and log 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.)
- Name and address of field contact
- Documentation of procedures for preparation of reagents supplied which 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
- Sample preservation
- Date and time of collection
- Collector's sample identification numbers

- Sample distribution and how transported (i.e., name of the laboratory and transporting agent)
- References such as maps of the sample site
- Any field measurements made (e.g., pH, specific conductance, temperature, and water depth)
- 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.

CORRECTIONS TO DOCUMENTATION

Unless prohibited by weather conditions, 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. 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 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.

LABORATORY CUSTODY

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

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT (FIGURE 8)

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.

PACKING AND SHIPPING 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

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 DU 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												
			ACID/BASE NEUTRAL CPDS ORGANIC SCAN UG/L TOX17574	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN UG/L TOX17584	WATER POLYCHLORINATED BIPHENYLS UG/L PES17144	WATER CHLORINATED PESTICIDES UG/L PES17134	TOTAL METALS IN WATER UG/L MET111	WATER CYANIDE UG/L MIN74513	NITRATE NITRITE MG/L MIN7284	AMMONIA MG/L MIN7284	RESIDUE FILTERABLE TDS MG/L MIN7382	RESIDUE NON-FILT TSS MG/L MIN7372			ACID/BASE NEUTRAL CPDS ORGANIC SCAN MG/KG TOX215722	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN MG/KG TOX219822	SEDIMENTS POLYCHLORINATED BIPHENYLS MG/KG PES211422	SEDIMENT CHLORINATED PESTICIDES MG/KG 211322	TOTAL METALS MG/KG MET413	CYANIDE MG/KG MIN46930	EP TOXICITY METALS MG/KG	AMMONIA MG/KG MIN42928				
(9)	(10)	(10)				(11)																						

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 8

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. Bagged samples 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.

LOW-CONCENTRATION SAMPLES

1. Prepare coolers for shipment.

   - o Tape drains shut.
   - o Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
   - o Place mailing label with laboratory address on top of coolers.
   - o Fill bottom of coolers with about 3 inches of vermiculite or use performed poly-foam liner.
   - o 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.

16. Tape cooler shut on both ends, making several complete revolutions with strapping tape. Do not cover custody seals (see Figure 9).

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:

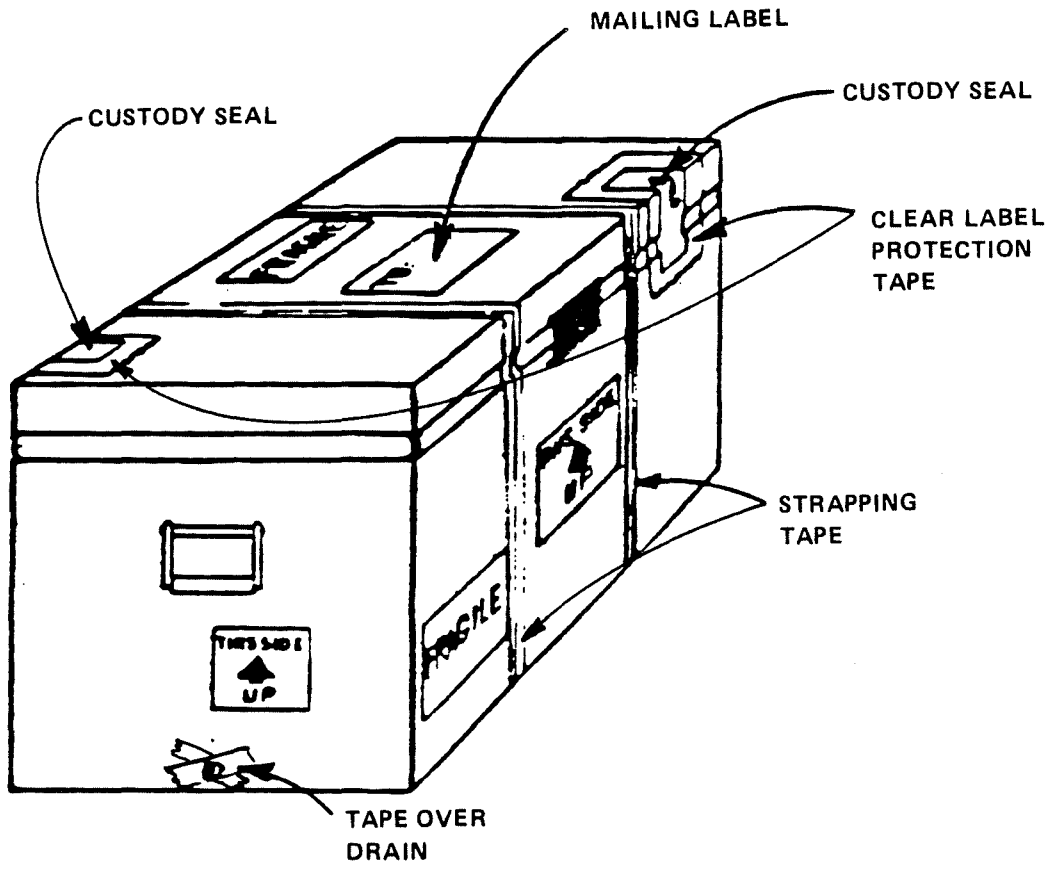

MAILING LABEL
CUSTODY SEAL
CUSTODY SEAL
CLEAR LABEL
PROTECTION
TAPE
STRAPPING
TAPE
THIS SIDE
UP
FRAGILE
TAPE OVER
DRAIN

FIGURE 9

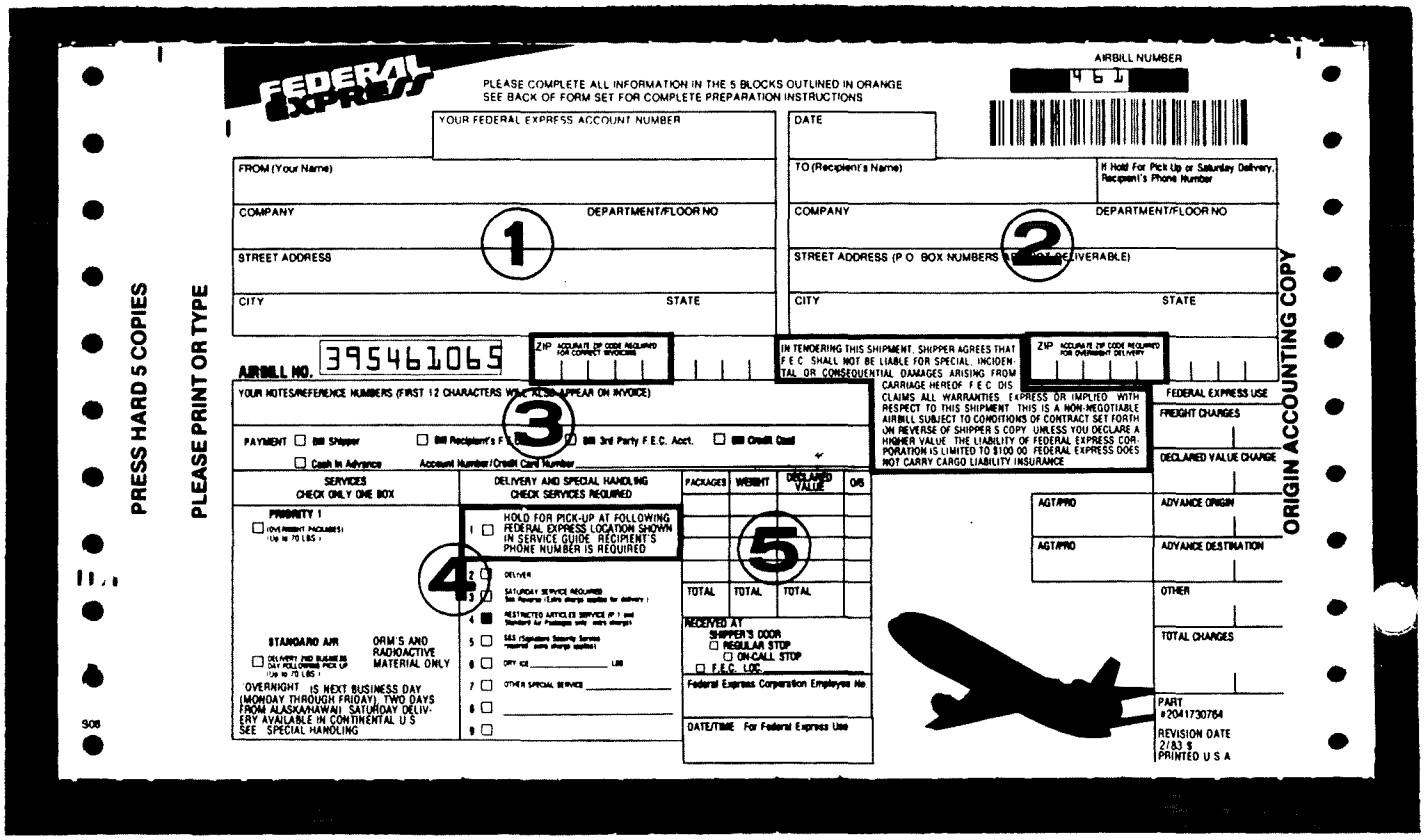
- Your name
- Project name
- Case number
- Number of samples sent to each laboratory for analysis
- Airbill numbers

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:

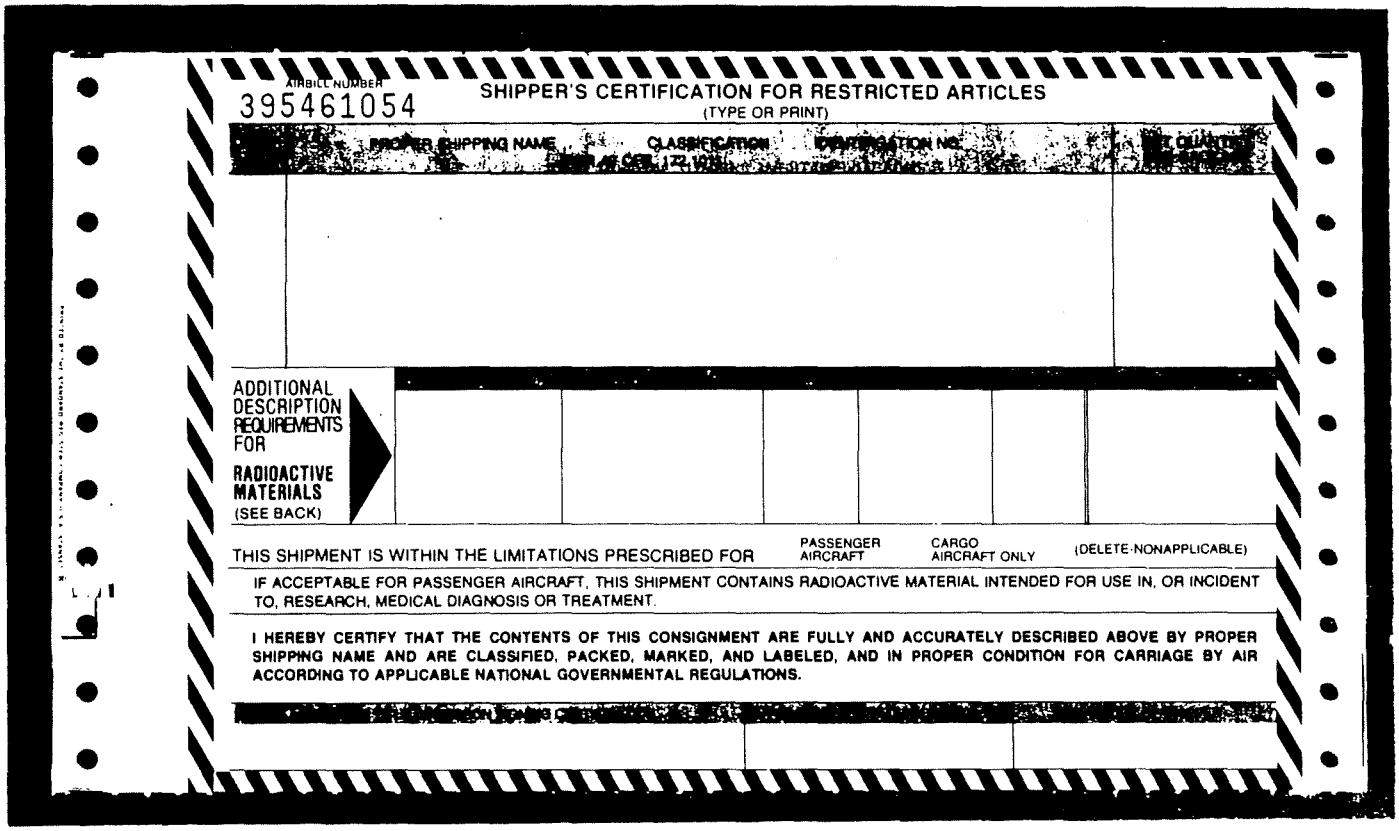
Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required (Figures 10 and 11). Second, "Flammable Liquid 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.

- Place approximately one-half inch of vermiculite in the bottom of the can.
- Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.
- Place the jar in the can and fill the remaining volume with vermiculite.
- Close the can and secure the lid with metal clips.
- Write the traffic report number on the lid.
- Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.
- Place the cans in the cooler.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 10



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 11

DISTRIBUTION OF COMPLETED DOCUMENTS

Final disposition of the completed documents is as follows:

- Shipped with Samples:
  - Chain-of-custody form, white original
  - Traffic report forms, white and yellow copies
  - SAS packing list, pink and gold 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, pink and yellow copies
  - Traffic report forms, white original and pink copies
  - SAS packing list, white original and yellow copy
  - Notice of transmittal

SPECIAL INSTRUCTIONS FOR SHIPPING SAMPLES BY FEDERAL EXPRESS

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 NA#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.

GLT824/62