ARCS V

Remedial Activities at Uncontrolled Hazardous Waste Sites in Region V

EPA United States Environmental Protection Agency

Quality Assurance Project Plan

Onalaska Municipal Landfill
Onalaska, Wisconsin

Groundwater Remedial Action Startup Testing
WA No. 38-5NL5 / Contract No. 68-W8-0040

CH2M HILL

Quality Assurance Project Plan

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January 1994

Section 1

Title Page

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

Project Title: Groundwater Remedial Action-Startup Testing
Onalaska Municipal Landfill
Onalaska, Wisconsin

EPA No: WA 38-5NL5

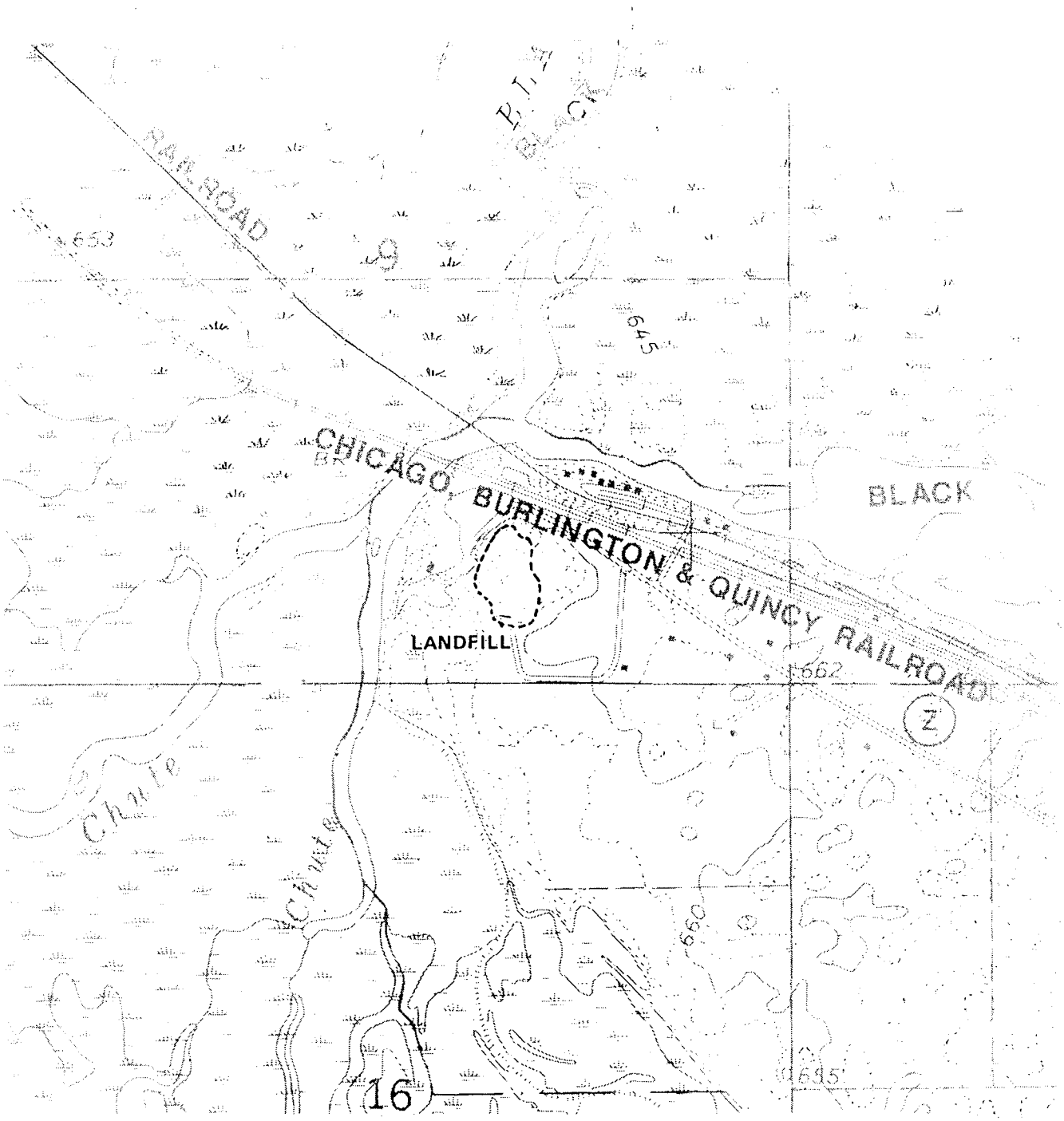
EPA Remedial Project Officer: Kevin Adler

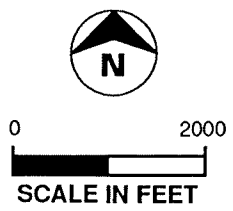
Prepared by: CH2M HILL Date: January 1994

Approved by: _____ Date: _____
Stevan Keith
CH2M HILL Site Manager

Approved by: _____ Date: _____
Alpheus Sloan, III
Acting CH2M HILL Program Manager

Approved by: _____ Date: _____
Kevin Adler
EPA Region 5
Remedial Project Manager





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

FIGURE 1
SITE LOCATION MAP
ONALASKA
Startup Sampling QAPP

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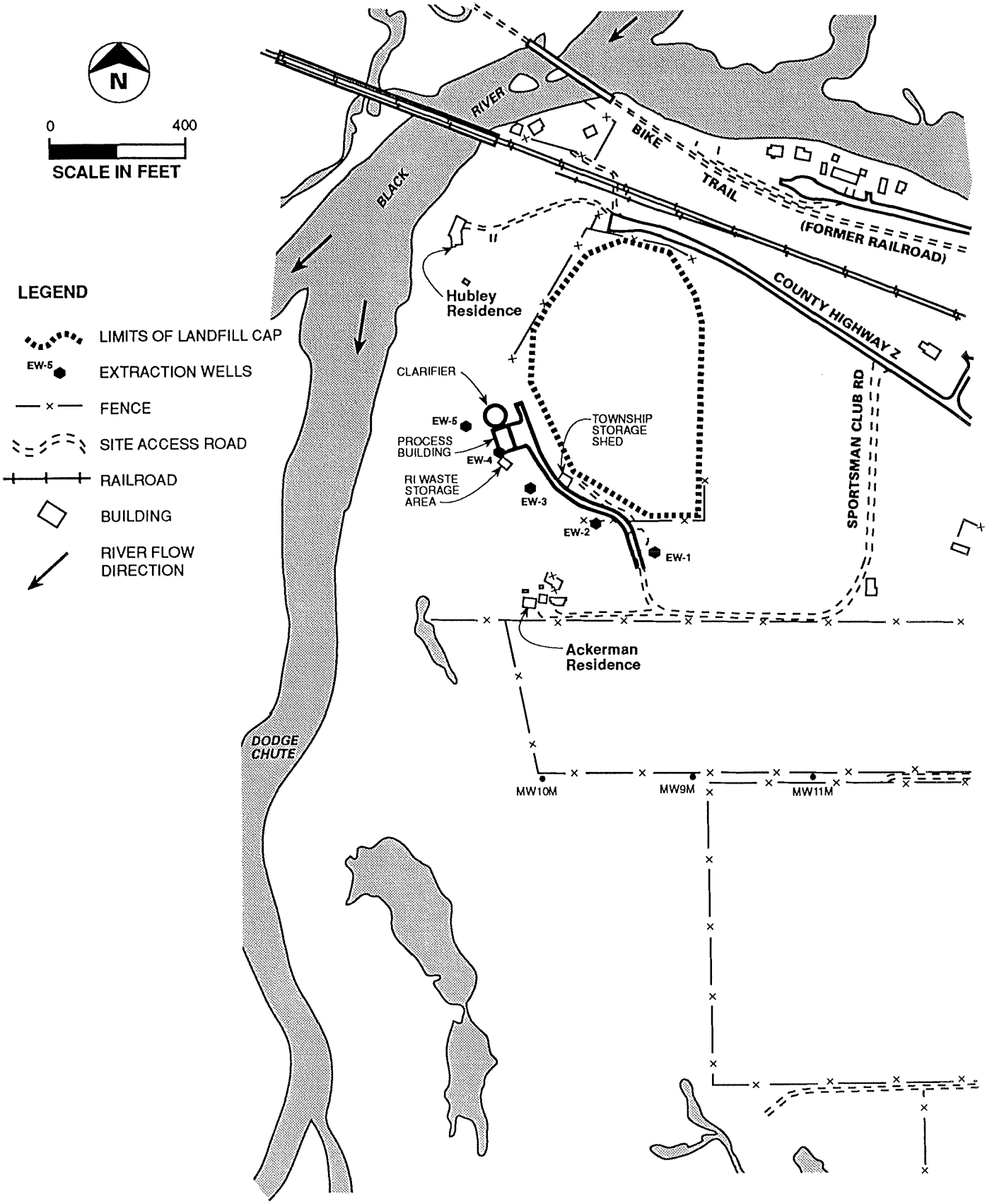

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FIGURE 2
SITE MAP
ONALASKA LANDFILL
Startup Sampling QAPP

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Section 3
Project Description

3.1 Introduction

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

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

This QAPjP covers specifically sampling at discreet steps during startup of the groundwater treatment process to assess effectiveness of the treatment system and compliance with effluent requirements. Groundwater sampling from monitoring wells and extraction wells that will occur during remediation was covered in a QAPjP dated May 1992.

3.2 Site Description

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

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

3.3 Site History and Background

3.3.1 Site History

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

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

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

Outers Laboratories and Metallics, Inc. (two companies owned by one person), contributed significant quantities of industrial wastes to the site. Daily landfill operation reports indicate that these two companies were disposing of industrial waste oils and solvents as early as July 7, 1970. Early DNR records report that Outers delivered liquid solvent residues to the site for burning. The waste solvents consisted primarily of naphtha, toluene, and paint residues. Initially, both Outers and Metallics hauled solvent

wastes in 55-gallon barrels. Once a week, 20 to 25 barrels of industrial wastes from the companies were hauled to the landfill. The barrels were emptied and the waste was burned. After burning was banned, the liquid waste was dumped in the designated area and poured into excavated holes for immediate burial. Occasionally, full barrels were left at the site if they could not be easily emptied or if they were damaged or leaking. In later years, the liquid waste was hauled in a 500-gallon truck instead of barrels. At that time, about 300 barrels were additionally mass buried at the landfill.

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

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

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

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

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

3.3.2 Background

Except for the industrial waste solvents from Outers Laboratories and Metallics, Inc. There is little indication that the wastes within the 7 acres used for open pit disposal were segregated. Industrial, commercial, and municipal wastes are considered to be mixed throughout the fill area. Outers and Metallics used a specific area designated for liquid industrial waste disposal according to DNR correspondence and license applications. However, the designated disposal area was not strictly limited to the industrial wastes from Outers and Metallics. Records indicate that other commercial wastes were deposited simultaneously in the area designated for liquid industrial waste disposal in October 1981 and October 1982.

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

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

Some of the organic compounds detected in the groundwater during past analyses may have been derived from naphtha wastes floating on the water table. The liquid naphtha waste could generate a complex mixture of dissolved organic compounds in the groundwater over a period of time. Both types of naphtha would each produce a different suite of degradation products of varying composition. It is impossible to predict

the exact composition of each mixture, but generally naphtha degradation products consist of aliphatic and aromatic carboxylic acids, toluene, and other complex mixtures of aromatic and aliphatic hydrocarbons.

3.4 Target Compounds

3.4.1 Remedial Investigation Target Compounds

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

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

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

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

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

Volatile Organic Compounds (VOCs) were generally observed to be present at concentrations much greater than semivolatile organics (sometimes more than an order of magnitude greater). The majority of the VOCs detected during the Remedial

Investigation were found in shallow monitoring wells (MW-5S and MW-3S and B4S) and were BTEX compounds. The vertical extent of BTEX and chlorinated compounds contamination is mostly confined to the upper 10 to 20 feet of the aquifer. Ethylbenzene, 1,1-DCA and chloroethane were detected, however, at depths up to 50 to 60 feet below the water table. The vertical extent of semivolatile organic compounds (SVOCs) contamination is also mostly confined to the upper 10 to 20 feet of the aquifer. There were no SVOCs detected in any of the deep monitoring wells.

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

3.4.2 Remedial Action Cleanup Standards

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

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

The ROD requires that the more stringent Wisconsin standards promulgated in NR 140, WAC, be achieved "at any point beyond the property boundary or beyond the three-dimensional design management zone, whichever is closer to the waste boundary." The DMZ as defined in NR 140 is a three-dimensional boundary surrounding a regulated facility. The boundary extends from the ground surface through all saturated geological strata. The DMZ defined for the Onalaska site extends 250 feet horizontally from the

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

Well	Chemical	Detected Concentration (μg/L)	Criteria Exceeded[a]	Criteria Level (μg/L)
MW02S-01	Benzene	5	ES	0.67
			PAL	0.067
	Arsenic	9.5	PAL	5
	Chromium	24.8	PAL	5
MW02M-01	Arsenic	19.4	PAL	5
	Barium	1,390	ES	1,000
			PAL	200
MW03S-01	1,1-Dichloroethene	15	ES	0.24
			PAL	0.024
	Benzene	13	ES	0.67
			PAL	0.067
	1,1,1-Trichloroethane	240	ES	200
			PAL	40
	Trichloroethene	11	ES	1.8
			PAL	0.18
	Toluene	8,300	ES	343
			PAL	68.6
	Xylene	2,300	ES	620
			PAL	124
	Arsenic	19.4	PAL	5
	Barium	593	ES	1,000
MW03M-01	Arsenic	68.4	ES	50
			PAL	5
	Barium	2,760	ES	1,000
			PAL	200
MW04S-01	Toluene	530	ES	343
			PAL	68.6
	Arsenic	6.9	PAL	5
	Barium	1,140	ES	1,000
MW05S-01	Benzene	7	ES	0.67
			PAL	0.067
	Toluene	8,300	ES	343
			PAL	68.6
	Xylene	1,400	ES	620
			PAL	124
	Arsenic	8	PAL	5
	Barium	347	PAL	200
MW06M-01	Barium	1,370	ES	1,000
			PAL	200
MW08M-01	Barium	600	PAL	200
W21S-01	Barium	201	PAL	200

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

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

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

Well	Chemical	Detected Concentration (µg/L)	Criteria Exceeded[a]	Criteria Level (µg/L)
MW02S-01	Benzene	5	MCL	5
			MCLG	0
			WQC-Risk	0.67
	Ethylbenzene	210	MCL2°	30
MW02M-01	Barium	1,390	MCL	1,000
MW03S-01	Benzene	13	MCL	5
			MCLG	0
			WQC-Risk	0.67
	1,1-Dichloroethene	15	MCL	7
			MCLG	7
			WQC-Risk	0.033
	1,1,1-Trichloroethane	240	MCL	200
			MCLG	200
			DWLHA	200
	Trichloroethene	11	MCL	5
			MCLG	0
			WQC-Risk	2.8
	Toluene	8,300	MCL-Prop	2,000
			MCL2°	40
			MCLG-Prop	2,000
			DWLHA	2,040
	Xylene	2,300	DWLHA	400
			MCL2°	20
MW03M-01	Arsenic	68.4	MCL	50
			DWLHA	50
			WQC-Risk	0.0025
	Barium	2,760	MCL	1,000
			DWLHA	1,500
MW04S-01	Ethylbenzene	42	MCL2°	30
	Toluene	530	MCL2°	40
MW05S-01	Benzene	7	MCL	5
			MCLG	0
			WQC-Risk	0.67
	Ethylbenzene	160	MCL2°	30
	Toluene	8,300	MCL-Prop	2,000
			MCL2°	40
			MCLG-Prop	2,000
			DWLHA	2,040
	Xylene	1,400	DWLHA	400
			MCL2°	20
MW06M-01	Barium	1,370	MCL	1,000

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

[a]Criteria abbreviation:

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

Table 3 Analytical Methods	
Analysis	**Method**
BOD	EPA 405.1 (5-day)
TSS	EPA 160.2
TDS	EPA 160.1
Metals	CLP 3/90 SOW/ILM 01.0
Hardness	EPA 130.2
Nitrate/Nitrite	EPA 353.2
Ammonia	EPA 350.1
VOCs	CLP 3/90 SOW/OLM 01.1
BTEX	EPA 602
SVOC	CLP 3/90 SOW/OLM 01.1
Pesticides (low level)	EPA 508
Pesticides/PCBs	CLP 3/90 SOW/OLM 01.1
Acute Toxicity Bioassay	See Appendix E
Chronic Toxicity Bioassay	See Appendix E
Iron	CLP 3/90 SOW/ILM 01.0
TCLP Metals, VOCs, SVOCs, Pesticides	EPA 1311 (ext) CLP SOW 3/90 OLM 01.1 (organics) ILM 01.0 (inorganics)
Chloride	EPA 325.2
Bulk Density	ASTM D5057
Percent Solids	EPA 160.3
Total Cyanide	EPA 335.2
Paint Filter Liquids Test	SW-846 Method 9095
Total Phenols	EPA 420.1

waste boundary as shown in Figure 3. Because the property boundary generally is closer to the waste boundary than to the DMZ, the property boundary is considered the point at which PALs apply. As the plume is reduced in size, however, the mcls or mclGs could apply.

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

Groundwater sample analyses from the monitoring and extraction wells were described in the QAPjP dated May 1992.

3.4.3 Treatment System Standards

Treatment of extracted groundwater is required as part of the remedial action selected in the ROD. Table 3 lists the analytical methods of the chemical parameters that will be monitored as part of the groundwater treatment system startup testing. For process startup purposes, the contaminants of concern have been identified as the BTEX compounds, iron, and the conventional parameters that will allow assessment of process performance. Table 4 lists the contaminants of concern and their expected process influent and effluent levels. A secondary list of contaminants with WDNR computed effluent limits will also be monitored. Table 5 lists these constituents, their allowable effluent levels, and method detection limits. For the detection limits of constituents not listed in Table 5; see the SASs in Appendix B. Sampling and analyses requirements were established by the Wisconsin Department of Natural Resources.

The Process Startup Sampling Plan (Appendix A) describes sampling locations, frequency, and effluent limits that must be met.

Table 6 lists the sample bottle and preservative requirements.

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

Table 4 Contaminants of Concern Summary of Influent/Effluent Levels		
Contaminant	**Average Influent Concentration**	**Effluent Concentration**
Benzene	4 μg/L	< 1 μg/L
Ethylbenzene	79 μg/L	< 1 μg/L
Toluene	2,800 μg/L	140 μg/L
Xylene	625 μg/L	31 μg/L
Iron	25 mg/L	1 mg/L
BOD	3 mg/L	2 mg/L
TSS	22 mg/L	10 mg/L
NH_3	10 mg/L	8 mg/L

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Table 5
Secondary List of Contaminants
Constituents with WDNR Computed Process Effluent Limits
(Page 1 of 2)

Effluent Characteristic	Daily Maximum (mg/L)	Detection Limit (mg/L)
Water Quality Limits		
Ammonia, total (varies w/pH & temp)	0.8	0.01
Iron, total	2.48	0.10
Chloride	1,700	1.0
Arsenic, total	0.73	0.01
Cadmium, total	0.17	0.005
Chromium (+3 or tot)	8.1	0.01
Chromium (+6)	0.028	0.01
Copper, total	0.08	0.025
Lead, total	1.1	0.003
Nickel, total	4.8	0.04
Zinc, total	0.46	0.02
Aluminum, total	1.5	0.20
Benzene*	22	0.01
Chloroform	29	0.01
1,1-Dichloroethylene	6	0.01
1,2-Dichloroethylene	30	0.01
Ethylbenzene*	45	0.01
Toluene*	17	0.01
1,1,1-Trichloroethane	53	0.01
Trichloroethylene	41	0.01
Vinyl Chloride	1.0	0.01
Pentachlorophenol	0.034	0.01
Phenol	7.0	0.01
Di-n-butyl Phthalate	0.94	0.01

Table 5
Secondary List of Contaminants
Constituents with WDNR Computed Process Effluent Limits
(Page 2 of 2)

Effluent Characteristic	Daily Maximum (mg/L)	Detection Limit (mg/L)
Water Quality Limits (cont'd)		
1,4-Dichlorobenzene	1.4	0.01
2,4-Dinitrotoluene	32	0.01
Naphthalene	6.6	0.01
Pyrene	0.0125	0.01
Aldrin	0.0043	0.00005
Gamma-BHC	0.0076	0.00005
4,4'-DDD	2.7 E-6	1.0 E-6
Proposed Technology Based Limits*		
BTEX, total	0.2	0.05
Benzene	0.05	0.001
% Removal BTEX		

* Note: Technology based limits are more restrictive than water quality limits above for BTEX (benzene, ethylbenzene, toluene and xylene)—cond. B(8) and Table 4.

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Table 6
Sample Bottle and Preservation Requirements

Analyte	Bottle(s)	Preservative	Comments	EPA Hold Time (days)
VOCs[a,b]	(3) 40 mL vials	HCl to pH of 2	No headspace—check for bubbles	14
SVOCs	(1) 2-L amber glass	---	---	7/40
Pesticides/PCBs	(1) 2-L amber glass	---	---	7/40
BTEX	(3) 40 mL vials	HCl to pH of 2	No headspace—check for bubbles	14
Metals including Fe[a,c]	(1) 1 L poly	HNO3 to pH of 2	---	6 months
Ammonia	(1) 1 L poly	H2SO4 to pH of 2	---	28
Nitrate	(1) 500 mL poly	---	---	48 hours
BOD_5	(1) 1 L poly	---	Fill completely	48 hours
Hardness	(1) 250 mL poly	HNO3 or H2SO4 to pH of 2	---	6 months
TSS/TDS	(1) 500 mL poly	---	---	7
Cyanide	(1) 500 mL poly	NaOH to pH > 12	---	14
Paint Filters	(1) 8-oz. glass	---	Collect ≥ 100 g	---
Phenols	(1) 1 L glass	H_2SO_y	---	28
% Solids	(1) 500 mL poly	---	---	7
TCLP VOC	(2) 2-oz. glass	---	---	14
TCLP SVOC	(2) 8-oz. glass	---	---	7/40
TCLP Pesticides	(2) 8-oz. glass	---	---	7/40
TCLP Metals	(1) 8-oz. glass	---	---	6 months
Acute and Chronic Toxicity	(1) poly 4-gallon total for each 24-hour composite sample	---	---	48 hours

[a] A Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample is required for each sample group of 20 or less.
[b] For MS/MSD, triple volume must be collected and labeled "MS/MSD."
[c] For MS/MSD, no extra volume is required. Sample must be labeled "MS/MSD.

3.5 Project Data Collection Objectives

The objectives of the startup sampling are to:

- Document performance of the groundwater treatment process during system startup
- Document treatment system efficiency at startup
- Document compliance with effluent limits at startup
- Document compliance for important indicator parameters

3.5.1 Intended Data Usage

These data shall be used to evaluate the effectiveness of the remedial action groundwater treatment system. The data will be used to identify components in the process that may not be performing to the degree specified so that the process operation and performance can be corrected, if necessary, before full-scale implementation.

3.5.2 Data Quality Objectives

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

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

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

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

Table 7
Data Uses and Associated EPA Levels

Media	Sample Analyses	Data Use	DQO Level
Compliance Monitoring			
Process Water	• Acute and Chronic Bioassay • Complete priority pollutant scan • All parameters: TSS, TDS, Ammonia as N, BOD, hardness, nitrate	Phase 1: Document compliance with effluent limits and requirements prior to discharge	IV V III
	Parameters with effluent limits, BOD_5, and TSS	Phase 2: Document performance of process during startup	V III
	BTEX compounds, ammonia, iron, BOD_5, TSS	Phase 3: Demonstrate successful startup performance of treatment process	V III
Performance Monitoring			
Process Water	See compliance monitoring above	Document treatment system efficiency (% pollutant removal) for BTEX, VOC, and iron	V
	Iron, TSS, TDS, pH, flow, temp.	Clarifier performance monitoring	III
	BTEX, ammonia, flow, temp, _____	Stripper performance monitoring	III
	• Flow, TSS (6), (7), and ammonia • % solids, bulk density, vol (8)	Filter press performance monitoring	III
	Flow, pH, temperature	Process mass balance	I
Additional Testing			
Process Solids	TCLP, paint filter	Landfill disposal testing	V
Note: Conventional analyses will have DQO Level III			

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

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

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

Table 7 lists the data uses and associated EPA levels.

Levels I and V analytical data will be generated during groundwater treatment system startup testing at the Onalaska Municipal Landfill. DQO Level I data to be generated include field measurements of process water pH, temperature, and specific conductance. The laboratory analyses requested include the Level V chemical, biological, and physical analyses. Level V data will be needed:

- To provide the rigorous QA/QC required to track and monitor the groundwater treatment system effectiveness

- To be able to assess the need for and perform necessary modifications to the treatment system to meet effluent limits, and

- Because nonstandard procedures are required to meet the lower detection limits of VOC and inorganic analyses.

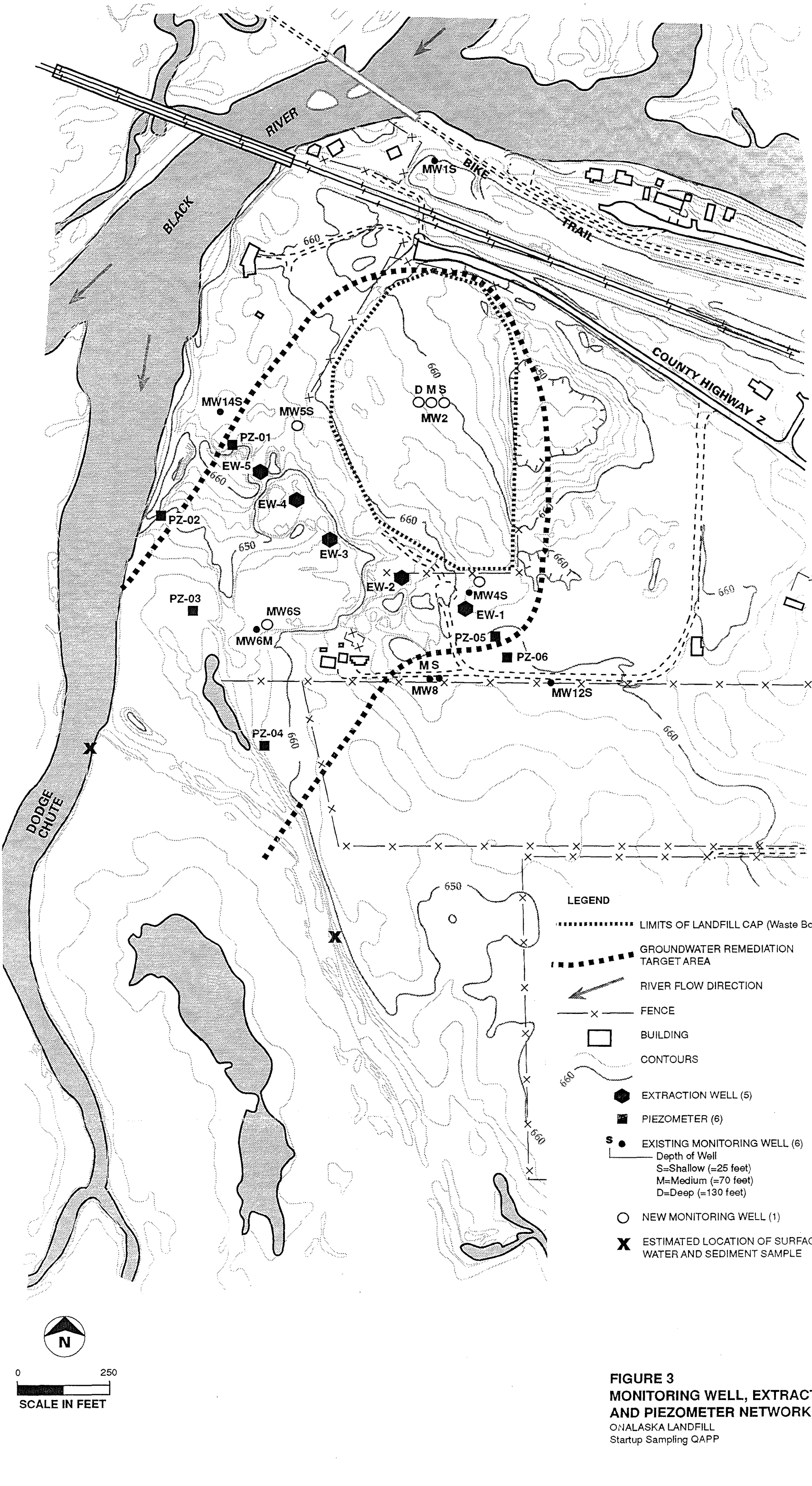


FIGURE 3
MONITORING WELL, EXTRACTION WELL, AND PIEZOMETER NETWORK
ONALASKA LANDFILL
Startup Sampling QAPP

3.6 Sample Network Design and Rationale

The treatment process water sampling locations and frequency for the sampling to be performed during system startup are described in Section 6 of the Process Startup Sampling Plan (Appendix A).

3.7 Project Schedule

The process startup sampling will be performed within one month of completion of system construction and equipment operations checks. The schedule for ongoing startup sampling is discussed in Section 6 of the Process Startup Sampling Plan (Appendix A).

Section 4
Project Organization and Responsibility

At the direction of the Region 5 Remedial Project Manager (RPM), with final authority by the Region 5 Regional Project Officer (RPO), CH2M HILL has responsibility for the process startup sampling and associated sample QA/QC. CH2M HILL will perform the field sample collection, field screening, and prepare the startup sampling report. The project organization chart is included as Figure 4. Operation, maintenance, and sampling associated with the system O and M will be performed under a separate contract, yet to be awarded.

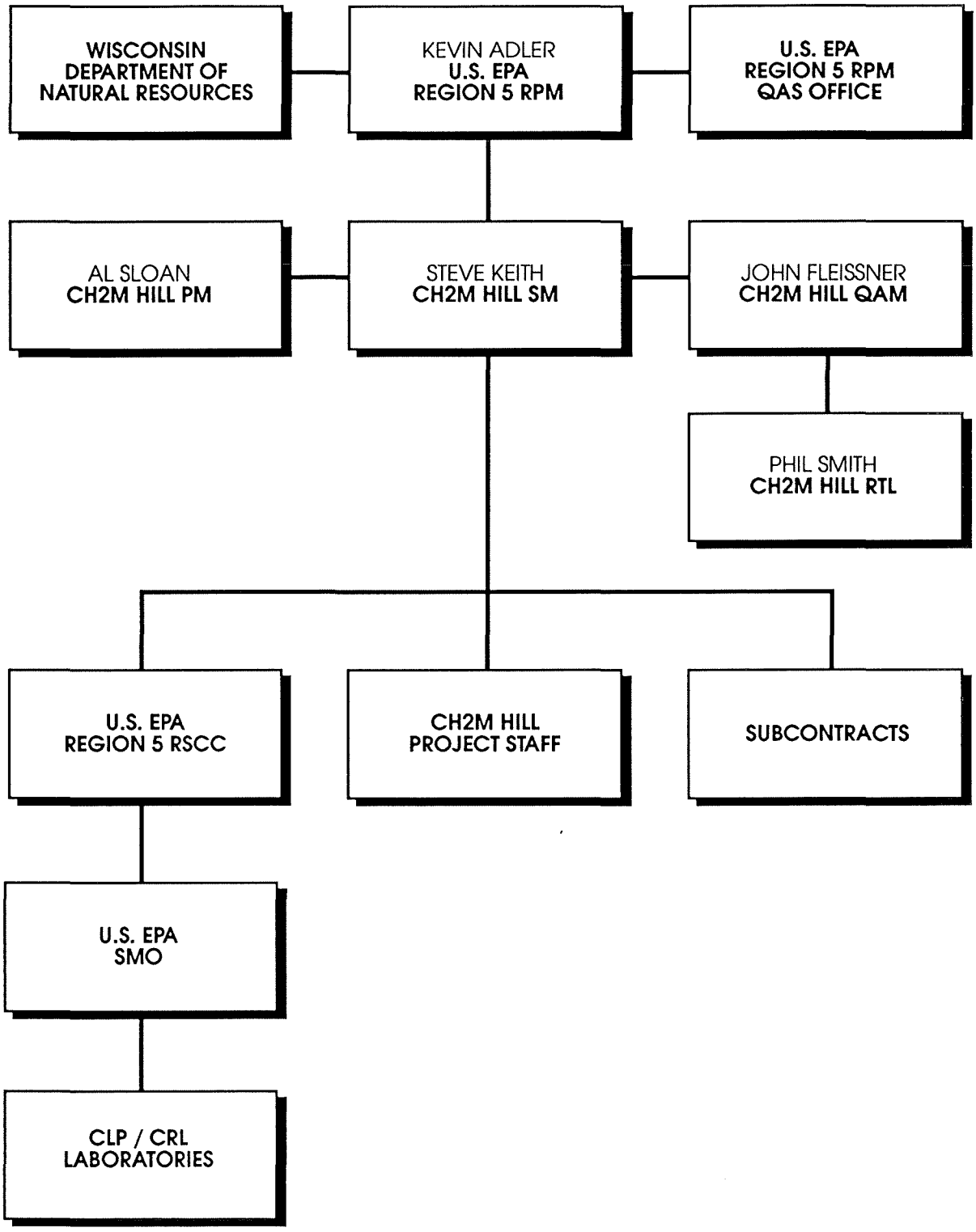
4.1 Management Responsibilities

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

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

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

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



GLE65624.SU.12 ORG CHART 1-3-94 TLL

FIGURE 4
PROJECT ORGANIZATION
ONALASKA
Startup Sampling QAPP

- Program Manager (PM)
  Alpheus Sloan (CH2M HILL)

The responsibilities of the aforementioned personnel are described below.

Remedial Project Manager

The RPM has the responsibility for the implementation of the Remediation Plan and Startup Sampling Plan.

State Project Manager

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

Program Manager

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

Site Manager

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

- Define project objectives and develop a detailed work plan schedule
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task
- Acquire and apply technical and corporate resources as needed to maintain performance within budget and schedule constraints
- Orient all field leaders and support staff concerning the project's special considerations

- Monitor and direct the field leaders
- Develop and meet ongoing project and/or task staffing requirements, and develop mechanisms to review and evaluate each task product
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all external reports (deliverables) before their submission to EPA Region 5 and WDNR
- Be responsible for preparation and quality of interim and final reports
- Represent the project team at meetings and public hearings

4.2 Quality Assurance Organization

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

Tasks	Responsible Organization/Personnel
• Final review and approval of QAPjP	Kevin Adler, U.S. EPA Region 5 RPM U.S. EPA Region 5 QA Officer
• QA program for CLP, SAS performance and systems audits for CLP SAS	U.S. EPA Headquarters U.S. EPA Sample Management Office U.S. EPA Region 5 CRL LSSS U.S. EPA EMSL-Las Vegas, QA Division
• QA review and approval of reports, plans and procedures, and field activities; and identifying and controlling non-conformance for	John Fleissner, CH2M HILL, Quality Assurance Director (QAD)

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

4.3 Field Operations

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

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

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

Field Team Leaders

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

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

Field Team Members

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

4.4 Laboratories

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

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

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

Section 5
Quality Assurance Objectives for Measurement Data

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

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

5.1 Level of Quality Control Effort

Field blank, trip blank, duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks, consisting of distilled water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the site which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes (MSs) provide information about the effect of the sample matrix on the extraction/digestion and measurement methodology. Whereas, inorganic analyses require one MS sample, volatile organic matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

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

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

The solids and process water samples will be sent to the Contract Laboratory Program/ Central Regional Laboratory for analysis. Table 8 lists the QA/QC samples that will be collected. The level of laboratory QC effort for SAS analyses is outlined individually in each SAS contained in Appendix B.

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

5.1.1 Accuracy, Precision, and Sensitivity of Analysis

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

The accuracy, precision, and sensitivity requirements for SAS for the CLP are specified in each individual SAS request contained in Appendix B. The standard operating procedures (SOPs) for the field equipment to measure pH, conductivity, temperature, and dissolved oxygen are outlined in Appendix C. QA requirements for field screening analyses are also included in SOPs found in Appendix C.

Table 8
QA/QC Sampling Summary
Onalaska Startup Sampling Plan
Onalaska, Wisconsin

Sample Analyses	Number of Samples	QC Samples		
		Duplicates	Trip Blanks	MS/MSD
Process Water Samples				
• Acute and Chronic Bioassay (effluent at (4) and (5))	2	--	--	--
• Complete priority pollutant scan	3	1	1	1
• All parameters: pH, TSS, TDS, Ammonia as N, Temp, BOD_5, hardness, nitrate	3	--	--	--
Parameters with effluent limits (Table 5-1) and pH, temperature, BOD_5, and TSS	6	1	2	1
BTEX compounds, pH, ammonia, iron, temperature, BOD_5, TSS	36	4	12	2
Iron, TSS, TDS, pH, flow, temp (filtered and unfiltered iron)	28	--	--	--
BTEX, ammonia, flow, temp	14	--	--	--
• Flow, TSS • % solids, bulk density, vol	6 3	--	--	--
Flow				
Process Solid Samples				
TCLP, paint filter	1	--	--	--

Note: Trip blanks are for VOCs only. Conventional analyses will not have duplicates of MS/MSDs.

conditions. It is expected that the field measurement and CLP laboratories will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

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

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling program was designed to provide data representative of specific treatment system processes' effectiveness. The rationale of the sampling program is discussed in detail in the Process Startup Sampling Plan (Appendix A). Representativeness will be satisfied by seeing that the Process Startup Sampling Plan is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness also will be assessed by analyzing of field duplicated samples.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives.

5.2 Method Detection Limits

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

Section 6
Sampling Procedures

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

Section 7
Sample Custody Procedures

7.1 Introduction

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

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

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

7.2 Field Custody Procedures

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

7.2.1 Field Procedures

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

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

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

7.2.2 Sample Documentation Procedures

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

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

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

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

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

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

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

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

5. Complete an airbill for each laboratory address.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

18. Prepare samples for shipment.

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

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

7.2.3 Transfer of Custody Procedures

Transfer of custody procedures are as follows:

1. Samples must be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to or from a secure storage area.

2. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record

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

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

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

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

7.2.4 Field Log Book

All information pertinent to a field survey or sampling effort will be recorded in a bound log book or equivalent standard form. Each page or form will be consecutively numbered and will be at least 4-1/2 inches by 7 inches in size. All entries will be made in indelible ink or, if weather conditions dictate, in hard lead pencil, and all corrections will consist of line-out deletions that are initialed and dated. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

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

- Project start date
- End date

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

- Purpose of sampling
- Location, description, and log of photographs of the sampling point
- Details of the sampling site (e.g., the elevation of the casing, casing diameter and depth, integrity of the casing, etc.)
- Weather conditions
- Name and address of field contact
- Documentation of procedures for preparation of reagents supplied that become an integral part of the sample (e.g., filters and absorbing reagents)
- Identification of sampling crew members
- Type of sample (e.g., groundwater, sludge, wastewater)
- Suspected waste composition
- Number and volume of samples taken
- Sampling methodology, including distinction between grab and composite samples
- Modifications from Field Sampling Plan
- Sample preservation
- Date and time of collection
- Sample identification designation and tag numbers
- Sample distribution and how transported (i.e., name of the laboratory and transporting agent)

- References such as maps of the sample site
- All field measurements data (e.g., pH, specific conductance, temperature, and water depth)
- Method of field measurement data reduction
- Signature and date by the personnel responsible for observations
- Decontamination procedures

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

7.2.5 Corrections to Documentation

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

7.2.6 Distribution of Completed Documents

Final disposition of the completed documents is as follows:

- Shipped with samples:
  - Chain-of-custody form, original (if new combined chain-of-custody traffic reports are used, two copies will be required)
  - Traffic report forms, two copies

- SAS packing list, two copies
- Sample tags

- Retained by project manager:
  - Sample identification matrix
  - Field log books (at completion of project)

- Sent to CH2M HILL documentation coordinator:
  - Chain-of-custody form, two copies
  - Traffic report forms, original and one copy
  - SAS packing list, original and one copy

7.2.7 Site Manager's Responsibility

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

7.3 Laboratory Custody Procedures for the Contract Laboratory

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

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

- Designation of a sample custodian

- Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt)

- Laboratory sample tracking and documentation procedures
- Secure sample storage (of the appropriate environment—refrigerated, dry, etc.)
- Proper data logging and documentation procedures including custody of all original laboratory records

7.4 Final Evidence Files Custody Procedures

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

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

Section 8
Calibration Procedures and Frequency

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

8.1 Special Analytical Services

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

8.2 Field Instruments

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

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

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

Section 9
Analytical Procedures

All samples collected during field sampling except bioassay samples will be analyzed by the CLP or CRL.

9.1 Special Analytical Services

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

Bioassay methods are contained in Appendix E.

9.2 Field Instruments

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

Section 10
Internal Quality Control Checks

10.1 Special Analytical Services

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

10.2 Field Instruments

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

Section 11
Data Reduction, Validation, and Reporting

11.1 Data Reduction

11.1.1 Laboratory Analysis

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

11.1.2 Field Measurements

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

11.2 Data Validation

11.2.1 Laboratory Analysis

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

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

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

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

11.2.2 Field Measurements

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

11.3 Data Reporting

11.3.1 Laboratory Analysis

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

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

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

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

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

11.3.2 Field Measurements

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

Section 12
Performance and System Audits

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

12.1 External Audits

12.1.1 CLP Laboratories

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

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

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

12.1.2 Field Audits

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

12.2 Internal Audits

12.2.1 Field Audits

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

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

The site manager will evaluate the following items:

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

Following the audit, the auditor will review the preliminary results of his evaluation with the field team leader. The auditor will prepare an audit report containing the results of the evaluation and recommendations for any corrective action. The audit report will be

reviewed by the project manager. The site manager shall implement any agreed upon corrective action. The site manager will also be responsible for verifying the implementation of the corrective action. Any noncompliance with standard procedures shall be identified and corrected.

Section 13
Preventive Maintenance

13.1 Laboratory Instruments

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

13.2 Field Instruments

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

Section 14
Specific Routine Procedures to Assess Data Precision, Accuracy and Completeness

14.1 Field Measurements

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

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

14.2 Laboratory Data

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

14.2.1 Precision

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

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

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

14.2.2 Accuracy

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

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

Where:

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

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

C = The amount of the spike added.

14.2.3 Completeness

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

14.3 Project Assessment

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

Section 15
Corrective Actions

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

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

15.1 Sample Collection/Field Measurements

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

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

- Evaluating all reported nonconformances;
- Controlling additional work on nonconforming items;
- Determining disposition or action to be taken;
- Maintaining a written log of nonconformances in a field record book;
- Reviewing nonconformance reports and corrective actions taken;

- Ensuring nonconformance reports are included in the final site documentation in project files.

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

Corrective action for field measurements may include:

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

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

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

15.2 Laboratory Analyses—Laboratory Corrective Actions

For the CLP Special Analytical Services (SASs), Corrective action is implemented at several different levels. The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is

discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures. If CRL is used, corrective actions by CRL will be implemented according to CRL Standard Operating Procedures (SOPs).

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

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

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

Section 16

Quality Assurance Reports to Management

The preparation of a separate QA report for this project is not anticipated. The summary report for the startup sampling will contain separate QA sections that summarize data quality information collected during startup. Changes in the Field Sampling Plan or QAPjP will be documented in the report. Qualified data will be summarized in tables.

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

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

APPENDIX A
PROCESS STARTUP SAMPLING PLAN

Appendix A—Process Startup Sampling Plan

Provided under separate cover

APPENDIX B
SAS REQUEST FORMS

Appendix B—SAS Request Forms

Provided under separate cover

APPENDIX C
FIELD TESTING PROCEDURES

Appendix C—Field Test Procedures

Provided under separate cover

APPENDIX D
SAMPLE DOCUMENTATION AND

Appendix D—Sample Documentation and Packing and Shipping Instructions

Provided under separate cover

APPENDIX E
BIOASSAY METHODS

Appendix E—Bioassay Methods

Provided under separate cover

Quality Assurance Project Plan
Appendices

Onalaska Municipal Landfill
Onalaska, Wisconsin

Groundwater Remedial Action Startup Testing
WA No. 38-5NL5 / Contract No. 68-W8-0040

January 1994

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MKE100130B8.WP5

APPENDIX A
PROCESS STARTUP SAMPLING PLAN

Section 1
Introduction

This plan provides the information necessary to monitor the overall startup operation of the Onalaska Groundwater Treatment System. Sampling will be conducted by CH2M HILL to monitor process operations that are not monitored by the treatment system subcontractors and to verify that the overall performance of the treatment system as designed meets regulatory requirements. R. E. Wright (CH2M HILL subcontractor) will also be collecting samples during process startup and will prepare a startup sampling plan. *When the subcontractor startup plan becomes available, this plan should be modified as necessary to complement the subcontractor startup plan.* For additional information on the groundwater treatment system, see the Operations and Maintenance Manual. Requirements for sampling, monitoring, and reporting are discussed in this plan.

The guidelines presented in this monitoring program are based on the best information available at the time of design and may not account for unanticipated field conditions. Therefore, the results of each data set collected shall be evaluated in the context of satisfying the intent of regulatory and contract requirements.

MKE1001305E.WP5

Section 4
Startup Monitoring Objectives

The objectives of the startup sampling plan are to:

- Document the compliance of treatment system discharge with effluent limits and treatment system performance requirements before discharging effluent to Bullet Chute
- Document continuing compliance for important indicator parameters
- Document treatment system efficiency
- Document the performance of the process during startup

Process water and solids will be monitored during process startup to verify that project objectives are being met. Monitoring will include both sample collection and field and laboratory analyses. Sampling activities are designed to meet WDNR sampling requirements as outlined by the WDNR.

MKE10013061.WP5

Section 5

Parameters Monitored

Monitoring will involve the analysis of process water samples and plate and frame filter cake. Four groups of analyses will be performed on the water samples: field parameters, organic and inorganic contaminants, conventional parameters, and whole effluent toxicity bioassays. The filter cake will undergo the analyses required for landfill disposal. The parameters that make up each analytical group are listed below by group. The quality assurance project plan (QAPP) describes the analytical methods, detection limits, quality control/quality assurance (QA/QC) sampling, and data quality objective (DQO) levels. Table 3 of the QAPP contains the contract laboratory analytical methods and Appendix B contains the Special Analytical Services (SAS) forms and QA/QC sampling requirements.

5.1 Water Sample Parameters

Field Analyses

The following conventional parameters will be analyzed in the field:

- pH and temperature
- Specific conductance

Organic and Inorganic Analyses

Process startup will occur in three phases. Different sets of organic and inorganic water sample analyses will be performed during the three phases of process startup:

- Phase 1—Prior to discharge: A complete priority pollutant scan will be performed on the first effluent from the process (see Table 6-1 for additional toxicity and conventional analyses required).

- Phase 2—Pumping at reduced rate: Monitoring will be limited to organic pollutants with effluent limits for process discharge to the Bullet Chute of the Black River (see Table 6-1 for conventional analyses required).

- Phase 3—Pumping at normal rate: The effluent will be monitored for BETX compounds and iron (see Table 6-1 for conventional analyses required).

Table 5-1 lists the inorganic and organic compounds and their WDNR-computed effluent limits.

Conventional Analyses

The following conventional analyses will be performed to monitor process operations and demonstrate compliance with effluent limits:

- Total dissolved solids (TDS)
- Total suspended solids (TSS)
- Nitrate, ammonia, 5-day biochemical oxygen demand (BOD_5)

Table 5-2 lists effluent limits for ammonia.

Toxicity Tests

A whole effluent toxicity test battery will be conducted on the first effluent from the process. Testing will include both acute and chronic bioassays. Acute toxicity testing will be performed with three freshwater species. Chronic toxicity testing will be performed with two freshwater species (*Ceriodaphnia dubia* and larval fathead minnows). Whole effluent toxicity bioassays will be performed in accordance with the laboratory procedures in Appendix E.

5.2 Filter Cake Sample Parameters

TCLP, paint filter, and percent solids analyses will be conducted on the filter cake solids generated during startup.

MKE10013062.WP5

Section 6

Sampling Summary, Location, and Frequency

6.1 Sampling Program Summary

Table 6-1 summarizes the sampling program and lists the sample locations, sample frequencies, sample analyses, and general data evaluations. Table 6-2 lists the sampling locations and purposes for sampling.

6.2 Groundwater Treatment System Sampling Locations

Process monitoring will involve the collection of samples from eight locations. Sample locations are provided for sampling process influent, clarifier influent, clarifier effluent, stripper tower influent, stripper tower effluent, filter press influent, filter cake, and process effluent. The sampling locations are shown in Figure 6-1.

Process influent samples will be collected at the valve provided at sample point (1) and shown in Figure 6-2. Process influent samples will be collected before the effluent reaches the aeration tank and will represent the combined flow of the groundwater extraction system.

Clarifier influent and effluent samples will be taken at the valve located at sample point (2) and the valve located at sample point (3). Figures 6-3 and 6-4 show the locations of the valves.

Stripper influent samples will be collected from the same sample point (3) as the clarifier effluent, located in the clearwell discharge pipe after the clearwell pump. Stripper effluent will be sampled at the valve located in the stripper effluent pipe (4) and shown in Figure 6-5. Process effluent samples will be collected before the carbon column at the same location as stripper tower effluent samples (4). Process effluent samples will also be collected after the carbon column at the valve located at sample point (5).

Filter press influent samples will be collected from the valve located in the pipe between the sludge tank and sludge feed pump (6). Effluent (filtrate samples) will be collected from the valve located at sample point (7). Filter cake samples will be collected from the solids bin (8). Filter press sample locations are shown in Figure 6-6.

6.3 Sampling Frequency

Whole effluent toxicity test batteries, a priority pollutant scan, and testing for all parameters will be conducted immediately upon treatment system startup. After startup, samples for more select analyses will be collected daily.

Table 6-1
Startup Sampling Summary
Onalaska Startup Sampling Plan
Onalaska, Wisconsin

Objective	Sample Location	Sample Frequency	Sample Analyses	Data Evaluation	Number of Samples
Compliance Monitoring					
Phase 1: Document compliance with effluent limits and requirements prior to discharge	Influent and effluent; (1), (4), (5)	Collect samples within 24 hours of pumps operating and overall plant startup	• Acute and Chronic Bioassay (effluent at (4) and (5)) • Complete priority pollutant scan • All parameters: pH, TSS, TDS, Ammonia as N, Temp, BOD_5, hardness, nitrate	Pass/fail discussion compare to effluent limits	2 3 3
Phase 2: Document performance of process during startup	Influent and effluent before and after carbon column; (1), (4), (5)	Resume pumping at reduced rate—daily for 2 days	Parameters with effluent limits (Table 5-1) and pH, temperature, BOD_5, and TSS	Tabulate data, compute percent removal and compare to Table 5-1 effluent limits	6
Phase 3: Demonstrate successful startup performance of treatment process	Influent and effluent before carbon column; (1), (4), (5)	Increase pumping to normal rate—daily for 12 days (may change to 5 days)	BTEX compounds, pH, ammonia, iron, temperature, BOD_5, TSS	Compute percent removal and compare to effluent limits in Table 5-1	36
Performance Monitoring					
Document treatment system efficiency (% pollutant removal) for BTEX, VOC, and iron	See 2nd item above				
Clarifier performance monitoring	Clarifier influent/effluent; (2), (3)	Daily (for 14 days)	Iron, TSS, TDS, pH, flow, temp. (filtered and unfiltered iron @ 3)	Mass balance for iron removal from clarifier	28
Stripper performance monitoring	Stripper influent; (3)	Daily (for 14 days)	BTEX, ammonia, flow, temp,	Mass balance BTEX and ammonia removal	14
Filter press performance monitoring	Press influent/solids bin; (6), (7), (8)	As required	• Flow, TSS (6), (7) • % solids, bulk density, vol (8)	Solids mass balance	6 3
Process mass balance	Before and after each process and at groundwater extraction wells	Daily (for 14 days)	Flow	Mass balance computations	
Additional Testing					
Landfill disposal testing	Solids bin; (8)	First filter cake	TCLP, paint filter	Landfill disposal approval	1

Table 6-2
Process Sampling Locations
Onalaska Municipal Landfill

Sample Point	Location	Line No./ Pipe Size and Type	Purpose
(1)	Process influent/aeration tank influent line—Inside treatment plant building	11 (8″ PVC)	Determine overall percent removals. Monitor levels of constituents in the influent.
(2)	Clarifier influent—Outside treatment plant building	12 (10″ PVC)	Monitor the levels of iron in the aeration tank effluent.
(3)	Clarifier effluent/air stripper influent—Inside building. Located just prior to clearwell.	16 (8″ PVC)	Determine percent removal of iron. Monitor the levels of iron going to the air stripper tower. Monitor the levels of VOCs going to stripper tower.
(4)	Stripper effluent—Outside building	17 (10″ PVC)	Determine percent removal of VOCs in stripper tower. Monitor levels of VOCs in stripper tower effluent.
(5)	Process effluent	17 (10″ PVC)	Determine if effluent concentrations are below regulatory levels.
(6)	Filter press influent—Inside treatment building	21 (3″ GS)	Monitor percent solids to filter press.
(7)	Filter press effluent (titrate)—Inside treatment plant	48 (6″ PVC)	Monitor percent solids in recycle.
(8)	Solids bin—Inside treatment plant	---	Monitor solids generated. Determine if filter cake meets landfill disposal criteria.

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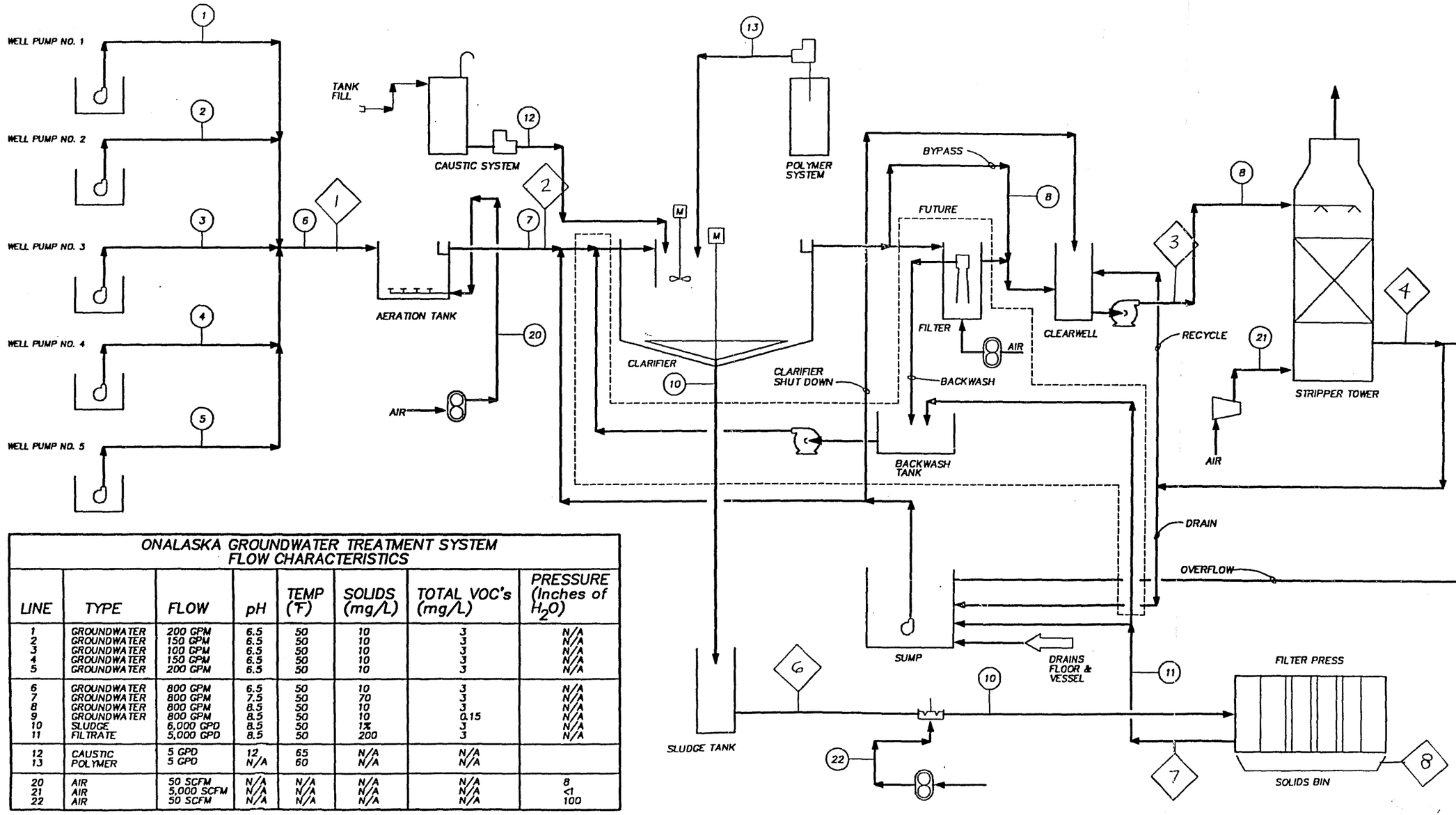

ONALASKA GROUNDWATER TREATMENT SYSTEM FLOW CHARACTERISTICS

LINE	TYPE	FLOW	pH	TEMP (F)	SOLIDS (mg/L)	TOTAL VOC's (mg/L)	PRESSURE (Inches of H_2O)
1	GROUNDWATER	200 GPM	6.5	50	10	3	N/A
2	GROUNDWATER	150 GPM	6.5	50	10	3	N/A
3	GROUNDWATER	100 GPM	6.5	50	10	3	N/A
4	GROUNDWATER	150 GPM	6.5	50	10	3	N/A
5	GROUNDWATER	200 GPM	6.5	50	10	3	N/A
6	GROUNDWATER	800 GPM	6.5	50	10	3	N/A
7	GROUNDWATER	800 GPM	7.5	50	70	3	N/A
8	GROUNDWATER	800 GPM	8.5	50	10	3	N/A
9	GROUNDWATER	800 GPM	8.5	50	10	0.15	N/A
10	SLUDGE	6,000 GPD	8.5	50	1%	3	N/A
11	FILTRATE	5,000 GPD	8.5	50	200	3	N/A
12	CAUSTIC	5 GPD	12	65	N/A	N/A	
13	POLYMER	5 GPD	N/A	60	N/A	N/A	
20	AIR	50 SCFM	N/A	N/A	N/A	N/A	8
21	AIR	5,000 SCFM	N/A	N/A	N/A	N/A	<1
22	AIR	50 SCFM	N/A	N/A	N/A	N/A	100

CH2M HILL

DSGN P.M.BOERSMA
DR PFD-1 D.OXLEY
CHK S.M.KEITH
APVD S.M.KEITH

NO.	DATE	REVISION	BY	APVD

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GROUNDWATER REMEDIAL ACTION
ONALASKA MUNICIPAL LANDFILL SITE
ONALASKA TOWNSHIP, WISCONSIN

FIGURE 6-1
PROCESS FLOW DIAGRAM

SHEET 48
DWG NO. 01-N-02
DATE JULY 1992
PROJ NO. GL065509.FD

1-L8

07-08-92 E:\GL5607\HOLDING\PID-1.DWG

MCP ◆

ON/ OFF-RESET HS 1-1-1 (5) | QL 1-1-1 OO (5) | QA 1-1-1 FAIL (5) | AUTO-DIALER | FY 1-4-1 ADD (2) | FIR/SHL 1-4 | "HIGH/LOW" FAHL 1-4 | AUTO-DIALER

"CLEARWELL OR SUMP LEVEL HIGH, STOP PUMPS" (4) N-05 4-A | I "RUN" (5) | "PUMP FAIL" (COMMON) | FSL 1-3-1 (5)

ON/ OFF-RESET HS 2-2-1 | QL 2-2-1 OO | QA 2-2-1 FAIL | AUTO-DIALER

"ON/OFF" (2) | I "RUN" | "FAIL" | N-06 5-A | OR "BLOWER OR FAN FAILED, STOP PUMPS" | "STRIPPING TOWER FAN FAIL"

2-A N-04

ON/ OFF-RESET HS 2-2-2 | QL 2-2-2 OO | QA 2-2-2 FAIL

"ON/OFF" (2) | I "RUN" | "FAIL"

(10) (4) (8) (2)

120V PI 1-2-1 | FIT/QI 1-3-1 | FE 1-3-1

4"-PE-1 | V111 | 4"-PVC-6 | V112 | 8"-PVC-11

"ON/OFF" CS-1 P-1-1-1 WELL PUMP NO. 1

120V PI 1-2-2 | FIT/QI 1-3-2 -A | FE 1-3-2

4"-PE-2 | V121 | 4"-PVC-7 | V122

CS-1 P-1-1-2 WELL PUMP NO. 2

120V PI 1-2-3 | FIT/QI 1-3-3 -A | FE 1-3-3

4"-PE-3 | V131 | 4"-PVC-8 | V132

CS-1 P-1-1-3 WELL PUMP NO. 3

120V PI 1-2-4 | FIT/QI 1-3-4 -A | FE 1-3-4

4"-PE-4 | V141 | 4"-PVC-9 | V142

CS-1 P-1-1-4 WELL PUMP NO. 4

120V PI 1-2-5 | FIT/QI 1-3-5 -A | FE 1-3-5

4"-PE-5 | V151 | 4"-PVC-10 | V152

8"-PVC-11

BELOW GROUND | INSIDE BLDG. | CS

CS-1 P-1-1-5 WELL PUMP NO. 5

INSIDE | OUTSIDE

40° | 2"-SS-31 | 40°

T-2-1 AERATION TANK

40° | 10"-PVC-12 | 2-A N-04 TO CLARIFIER

V163 | 3"-PVC-19 | 2-B N-08 TO PLANT DRAIN SUMP

FILTER, TYP. T-2-2-1-1 | T-2-2-1-2 SILENCER, TYP.

PI 2-3-1 | (2) "ON/OFF" | CS-1 M-2-2-1 AERATION TANK BLOWER

T-2-2-2-1 | T-2-2-2-2

PI 2-3-2 | (2) "ON/OFF" | CS-1 M-2-2-2 AERATION WELL BLOWER

CS | 6"-PVC-60 | 6"-PE-60 TO AERATION WELLS

6"-PVC-62 | 6"-PE-62

CH2M HILL	DSGN KEITH/OHLSSON; DR PDC OXLEY/OHLSSON; CHK R.NAGEL; APVD S.M.KEITH	NO.	DATE	REVISION	BY	APVD

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GROUNDWATER REMEDIAL ACTION
ONALASKA MUNICIPAL LANDFILL SITE
ONALASKA TOWNSHIP, WISCONSIN

FIGURE 6-2
PROCESS & INSTRUMENTATION DIAGRAM
WELL FIELD PUMPS & AERATION

SHEET 49
DWG NO. 01-N-03
DATE JULY 1992
PROJ NO. GL065607.FD

08-26-92 E:\83607\HOLDING\PID-2.DWG

MCP ◆

HS 9-1 OO — "CLEARWELL OR SUMP LEVEL HIGH, STOP PUMP" N-05 4-B — "BLOWER OR FAN FAILED, STOP PUMP" N-04 2-A — "RUN"

HS 3-2 ON/OFF-RESET — QL 3-2 OO — QA 3-2 FAIL — (2)

HS 3-1 ON/OFF-RESET — "RUN"

QL 3-1 OO — QA 3-1 FAIL — NAH 3-1 TORQUE — AUTO-DIALER — AUTO-DIALER

AAHL 3-3 pH — AI 3-3 pH — TALL 10-1 — AUTO-DIALER

QL 3-4 OO — "TANK LEVEL HIGH, STOP PUMP" N-07 6-A

P-9-1 POLYMER PUMP — POLYMER DRUM — INSIDE OUTSIDE — V271 — V272 — 1/2"-PVC-53 — 40° — D

M-3-2 MIXER — M — AS — M-3-1

"ON" (2) — 75° — OUTSIDE INSIDE

"ON/OFF" — NSH 3-1 TORQUE — NSHH 3-1 TORQUE — "HIGH/LOW pH" — CS-1 "STOP"

FROM AERATION BASIN N-03 2-A — 10"-PVC-12 — 40°

AIC 3-3 pH SHL — 120V — AE 3-3 pH

INSIDE OUTSIDE — FUTURE — FROM BACKWASH TANK N-05 4-C — 3"-PVC-24 — 40°

OUTSIDE INSIDE

T-3-1 CLARIFIER

BELOW GROUND INSIDE — A

FROM PLANT DRAIN SUMP N-08 9-A — 6"-PVC-41

3"-PE-20 — V261 — V262 — F — 3"-GS-20

FP-3-4 ** — 120V — 100 — ** P-3-4 SLUDGE TRANSFER PUMP

TO SLUDGE TANK 3-B N-07 — 3"-GS-20

TO PLANT DRAIN SUMP 4"-PE-50 3-C N-08

S — 10"-PVC-13 — TO FILTER/CLEARWELL 3-A N-05

(2) "ON/OFF"

OUTSIDE INSIDE — 75° — 2"-SS-27 — TRUCK FILL — V231

"LOW-LOW"

75° — LG 10-1 — T-10-1 CAUSTIC STORAGE TANK — TE 10-1 — TISL/LL 10-1 — 3"-SS-28

CALIBRATION COLUMN ** T-10-2 — A

FP-10-2 ** — 120V

V233 — 1"-SS-29 — V236 — 1/2"-SS-30 — AS — ** P-10-2 CAUSTIC PUMP

V232 — TANK HEATING STRIPS — 120V

V235

CONTAINMENT SUMP

TO PLANT DRAIN SUMP 10A N-08

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	DR PED-2 OXLEY/OHLSSON				THIS DOCUMENT, AND THE IDEAS AND DESIGNS INCORPORATED HEREIN, AS AN INSTRUMENT OF PROFESSIONAL SERVICE, IS THE PROPERTY OF CH2M HILL AND IS NOT TO BE USED, IN WHOLE OR IN PART, FOR ANY OTHER PROJECT WITHOUT THE WRITTEN AUTHORIZATION OF CH2M HILL. ©CH2M HILL	IF NOT ONE INCH ON THIS SHEET, ADJUST SCALES ACCORDINGLY.	ONALASKA MUNICIPAL LANDFILL SITE ONALASKA TOWNSHIP, WISCONSIN
	CHK R.NAGEL						
	APVD S.M.KEITH	NO.	DATE	REVISION	BY	APVD	

FIGURE 6-3
PROCESS & INSTRUMENTATION DIAGRAM CLARIFICATION & CAUSTIC SYSTEM

SHEET 50 | DWG NO. 01-N-04 | DATE JULY 1992 | PROJ NO. CL855602.FD

MCP ◆

LIC 4-4 AM — A

LSH 4-4 — A

"SUMP LEVEL HIGH-HIGH, STOP PUMPS" N-08 7-A

AUTO-DIALER

OR

LAH 4-4

"CLEARWELL OR SUMP LEVEL HIGH, STOP PUMPS"

4-B N-04

4-A N-03

N-06 5-B

LSM 4-4 — A

LSL 4-4 — A

HS 4-5 — ON/OFF-RESET/AUTO

QL 4-5 — OO

QA 4-5 — FAIL

AUTO-DIALER

"STOP"

(2)

"START"

"FAN FAILED, STOP PUMP"

"RUN"

FROM PLANT DRAIN SUMP N-08 9-B — 6"-PVC-42

OUTSIDE | INSIDE

FROM CLARIFIER N-04 3-A — 10"-PVC-13 — V311 — 10"-PVC-52

LIT 4-4 — 120V

LE 4-4

4"-PVC-44 — 10"-PVC-14

S

FUTURE

PI 4-2

T-4-1 FILTER

T-4-1 BACKWASH TANK

4"-PVC-23

P Z

CS-1

M-4-2 FILTER COMPRESSOR

P-4-3 BACKWASH PUMP

3"-PVC-XX

8"-PVC-26

V332

V333

T-4-4 CLEARWELL

4"-PVC-25

V321 — 10"-PVC-15

(2) "ON/OFF"

V323

CS

P-4-5 CLEARWELL PUMP

P Z 40

LCV 4-4

3

8"-PVC-16 — TO STRIPPING TOWER 4-A N-08

TO PLANT DRAIN SUMP 4-B N-08

TO CLARIFIER 4-C N-04 — 3"-PVC-24

4-D N-08 TO PLANT DRAIN SUMP

BELOW GROUND | ABOVE GROUND

FROM STRIPPING TOWER N-06 5-B — 10"-PVC-35

DSGN	KEITH/OHLSSON						
DR PID-3	OXLEY/OHLSSON						
CHK	R.NAGEL						
APVD	S.M.KEITH	NO.	DATE	REVISION	BY	APVD	

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GROUNDWATER REMEDIAL ACTION

ONALASKA MUNICIPAL LANDFILL SITE
ONALASKA TOWNSHIP, WISCONSIN

FIGURE 6-4

PROCESS & INSTRUMENTATION DIAGRAM
FILTRATION AND CLEARWELL

SHEET 51
DWG NO. 01-N-05
DATE JULY 1992
PROJ NO. CLD65602.FD

08-29-92 I:\63602\VIGLDING\PID-4.DWG

MCP ◆

HS 11-1 OOA
QL 11-1 OO
LAL 5-2
PAL 11-2
HS 5-3 ON/OFF-RESET
QL 5-3 OO
QA 5-3 FAIL
AUTO-DIALER
AUTO-DIALER
(2)
I
(2)
I
"TOWER FAN FAILED, STOP PUMPS"
5-A N-03
5-B N-05
LAH 5-2
AIR/SHL pH 5-4
AAHL pH 5-4
AUTO-DIALER

FROM PLANT DRAIN SUMP
N-05 9-C 8"-PVC-43
FROM CLEARWELL
N-05 4-A 8"-PVC-16
30"-FRP-32
T-5-3 SILENCER
(2)
"ON/OFF"
M-5-3 STRIPPING TOWER FAN
CS-1
30"-FRP-32
INSIDE OUTSIDE
V
T-5-1 AIR STRIPPING TOWER
1"-PVC
V421
S
180
DPI 5-1
180
LSH 5-2
LSL 5-2
OUTSIDE INSIDE
10"-PVC-33
40°
V433
V432
10"-PVC-17
10"-PE-18
TO OUTFALL
S
4
S
5
CARBON COLUMNS
TO SUMP (START-UP)
10"-PVC-34 5-A N-08
TO CLEARWELL (CLEANING)
10"-PVC-35 5-B N-05
TO SUMP
5-C N-08
◆ pH AIT 5-4
120V
◆ pH AE 5-4
INSIDE BUILDING CS BELOW GROUND SUBMERGED
V442
40°
3"-PVC-45
V440
V441
PI 11-2
PSL 11-2
PSH 11-2
PSL 11-2
(2)
"STOP"
"START"
(2)
"ON/OFF"
PSV 11-2
T-11-2 SERVICE WATER PRESSURE TANK
V443 V444
CS-1
P-11-1 SERVICE WATER PUMP
2"-GS-46
HV1
1"-GS HOSE DROPS, TYP.
HV2
TO PRECOAT TANK
11-A N-07

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GROUNDWATER REMEDIAL ACTION
ONALASKA MUNICIPAL LANDFILL SITE
ONALASKA TOWNSHIP, WISCONSIN

FIGURE 6-5
PROCESS & INSTRUMENTATION DIAGRAM
AIR STRIPPING/SERVICE WATER SYSTEM

SHEET 52
DWG NO. 01-N-08
DATE JULY 1992
PROJ NO. GL065602.FD

08-25-92 E:\A5602\HOLDING\PID-5.DWG

MCP ◆

HS 6-5 OO
HS 6-6 OO
QL 6-5 OO
QL 6-6 OO
LI 6-1
LSH 6-1 A "STOP SLUDGE PUMP" 6-A N-04
LSL 6-1 A LAL 6-1
LIT 6-1 120V
LE 6-1
T-6-1 SLUDGE TANK
4"-PVC-37
V514 2"-PVC-61
V513 3"-GS-21
V512 2"-PVC-35
V511
SLUDGE FROM CLARIFIER N-04 3-B 3"-GS-20 3"-GS-38
S 6
FP-6-2** 100 120V
** ZS 6-2 STROKE
** P-6-2 SLUDGE FEED PUMP
V532 2"-GS-21
V533 6"-GS-21
** PSL 6-3
** HPU
** PI 6-4-1
** FILTER PRESS M-6-3
** PRV-6-4-1
V521
V524
V525
V527
V528
V526
CS V519
V520
S 7 6"-PVC-48
** PI 6-4-2
** PRV-6-4-2
V522 V523
V529
V530
4"-PVC-59
8 SOLIDS BIN
TO PLANT DRAIN SUMP 6-A N-08
TO PLANT DRAIN SUMP 6-B N-08
TO PLANT DRAIN SUMP 6-C N-08
V531
6"-PVC-48
COMPRESSED AIR N-08 8-A 3/4"-GS-51
(2) "ON/OFF"
PRECOAT MIXER ** M-6-5 M CS-1
FROM SERVICE WATER SYSTEM N-06 11A 2"-GS-46
V515
4"-PVC-56
4"-PVC-57 V516
V517 8"-PVC-58
** T-6-5 PRECOAT TANK
(2) "ON/OFF"
PI 6-7
V518
PE 6-7
CS-1
** P-6-6 RECIRCULATION PUMP
6"-PVC-55

CH2M HILL

DSGN	KEITH/OHLSSON				
DR PD	OXLEY/OHLSSON				
CHK	R.NAGEL				
APVD	S.M.KEITH	NO.	DATE	REVISION	BY APVD

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GROUNDWATER REMEDIAL ACTION
ONALASKA MUNICIPAL LANDFILL SITE
ONALASKA TOWNSHIP, WISCONSIN

FIGURE 6-6
PROCESS & INSTRUMENTATION DIAGRAM
SLUDGE DEWATERING

SHEET S3
DWG NO. D1-N-07
DATE JULY 1992
PROJ NO. GL065602.FD

The purpose of sampling daily is to assess the performance of the individual processes within the treatment system and the water quality of the process effluent before and after the carbon column and before discharge. Phase 1 sampling will be conducted the first day. Phase 2 sampling will be conducted on day 2 and day 3. Phase 3 sampling will be conducted until successful process startup has been demonstrated. Daily sample collection will commence at 8 a.m.

If CH2M HILL demonstrates that the WDNR-specified effluent limits are not exceeded for a period of 2 weeks, the startup sampling will be discontinued and sampling following the Operations and Maintenance Sampling Plan will begin.

If WDNR specified effluent limits are exceeded in any week, sampling will continue for another week. After that week, the sampling schedule will be re-evaluated.

6.4 Methods

Sample collection methods are discussed in Section 7.

MKE1001308F.WP5

Section 7

Sampling Equipment and Procedures

A summary of samples to be collected (including QA/QC samples) at the Onalaska site is presented in the QAPP. Section 6 of this plan summarizes startup sampling.

General procedures for performing field tests and collecting water quality samples are described below. Additional details related to specific sampling and decontamination procedures, sampling frequencies, and analytical requirements are discussed in Section 12 and Section 6 of this document and in the QAPP.

All sample locations will be sampled using the same equipment and procedures. All water and solid samples will be collected using the equipment identified below.

7.1 Sampling Equipment

The following items are required to sample process water:

- Sample collection and field testing equipment
  - Quick connect fittings and sample tubing
  - Sample containers with preservatives and labels provided by the analytical laboratory
  - Clean glass or stainless steel beaker for temperature, conductance, dissolved oxygen, and pH measurements
  - Ice chest, ice
  - Thermometer (0° to 50°C range)
  - Conductance and pH meter
  - Dissolved oxygen meter
  - Process water quality field test data sheets
  - Decontamination solutions, containers, brushes, etc.

- Sample Records

  - Daily activity logs and field notebooks
  - Laboratory log sheets
  - EPA chain-of-custody forms
  - EPA Region 5 sample tags

The following items are required to sample the process solids:

- Equipment needed to sample solids

  - Uncoated scoop for sample collection
  - Stainless steel bowl
  - Stainless steel spoon for media transfer to sample container
  - Sample containers

- Sample records

  - Daily activity logs and field notebooks
  - Laboratory log sheets
  - EPA chain-of-custody forms
  - EPA Region 5 sample tags

7.1.1 Sampling Equipment Description

Each sample point assembly has a quick-connect fitting for attachment of the discharge line for the discharge of water into a sample container.

7.1.2 Sampling Equipment Calibration

The calibration procedures and frequency of calibration for sampling equipment are provided in Appendix C.

7.1.3 Sampling Equipment Preventive Maintenance

Each piece of sampling equipment will be tested to verify that it is in proper working order before it is sent to the site. Equipment will also be tested before each use. The instrument operator's manual will dictate the frequency of calibration and maintenance.

7.2 Sampling and Measurement Procedures

The general sampling procedures and sequence described below are recommended as a guide to sampling each port. Process measurements and sampling will proceed in the following sequence:

1. Organize and decontaminate sampling equipment and calibrate instruments.

2. Purge port by opening the valve and briefly releasing process water from the sample port.

3. Perform field analyses (record pH, temperature, conductance, and flow readings).

4. Collect water samples per procedures outlined below (see Water Sample Collection Procedures).

5. Preserve samples for storage and laboratory analyses (aqueous VOC samples must have preservative added to the vials before filling).

6. Complete sample records and chain-of-custody forms and seals.

7. Ship samples by overnight courier to analytical laboratory.

7.2.1 Water Sample Collection Methods

Samples will be collected after purging the port. Volatile organic analysis (VOA) vials will be filled first; containers for filtered metals will be filled last. In all cases, the samples will be collected directly from the discharge line. To collect the VOA samples:

- Reduce volume of discharge from the pump sample line by adjusting the control box until a trickle flow is achieved.

- Add preservative (HCl) to the vials.

- Place the mouth of the VOA vial at the end of the discharge tube and allow bottle to fill slowly.

- Fill vial in a steady, gentle stream with a minimum of agitation.

- Fill until a meniscus forms on the mouth of the VOA vial.

- Cap the vial, and check for air bubbles by inverting the vial and tapping on the palm of the hand. If bubbles are present, repeat procedure until a bubble-free sample is obtained.

To collect nonfiltered samples:

- Increase discharge rate and fill the sample containers to the shoulder.
- Add preservative as required.

To collect filtered metals samples:

- Attach an inline sample field filter directly to the pump discharge tube per manufacturer's instructions (Appendix D).
- Increase discharge rate and fill the metals sample container to the shoulder.
- Add preservative (HNO_3) to the sample.

7.2.2 Solids Sample Collection Methods

Solids samples will be collected by dipping an uncoated scoop into the tank or bin holding the solids. The solids will be transferred from the scoop to a stainless steel bowl and then transferred from the bowl to the sample jars using stainless steel spoons.

7.3 Sample Shipping

Coolers will be used to transport samples from the field to the analytical laboratory. Samples requiring preservation by cooling will be kept cold at a constant temperature (4°C).

All shipments will be accompanied by a chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the sampler.

All shipping coolers must have two Region 5 custody seals placed over the lid opening, one on each side.

The copy of the airbill accompanying each shipping container will be retained as part of the permanent documentation. Commercial carriers are not required to sign the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

MKE10013464.WP5

Section 8
Field Measurements and Screening

SOPs for OVA, pH/temperature, dissolved oxygen, and conductivity measurement and screening techniques are provided in Appendix C of the QAPP.

MKE10013465.WP5

Section 9

Sample Container Preparation, Sample Preservation, and Maximum Holding Time

9.1 Bottle Requirements

Table 6 of the QAPP lists the sample containers used for each of the analyses. The sample containers (bottles) used for this sampling effort will be prepared according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990.*

9.2 Sample Preservation and Holding Time

Table 6 of the QAPP summarizes the requirements for sample containers, preservatives, and sample holding times. Sample containers that are certified by the laboratories as precleaned will be used. Preservatives will be prepared using reagent grade chemicals. Samples will be kept iced to maintain a temperature of 4°C for preservation.

MKE10013092.WP5

Section 10

Sample Documentation and Custody Procedures

10.1 Sample Identification System

A sample numbering system devised by CH2M HILL will be used to identify each sample, including duplicates and blanks. A list of sample identification numbers will be maintained in the field logbook by the field activity manager. Each CH2M HILL sample number will consist of three components, as described below.

Each sample will have up to a four-digit code corresponding to the media, (and identifying the Onalaska Municipal Landfill site), followed by a three-digit code identifying the sample location. The last three digits are the sequential sample number. Sample numbers will not be repeated within a sample station, medium, or among differing media. Duplicate samples will not be distinguished within the sample numbers, but will be distinguished through the subsample identification within the sample tracking and data management systems. This is done so that no bias is given to these samples during analyses. The media letter codes and reserved sample numbers are as follows:

- PW—Process Water Sample
- PS—Process Solids Sample

For QA/QC samples the following designations are added to the end of the sample number:

- Blanks—B
- Duplicates—D
- MS/MSD—MS

Examples of sample numbers are as follows:

- PWOL-V08-S002—Groundwater sample collected from the Onalaska Municipal Landfill site, Valve No. 8, Sample No. 2

- PSOL-V01-S01—Process solids sample collected from the Onalaska Municipal Landfill site, Valve No. 1, Sample No. 1

- PWOL-V05-S002-MS—Groundwater sample collected from the Onalaska Municipal Landfill site, Valve No. 5, Sample No. 2, matrix spike/matrix spike duplicate.

10.2 Initiation of Field Custody Procedures

EPA Region 5 chain-of-custody protocols, as described in the National Enforcement Investigations Center (NEIC) Policies and Procedures, EPA-330/9-78-DDI-R, revised June 1985, will be followed for all samples collected for analysis. The custody procedures are described in Section 7.0 of the QAPP.

10.3 Field Activity Documentation and Logbook

The field logbook, as described in the QAPP, will be initiated at the start of the first startup sampling activity and should be used to record onsite activities during process startup. The field logbook is a controlled document that becomes part of the permanent site file.

Instructions for recording information into the field logbook are provided in Section 7.2.4 of the QAPP, "Field Log Book." The following additional information should also be recorded:

- Arrival and departure times of site visitors
- Arrival and departure times of equipment
- Project number (GLE65624.SU)
- Equipment calibration information (equipment type and I.D. number, calibration standards used, instrument response, problems and repairs, etc.)
- A summary of sample line purging (type and length of tubing used, if any, time purged, flow, and volume)
- Identification of the startup phase (I, II, or III)
- Sample bottle QC lot numbers
- Method of shipment
- Health and safety issues (level of protection, ambient screening results, etc.)
- Any unusual conditions or observations

The following entries will be made each day of the startup period. Entries will be in ink, and no erasures will be permitted. Each page will be initialed. Incorrect entries will be crossed out with a single strike mark and initialed. At the beginning of each entry, the

date, start time, and the names of site personnel and visitors present will be recorded. The following will be included in each entry:

- A summary of daily site activities and level of personal protection
- References to other project notebooks kept onsite (e.g., health and safety officer's notebook)
- Record of photographs taken with a description of each and its key points of interest. Videotape, slides, or photographs taken onsite or at sampling locations should be numbered to correspond to logbook entries. Include the photographer's name, date, time, site location, and site description.

10.4 Sample Shipment and Chain-of-Custody

Only EPA chain-of-custody forms, sample bottle tags, and custody seals will be used when EPA Contract Laboratory Program (CLP) services are used.

All necessary information assigned by the EPA (case numbers, SAS numbers, Site Spill I.D. numbers, and laboratory information) will be provided before sampling. Generation of EPA sample ID numbers (not to be confused with the sample location ID code, which is field generated) must be done according to EPA protocol.

Presentation of custody issues and procedures is provided in the QAPP in Chapter 7, "Sample Custody Procedures."

Sample documentation, packing and shipping instructions are provided in Appendix D in the QAPP.

MKE1001345F.WP5

Section 11
Sample Handling, Packaging, and Shipment

Sample handling, packaging, and shipping procedures are described in Appendix D of the QAPP.

MKE10013461.WP5

Section 12

Decontamination Procedures

This section provides the general guidelines for the decontamination of personnel, sampling and monitoring equipment, and sample bottles.

The following will be onsite:

- Distilled water
- 2.5 percent (by weight) TSP and water solution for decontamination
- 10 percent (by volume) methanol and water solution
- Large plastic pails or tubs for TSP and water; scrub brushes; squirt bottles for TSP, methanol, and water; plastic bags and sheeting (visqueen)
- Department of Transportation (DOT)-approved 55-gallon drum for disposal of waste

12.1 Personnel Decontamination

The following personnel decontamination procedures will be performed after tasks are completed that may have contaminated the worker and when the worker leaves the contaminated area:

1. Remove outer gloves and discard.
2. Remove respirator (if worn).
3. Remove disposable coveralls (e.g., Tyveks®) and discard.
4. Remove latex boot covers (if worn) and discard
5. Remove inner gloves (if worn) and discard.
6. Sanitize respirator (if worn).

12.2 Sampling and Monitoring Equipment Decontamination

Where process solids or process water have been in contact with field equipment used during sampling (i.e., sample spoons, sampling lines, pH probe, and specific conductance probe), the equipment will be decontaminated after sampling with a TSP and distilled water solution, followed by a 10 percent methanol and distilled water solution, followed by a distilled water rinse.

Section 13

Data Analysis and Evaluation

Data will be used to document compliance with effluent limits and requirements before effluent is discharged. Influent water quality will also be monitored to document treatment system efficiency. Basic process parameter data will be used to document the performance of the treatment system and individual processes. Data validation is discussed in the QAPP.

The data from daily samples will be compiled and used to assess the implementation, operation, and maintenance of the groundwater treatment system and to adjust and improve system operation as results become available. The data will be used to identify and implement any corrective action required to maintain reliable operation.

13.1 Evaluation of Results

Phase 1 Results

At a minimum, Phase 1 test results for BTEX, ammonia and iron are expected to be complete and available after completion of startup, and during the first few days of normal operation. Upon receipt, Phase 1 results will be used to assess process effluent quality. All results will be compiled and tabulated. The results for organic, inorganic, and other parameters will be compared to WDNR computed effluent limits to determine if allowable levels have been exceeded. The initial whole effluent toxicity bioassays will be used to determine if the process effluent is acutely toxic as defined in NR 106. A chronic toxicity test battery shall be determined positive if the IC_{25} associated with the effluent is less than 3.7 percent for any test species. Should testing indicate that the effluent is toxic, additional testing will be performed. For additional test requirements see Section 13.2.

Phase 2 and 3 Results

The results of the daily sampling and analysis program will be compiled. The analytical results will be averaged (where applicable) and the data evaluated to examine temporal trends. Process removal efficiencies and performances will be evaluated. The analytical results from process effluent sampling will be compared to WDNR computed effluent limits. Data analyses will include:

- Tables of the concentration for each parameter analyzed for the individual sample points
- If required, plots of concentration versus time to determine direction of trends

- Comparison of effluent concentrations to allowable effluent levels

The entire monitoring program will be reassessed weekly. Necessary adjustments to the program may include:

- Sampling frequencies—Is daily sampling adequate or excessive?

- Sampling points—Is the sampling network adequate? Do any sample points need to be replaced? Should additional sample points be used? Can some of the sample points be deleted from the sampling program?

- Sampling program—Do the available analytical data indicate that the process is operating as required? Should the monitoring program continue?

13.2 Additional Testing If Effluent Is Acutely Toxic

If the effluent is acutely or chronically toxic, samples for two second toxicity test batteries will be taken. The U.S. EPA and WDNR shall be notified and provided with test results within 3 business days following receipt of the test data (facsimile notification is acceptable). The additional test batteries shall be completed within 21 calendar days following the completion of the regularly-scheduled test that yielded the positive result.

Within 30 days of notification of the second test results, a plan will be submitted to the U.S. EPA and the WDNR for approval to perform one of the following:

- Conduct a toxicity identification evaluation and complete all necessary steps to reduce acute whole effluent toxicity and establish process operation limitations

- Take appropriate actions as necessary to reduce potential whole effluent toxicity as determined in consultation with the U.S. EPA and the WDNR

- Continue discharge

MKE1001345D.WP5

Section 14
Preventive Maintenance Procedure and Schedule

Sample team members will refer to the sampling procedure SOP and/or the manufacturers' instrument manuals for the appropriate preventive maintenance procedures for the sampling equipment used at the site.

GLO100130A7.WP5

Section 15
Waste Disposal

Wastes generated during sampling will consist of purge water, excess solids, wastes from decontamination, and protective clothing.

Once the groundwater treatment system is in operation, all water will be treated in the onsite groundwater treatment system.

Solids will be disposed of offsite following sampling to meet landfill disposal acceptance criteria. Protective clothing will be disposed of by regular municipal waste collection.

GLO100130A9.WP5

Section 16
Corrective Action

Record any deviations from routine procedures and corrective measures in the startup field notebook, and report them to the WDNR. The corrective action must be suited to the situation and may include:

- Repetition of measurement to check the error
- Checking batteries
- Recalibration of the instrument
- Replacement of the instrument

GLO100130B4.WP5

Section 17
Reporting

The final report will consist of a Technical Memorandum (TM) to the U.S. EPA and the WDNR. The major components of the report will be:

- Date and time of the sampling events
- Personnel involved in the sampling events and their respective responsibilities
- Locations sampled during the event
- Summary of the procedures used during the sampling event, including any deviations from standard procedures
- Pertinent observations taken during the startup sampling event
- Summary of the analytical results received from the laboratory and the validated results
- Temporal trends of the contaminant concentrations
- A summary of the average contaminant concentrations (for each sample location)
- Appendix addressing the analytical data and the QA/QA evaluations of the laboratory data

Data attachments to the final report will include:

- Data validation report
- Chain-of-custody forms
- Data table of compiled values (water quality, etc.) for all monitoring locations
- Sampling parameter sheets
- Process mass balance for contaminants of concern values collected during the sampling event

The final report will be delivered within 30 days after receipt of all analytical data and QA reviews for the startup sampling.

GLO100130B5.WP5

Section 18
Post Startup Activities

Following the startup sequence and during the operation of the processes, the post startup activities will occur. These activities are designed to identify problem areas and to resolve them, to identify and document operating procedures for each equipment item, and to perform final tests on automatic control equipment and control loops. Post startup activities include the following tasks:

1. The first step is to identify warranty items. These are items that have been identified during the startup procedure as requiring additional work by the subcontractor. A list of these items (startup punchlist) must be prepared and must include a detailed description of what the problem is, where it is located, and who to contact for additional information regarding the problem.

2. Finalize the startup punchlist. All the items on the startup punchlist need to be corrected during the post-startup procedures.

3. Perform the functional acceptance test on the process control center. This test verifies proper operation of the control loops and automatic operation of the equipment items.

MKE100130B6.WP5

References

1. Groundwater Treatment Remedial Design Subcontract. CH2M HILL. October 1992.

2. Remedial Investigation Report, Onalaska Municipal Landfill. CH2M HILL. December 1989.

3. Onalaska Municipal Landfill, Groundwater Extraction and Treatment Predesign Report. CH2M HILL. October 1991.

4. Draft O&M Manual. CH2M HILL. March 1992.

5. Groundwater Treatment Facility Operation Subcontract. CH2M HILL. November 1993.

6. Activated Carbon Treatment Subcontract. CH2M HILL. October 1993.

MKE100130B7.WP5

APPENDIX B
SAS REQUEST FORMS

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for benzene, toluene, ethylbenzene, xylene (BTEX) compounds. A 14-day turnaround time is required for analysis.

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

   50 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

EPA Method 602, Purgeable Aromatics. Include Xylene as a target compound.

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

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

Check sample pH with wide range pH paper. If pH> 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 14 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit (μg/L)	Precision Desired (+/- % or conc.)
Benzene	1 μg/L	+/- 20%
Toluene	10 μg/L	+/- 20%
Ethylbenzene	1 μg/L	+/- 20%
Xylene	10 μg/L	+/- 20%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	< individual detection limits
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 20%
Matrix Spike	At least 1 per group of 10 or fewer samples	70–130% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for benzene, toluene, ethylbenzene, xylene (BTEX) compounds. A 48-hour turnaround time is required for analysis.

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

   6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 48 hours of receipt of the samples.

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

EPA Method 602, Purgeable Aromatics. Include Xylene as a target compound.

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

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

Check sample pH with wide range pH paper. If pH > 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 48 hours from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 48 hours of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit (μg/L)	Precision Desired (+/- % or conc.)
Benzene	1 μg/L	+/- 20%
Toluene	10 μg/L	+/- 20%
Ethylbenzene	1 μg/L	+/- 20%
Xylene	10 μg/L	+/- 20%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	< individual detection limits
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 20%
Matrix Spike	At least 1 per group of 10 or fewer samples	70–130% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for iron. Samples will be filtered and unfiltered. A 48-hour turnaround time is required for analysis.

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

   12 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 48 hours of receipt of the samples.

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

Follow methods per SOW/ILM03.0 for Metals.

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

Samples will be preserved with HNO_3 to pH < 2 and stored at 4°C until analysis and validation of results.

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 48 hours from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 48 hours of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Iron	0.10 mg/L	+/- 20%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for ammonia. Results will be reported as mg/L N. A 48-hour turnaround time required for analysis.

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

9 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 48 hours of receipt of the samples.

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

EPA Method 350.1, Ammonia (as N). Colorimetric, Automated Phenate procedure.

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

Samples will be stored at 4°C until analysis and validation of results. Samples aliquots will be preserved in the field with H_2SO_4. The working concentration range of Method 350.1 Auto Analyzer should be 0.01 to 2 mg/L N (NH_3) or lesser concentration.

Check sample pH with wide range pH paper. If pH > 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 48 hours from sample collection. All solutions should be made with ammonia-free water.

The calibration curve must include at least five standards, one of which shall be a zero concentration standard. All standards, blanks, dilution water, and diluted samples shall be acidified with 1 mL/L H_2SO_4.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number. Results are to be in mg/L - N.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 48 hours of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Ammonia	0.01 mg/L	Duplicate results must agree within 10% for concentrations ≥ 1/mg/L or to within 0.1 mg/L for concentrations < 1 mg/L

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 0.1 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 0.1 mg/L)
Matrix Spike	At least 1 per group of 10 or fewer samples	85–115% recovery
Calibration Verification Standard	At least 1 per group of 10 or fewer samples	90–110% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for select TAL metals. A 7-day turnaround time is required for analysis. The select TAL metals include:

Aluminum, total
Arsenic, total
Cadmium, total
Chromium, total
Copper, total
Lead, total
Nickel, total
Zinc, total

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

6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

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

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per SOW/ILM03.0 for Metals.

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

Samples will be preserved with HNO_3 to pH < 2 and stored at 4°C until analysis and validation of results.

Dilute and rerun samples with concentrations higher than the highest standard.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Contract Required Detection Limit (µg/L)	Precision Desired (+/- % or conc.)
Aluminum, total	200	As required by the Statement of Works.
Arsenic, total	10	
Cadmium, total	5	
Chromium, total	10	
Copper, total	25	
Lead, total	3	
Nickel, total	40	
Zinc, total	20	

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for a target list of pesticides. EPA Method 508, Revision 3 is required to achieve low detection limits. A 7-day turnaround time is required for analysis. The target list of pesticides include:

Aldrin
Gamma-BHC
4,4'-DDD

Either perform a method detection limit (MDL) study or show a current MDL study for the pesticide analytes according to Federal Register V49 No. 209, October 26, 1984, (Part 136, Appendix 136).

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

6 process influent/effluent water samples. All samples are low concentration water samples.

A method detection limit study consisting of a minimum of 7 replicate analyses and 1 verification spike must be demonstrated (Attachment 3).

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

Superfund Remedial

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

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 508, Revision 3.

Analytical protocol to perform a method detection limit study are taken from Federal Register V49 No. 209, October 26, 1984 (Part 136, Appendix B). The target MDLs are listed in Attachment 1. Copies of all methods, method modifications and specific options are shown in Attachment 2 and 3.

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

See Attachments 1, 2, and 3.

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

All procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
See Attachment 1		+/- 20%

II. Quality Control Requirements

All QA/QC requirements shall be performed and reported as specified in CLP SOW for Organics Analysis (03/90) for Pesticides/PCB Analytes, for Surrogates, Matrix Spike/Matrix Spike Duplicates, Laboratory Blanks, GC performance. The procedures, frequencies and acceptance criteria used shall be the same as specified in the SOW, except that surrogate acceptance criteria shall be as specified in Attachment 3, and method blank requirements shall be as specified in Attachment 3.

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

Attachment 1

Target Analyte List and Method Detection Limit

Compound	CAS Number	Detection Limit (µg/L)
Aldrin	309-00-2	0.05
Gamma-BHC	58-89-9	0.05
4,4'-DDD	72-54-8	0.001

MKE10013447.WP5

Attachment 2
Special Technical Instructions for the MDL Study

1. Method Detection Limit (MDL) Study

The MDL Study shall be performed prior to receipt and analysis of field samples. The MDL study shall consist of a statistically determined MDL using the procedures described in the Federal Register (V.49, No. 209, Appendix B to Part 136, 10-26) and verified through a spike at the computed MDL.

A. Computed MDL

Using the Federal Register procedure, prepare and analyze a minimum of 7 replicates containing all target compounds and surrogate standards (details of the analysis shall follow EPA Method 508 and Attachment 3). Use the SAS target detection limits for the spike levels for the 7 replicates (Attachment 1). At a minimum, the laboratory shall perform the MDL study at the method detection limits described in Attachment 2. The Superfund RI/FS project needs detection limits at the levels of the State of New Jersey Water Quality Criteria. If the laboratory can detect lower quantities than the method detection limits then those limits should be provided.

Compute the statistically determined MDL.

B. Verified MDL

Following 1A above, prepare and analyze reagent water spiked with all target compounds (at the computed MDL) and surrogate standards (details of the analysis shall follow EPA Method 508 and Attachment 3). All qualitative criteria used for identification of the target compounds (i.e., retention times and second column confirmation) must be met for all compounds (see EPA Method 508). If any compound does not meet al.l qualitative criteria at the computed MDL, reanalyze a reagent water blank with only the compound(s) which failed the criteria. Contact the SMO coordinator if this reanalysis does not still meet criteria.

C. Target Compounds and Surrogate Standards

i. Target Compounds: See Attachment 1

ii. Surrogate Standards: See Attachment 3

D. Data Deliverables

The test procedure used will be clearly identified. Bench records tabulating the sample preparation, order of analysis, instrument calibration, calibration verification, lab blanks, samples, lab control standards, etc., with resulting chromatograms will be provided along with calculation worksheets. All records will be legible and sufficient to recalculate all sample concentrations and QA audit results.

MKE10013447.WP5

Attachment 3
Modifications and Notes to EPA Method 508
Determination of Chlorinated Pesticides in Water by Gas Chromatography With An Electron Capture Detector

Note: Unless modification or notes to the Method are described the analysis shall be performed as written.

Section	Comments
1.1	The target compound list for analysis will be the Target Analyte List for pesticides
1.3	Demonstration of the analysts ability to perform the method is not required
1.6	Second column confirmation is required for all analytes quantificated on the first column
3.1	Not Applicable
3.4	Not Applicable
3.6	Not Applicable
3.7	Not Applicable
3.8	As described in Attachment 2
4.4	The analytical columns must be able to resolve all target analytes
7.6	Not Applicable
7.11	Not Applicable
7.12	Use 4,4'-dichlorobiphenyl
7.13	Not Applicable, requirement may be deleted from method
8.1	Not Applicable
8.2	Not Applicable, except samples must be refrigerated at 4°C from the time of sample receipt until extraction
9.1	Use the external calibration technique
9.3.1	Use five calibration standards. The lowest standards should represent analyte concentrations near, but above, their respective detection limits shown in Attachment 1
9.3.3	A single mid-calibration range check standard shall be analyzed daily prior to the analysis of samples and at the end of analysis or 1 per 10 samples, whichever is more frequent
9.3.4	Not Applicable
10.1	Internal standards will not be used
10.3	Not Applicable

10.4 Modifications to extraction techniques, GC detectors, surrogate compounds are not allowed

10.6 Not Applicable

10.7.1 Not Applicable

10.8.1 Matrix spike/matrix spike duplicates shall be analyzed 1 per 20 samples, or less

The added concentration shall be 10 times higher than the MDL

10.9 Not Applicable

The analysis of a detection limit verification spike is described in the SAS Request (Section 8)

11.3.1 Use a K-D concentrator

11.3.5 Analyze by Gas Chromatography/Electron Capture Detector (GC/ECD)

11.4.1 Use GC/ECD

11.4.3 Not Applicable

11.5.3 All components that fall within the retention time window on the quantification column must be reanalyzed on a second, dissimilar column for confirmation.

12.2 Not Applicable

13Not Applicable

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[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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1. General description of analytical service requested:

Analysis of system process influent and effluent water samples for EPA Priority Pollutant List Semivolatile Organic Compounds (SVOCs). A 7-day turnaround time is required for analysis. The list of analytes are shown in Table 1.

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

3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per EPA Method 625 for Semivolatile Organic Analysis.

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

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

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

Report pentachlorophenol down to 10 μg/L.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

See Table 1.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by EPA Method 625		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

Table 1
Summary of Parameters, Detection Limits, and Precision
(Page 1 of 2)

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
1,2,4-Trichlorobenzene		As required by EPA Method 625
1,2-Dichlorobenzene		
1,2-Diphenylhydrazine		
1,3-Dichlorobenzene		
1,4-Dichlorobenzene		
2,4,6-Trichlorophenol		
2,4-Dichlorophenol		
2,4-Dimethylphenol		
2,4-Dinitrophenol		
2,4-Dinitrotoluene		
2,6-Dinitrotoluene		
2-Chloronaphthalene		
2-Chlorophenol		
2-Nitrophenol		
3,3'-Dichlorobenzidine		
4,6-Dinitro-o-cresol		
4-Bromophenyl phenyl ether		
4-Chlorophenyl phenyl ether		
4-Nitrophenol		
Acenaphthene		
Acenaphthylene		
Anthracene		
Benzidine		
Benzo(a)anthracene		
Benzo(a)pyrene		

Table 1
Summary of Parameters, Detection Limits, and Precision
(Page 2 of 2)

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Benzo(b)fluoranthene		As required by EPA Method 625
Benzo(ghi)perylene		
Benzo(k)fluoranthene		
Butyl benzyl phthalate		
Chrysene		
Di-n-butyl phthalate		
Di-n-octyl phthalate		
Dibenzo(a,h)anthracene		
Diethyl phthalate		
Dimethyl phthalate		
Fluoranthene		
Fluorene		
Hexachlorobenzene		
Hexachlorobutadiene		
Hexachlorocyclopentadiene		
Hexachloroethane		
Indeno(1,2,3-c,d)pyrene		
Isophorone		
N-Nitrosodi-n-propylamine		
N-Nitrosodimethylamine		
N-Nitrosodiphenylamine		
Naphthalene		
Nitrobenzene		
Pentachlorophenol		
Phenanthrene		
Phenol		
Pyrene		
bis(2-Chloroethoxy)methanee		
bis(2-Chloroethyl) ether		
bis(2-Chloroisopropyl)ether		
bis(2-Ethylhexyl)phthalate		
p-Chloro-m-cresol		

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SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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1. General description of analytical service requested:

Analysis of system process influent and effluent water samples for EPA Priority Pollutant List Volatile Organic Compounds (PPL-VOCs). A 7-day turnaround time is required for analysis. See Table 1 for list of analytes.

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

3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per EPA Method 624 for Volatile Organic Analysis.

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

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

Check sample pH with wide range pH paper. If pH > 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

See Table 1.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by EPA Method 624		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

Table 1 Summary of Parameters, Detection Limits, and Precision		
Parameter	**Detection Limit**	**Precision Desired (+/- % or conc.)**
1,1,1-Trichloroethane 1,1,2,2-Tetrachloroethane 1,1,2-Trichloroethane 1,1-Dichloroethane 1,1-Dichloroethylene 1,2-Dichloroethane 1,2-Dichloropropane 1,2-Trans-dichloroethylene 2-Chloroethylvinyl ether Acrolein Acrylonitrile Benzene Bromoform Carbon tetrachloride Chlorobenzene Chlorodibromomethane Chloroethane Chloroform Dichlorobromomethane Dichlorodifluoromethane Ethylbenzene Methyl bromide Methyl chloride Methylene chloride Tetrachloroethylene Toluene Trichloroethylene Trichlorofluoromethane Vinyl chloride bis(Chloromethyl)ether cis-1,3-Dichloropropylene trans-1,3-Dichloropropylene		As required by EPA Method 624

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CLP Sample Management Office
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SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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1. General description of analytical service requested:

Analysis of system process influent and effluent water samples for EPA Priority Pollutant List Pesticides and PCB Aroclors. A 7-day turnaround time is required for analysis.

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

3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per EPA Method 608 for Pesticides and PCBs.

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

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

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

See Table 1.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by EPA MEthod 608		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

Table 1 Summary of Parameters, Detection Limits, and Precision		
Parameter	**Detection Limit**	**Precision Desired (+/- % or conc.)**
4,4'-DDD 4,4'-DDE 4,4'-DDT Aldrin Alpha-BHC Beta-BHC Chlordane Delta-BHC Dieldrin Endosulfan I Endosulfan II Endosulfan sulfate Endrin Endrin aldehyde Gamma-BHC Heptachlor Heptachlor epoxide PCB-1016 PCB-1221 PCB-1232 PCB-1242 PCB-1248 PCB-1254 PCB-1260 Toxaphene		As required by EPA Method 608

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1. General description of analytical service requested:

Analysis of system process influent and effluent water samples for a target list of semivolatile organic compounds (SVOCs). A 7-day turnaround time is required for analysis. The target SVOCs include:

Pentachlorophenol
Phenol
Di-n-butyl Phthalate
1,4-Dichlorobenzene
2,4-Dinitrotoluene
Naphthalene
Pyrene

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

6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per SOW/OLM01.9 for Semivolatile Organic Analysis.

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

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

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

Report pentachlorophenol down to 10 μg/L.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Report only the target list of SVOCs as specified in Section 1.

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Pentachlorophenol Phenol Di-n-butyl Phthalate 1,4-Dichlorobenzene 2,4-Dinitrotoluene Naphthalene Pyrene	As required by the Statement of Works, except pentachlorophenol which should be reported at 10 μg/L.	As required by the Statement of Works.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits ($\pm$ % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

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C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for a target list of volatile organic compounds (VOCs). A 7-day turnaround time is required for analysis. The target list of VOCs include:

Benzene
Chloroform
1,1-Dichloroethylene
1,2-Dichloroethylene
Ethylbenzene
Toluene
1,1,1-Trichloroethane
Trichloroethylene
Vinyl Chloride

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

6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

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

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per SOW/OLM01.9 for Volatile Organic Analytes.

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

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

Check sample pH with wide range pH paper. If pH > 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Report only the target list of analytes as specified in Section 1.

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
As required by the Statement of Works.		

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. **Regional Representative:** Brian Freeman

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** December 27, 1993

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 system process influent and effluent water samples for nitrate/nitrite. Results will be reported as mg/L N. A 7-day turnaround time is required for analysis.

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

   3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 353.2, Nitrate/Nitrite. This is a colorimetric, automated cadmium reduction procedure.

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

Samples should be stored at 4°C until analysis and validation of results. Samples should be preserved in the field with H_2SO_4 to pH < 2. Neutralize samples to pH 5–9 prior to analysis.

After checking pH, it is recommended that the laboratory check for residual chlorine (or oxidizing agents) and sulfide using test strips such as starch iodide and lead acetate papers. The laboratory must remove these interferences prior to analysis.

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.

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 the order of calibration standards, lab control standards, lab blanks, samples, spikes, duplicates, etc., with resulting absorbances or concentration readouts should be provided. Records of analysis and calculations should be sufficient to recalculate all concentrations.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Nitrate/Nitrite	0.1 mg/L	Duplicate results must agree within 20% for concentrations ≥ 1 mg/L or to within 0.1 mg/L for concentrations < 1 mg/L

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	< 0.1 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (20% or 0.10 mg/L)
Matrix Spike	At least 1 per group of 10 or fewer samples	80–120% recovery
Continuing Calibration Verification Standard	At least 1 per group of 10 or fewer samples	90–110% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013447.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for total dissolved solids (TDS). Results will be reported as mg/L dissolved solids. A 7-day turnaround time is required for analysis.

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

   3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 160.1, Filterable Residue. This is a gravimetric procedure.

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

Samples will be kept at 4°C until analysis and validation of results are complete. Holding time is 7 days from date of sample collection.

1. Use standard aliquots of 100 mL; 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 is less than 4.0, raise the pH of the aliquot using NaOH titrant to a pH between 4 and 8 and subtract the weight of sodium added from the weight of the residue.

3. Residue will be weighed to a constant weight pursuant to Section 7.6 of Method 160.1, which is the weight to be used for calculations. Constant weight is defined as (a) less than 0.5 mg or less than 4 percent 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.

All procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation, weighing, and analysis logbooks shall be submitted. Submit records of all weighings of samples, blanks, duplicates, and reference samples, including initial, final, and intermediate weighings. Dates and times of (a) determination of tare weights, (b) sample filtration, and (c) determination of residue weights and constant residue weights shall be included in the records. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TDS	20 mg/L	Duplicate results must agree within 10% for concentrations ≥ 200 mg/L or to within 2 mg/L for concentrations < 200 mg/L

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank (100 mL of reagent water)	At least 1 per group of 10 or fewer samples	± 20 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg/L)

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for EPA Priority Pollutant List Metals: Antimony, Arsenic, Beryllium, Cadmium, Chromium, Copper, Lead, Mercury, Nickel, Selenium, Silver, Thallium, and Zinc plus Cyanide. A 7-day turnaround time is required for analysis.

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

3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per SOW/ILM03.0 for Metals plus Cyanide.

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

Samples collected for metals analyses will be preserved with HNO_3 to pH < 2 and stored at 4°C until analysis and validation of results.

Samples collected for cyanide analysis should be analyzed as rapidly as possible after collection. Samples should be preserved with 2 mL of 10 N sodium hydroxide per liter of sample (pH $\geq$ 12) at the time of collection. Samples will be stored at 4°C until analysis and validation of results.

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 7 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Contract Required Detection Limit (µg/L)	Precision Desired (+/- % or conc.)
Antimony	60	As required by the Statement of Works.
Arsenic	10	
Beryllium	5	
Cadmium	5	
Chromium	10	
Copper	25	
Lead	3	
Mercury	0.2	
Nickel	40	
Selenium	5	
Silver	10	
Thallium	10	
Zinc	20	
Cyanide	1,000	

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for hardness. Results will be reported as mg/L $CaCo_3$. A 7-day turnaround time is required for analysis.

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

3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 130.2, Hardness, Total as $CaCO_3$. This is an EDTA titrimetric procedure.

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

Samples should be preserved with HNO_3 to a pH of < 2 and stored at 4°C until the time of analysis. Pretreatment of the samples includes neutralizing 50.0 mL of the sample with 1N ammonium hydroxide (note volume of NH4OH used). Any remaining sample should be stored in the same manner until the validation and the acceptance of the sample result.

Use inhibitors as necessary.

In order to avoid large titration volumes, sample volumes should be adjusted as to not contain more than 25 mg $CaCO_3$.

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

Bench records that clearly and legibly show the order of; EDTA titrant standardization, lab blanks, samples, lab control standards, and lab duplicates. In order to be able to reproduce the calculated hardness results, these bench records should also show sample volumes, titrant volumes, and the normality of the titrant.

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

Follow protocol as per stated methodology.

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Hardness	3 mg/L	Duplicate results must agree within 10% for concentrations ≥ 10 mg/L or to within 2 mg/L for concentrations < 10 mg/L

II. Quality Control Requirements

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

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
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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 Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for BOD_5. Results will be reported as mg/L oxygen. A 7-day turnaround time is required.

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

   9 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 405.1, BOD_5. BOD Method 507, Biological Oxygen Demand (BOD), "Standard Methods for the Examination of Water and Wastewater." (5-day, 20°C).

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

Set up three or more sample dilutions so that two or more sample dilutions overlap to result in a residual D.O. > = 1 mg/L and a D.O. depletion > = 2 mg/L. Measure the seed BOD using two or more dilutions. BOD results for two dilutions should agree within +/- 15 percent. Analyze unseeded dilution water blanks, and glucose-glutamic acid checks, 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 beginning of sample collection. Dilution water will be seeded so that the calculated D.O. uptake from BOD of seed will be between 0.6 and 1.0 mg/L. Do not use seeded blanks to estimate seed corrections. All procedures defined in the method must be followed precisely.

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. Report results of duplicates, unseeded dilution water blank, BOD of seed, calculated D.O. uptake of seed in seeded dilution water, and glucose-glutamic acid check. Report any reference sample, initial calibration verification sample used and identify as to source, lot number, and sample number. Corresponding "true" or target values and associated 95 percent confidence limits for analysis will be provided for all reference samples used.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
BOD_5	2.0 mg/L	Differences in duplicate series of sample results shall not exceed 2 mg/L for concentrations less than 20 mg/L.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Glucose-glutamic acid checks	1 pair per set of samples	160–240 mg/L
Duplicate (Full dilution series)	1 per group of 10 or fewer samples	+/-(10% or 2 mg/L)
Unseeded Dilution Water Blanks	1 pair per set of samples, including 1 pair for each lot of dilution water	≤ 0.2 mg/L
DO Uptake of seed in seeded dilution water (calculated)	Calculated for each lot of seeded dilution water	0.6–1.0 mg/L

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for chloride. Results will be reported as mg/L Cl. A 7-day turnaround time is required for analysis.

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

   6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA-II), 1983 ed.

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

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

Prepare all standard reagents, blanks, etc. with ASTM Type II reagent water or equivalent. Calibration standards shall be prepared daily from stock solutions. Use a working concentration range or standard curve between 0 to 20 mg/L or less. The calibration curve shall contain at least five different levels of standards, including a zero concentration standard. Dilute and re-analyze any samples with concentrations that fall outside of the calibration range. Remove any large amounts of turbidity prior to sample analysis. (see section 7 of method 325.2).

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

All procedures used shall be clearly identified. Bench records tabulating order of calibration standards, verification and control standards, samples, matrix spikes, titrant blanks, etc., with resulting peak height, concentration, or absorbance read-outs shall be provided, as well as copies of worksheets used to calculate results. A photocopy of instrument readouts, e.g., stripcharts, printer tapes, etc., shall be included with all results. All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Chloride	1 mg/L	Differences in duplicate sample results are to be < = 0.5 mg/L for concentrations < 5 mg/L and < = 10% for concentrations > 5 mg/L. Report chloride concentration to the nearest 0.1 mg/L between 0 and 20 mg/L.

II. Quality Control Requirements

Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 0.5 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 0.5 mg/L)
Matrix Spike	At least 1 per group of 10 or fewer samples	85–115% recovery
Calibration Verification Standard	At least 1 per group of 10 or fewer samples	90–110% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for total suspended solids (TSS). Results will be reported as mg/L total suspended solids. A 7-day turnaround time is required for analysis.

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

9 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 160.2, Nonfilterable Residue. This is a gravimetric procedure.

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

EPA Method 160.2, 1983 ed., (Gravimetric, dried at 103°to 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 to 60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples are to be held at 4°C until analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

1. Sample aliquot volumes are selected on the basis of the following factors:

   a. During initial sample filtration, filtration rate should not drop rapidly, or require more than 5 minutes of filtration time (Increase the filter area or decrease the sample volume as needed for re-analysis),

   b. The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200 mL in volume, and

   c. Sample aliquots should not exceed 200 mL in volume.

2. Duplicate sample aliquots shall be filtered with two 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 used for calculation), or dried overnight (at least 12 hours drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4 percent weight loss from the previous weight, whichever is smaller.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation, weighing, and analysis logbooks shall be submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples, shall be provided. Dates and times of filtration of initial 100 mL volume, determination of tare weight, sample filtration, and determination of constant residue weights, shall be included.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TSS	3 mg/L	Duplicate results must agree to < 0.5 mg/L for residues ≤ 5 mg or less than 10% for residues > 5mg

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank (200 mL aliquots)	At least 1 per group of 10 or fewer samples	± 0.5 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 0.5 mg)

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for total phenols. A 7-day turnaround time is required for analysis.

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

   3 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 420.1, Total Recoverable Phenolics. This is a manual spectrophotometric 4-AAP with distillation procedure.

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

Biological degradation is inhibited by the addition of 1 g/L of copper sulfate to the sample and acidification to pH < 4 with phosphoric acid. Samples will be stored at 4°C until analysis and validation of results. The holding time is not to exceed 24 hours from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Phenols, Total	5 µg/L	+/- 20%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 5 µg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 20%
Matrix Spike	At least 1 per group of 10 or fewer samples	70–130% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

U.S. Environmental Protection Agency
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SAS Number [ ]

Special Analytical Services Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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1. General description of analytical service requested:

Analysis of system process influent and effluent water samples for hexavalent chromium. A 7-day turnaround time is required for analysis.

Chromium (Hexavalent)

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

6 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

Follow methods per EPA 218.4 for Hexavalent Chromium.

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

Samples will be stored at 4°C until analysis and validation of results. The holding time for Hexavalent Chromium is **24 hours** from sample collection.

Dilute and rerun samples with concentrations higher than the highest standard.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 7 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Contract Required Detection Limit (µg/L)	Precision Desired (+/- % or conc.)
Chromium (Hexavalent)	10	As required by the Statement of Works.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 the bulk density of system process filter cake (solid) samples. A 14-day turnaround time is required for analysis.

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

3 process filter cake (solid) samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

ASTM Method D5057, Bulk Density.

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

Per Method D5057.

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 records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Per ASTM Method D5057.

II. Quality Control Requirements

Per ASTM Method D5057.

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

Designation: D 5057 – 90

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Screening Apparent Specific Gravity and Bulk Density of Waste[1]

This standard is issued under the fixed designation D 5057; 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 apparent specific gravity and bulk density in waste. For the purpose of this test method, materials to be measured will be classified into three groups:

1.1.1 *Group A*—Free-flowing liquids; apparent specific gravity (ASG),

1.1.2 *Group B*—Granules, powders and water reactive liquids, solids or sludges; bulk density (BD), and

1.1.3 *Group C*—Bulk solids (such as gravel, paper or wood, etc.); apparent specific gravity (ASG).

1.2 This test method is designed and intended as a preliminary test to complement the more sophisticated quantitative analytical techniques that may be used to determine specific gravity. This test method offers to the user the option and the ability to screen waste for apparent specific gravity or bulk density when the more sophisticated techniques are not available and the total waste composition is unknown.

1.3 *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.* For a specific hazard statement, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1192 Specification for Equipment for Sampling Water and Steam[2]

D 1193 Specification for Reagent Water[2]

D 3370 Practices for Sampling Water[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

3. Terminology

3.1 *Description of Term Specific to This Standard:*

3.1.1 *screening*—a *preliminary* qualitative or semi-quantitative test, developed from classical qualitative and quantitative techniques, that is designed to efficiently give the user specific information about a waste that will aid in determining waste identification, process compatibility, and safety in handling.

4. Summary of Test Method

4.1 The specific gravity of a material is the ratio of the masses of equal volumes of a waste and reagent water. The apparent specific gravity of materials in Groups A and C is determined by comparing the mass of a sample to the mass of the same volume of reagent water. The bulk density of wastes in Group B is determined as a direct mass/volume ratio of the sample alone and should be used for determinations on water reactive materials. The weights are used in determining mass.

5. Significance and Use

5.1 This test method is intended for use by those in the waste management industries for the determination of apparent specific gravity and bulk density of waste.

5.2 The apparent specific gravity and bulk density determined by this test method can be used for the conversion of measured volumes to weights.

5.3 The apparent specific gravity and bulk density, when correlated with other properties, can be used to indicate the character of the waste.

6. Interferences

6.1 Excessive temperatures causing loss of sample components due to vaporization could result in erroneous readings.

6.2 Large, obvious void spaces interfere in this test method and will give inaccurate results because of the false volume measured.

7. Apparatus

7.1 *Weighing Bottle*—Specific gravity bottle or equivalent container is needed.

7.2 *Spatulas.*

7.3 *Top Loader Balance*, with a sensitivity of 0.01 g is required.

8. Reagents and Materials

8.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

9. Hazards

9.1 Precaution—Avoid inhalation of and skin and eye contact with all hazardous materials.

10. Sampling

10.1 Collect the sample in accordance with Specification

[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D34.02 on Physical and Chemical Characterization.

Current edition approved May 25, 1990. Published July 1990.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.03.

D 1192 and Practices D 3370 and D 4057.

NOTE 1—Extreme temperature variations between the sample and reagent water should be avoided.

11. Procedure

11.1 Make all weight measurements to nearest 0.01 g.

11.2 Weigh the empty container (weighing bottle or specific gravity bottle) with lid on, and record weight as W.

11.3 Fill the container with water and place lid on container. Wipe off excess water and weigh. Record weight of water-filled container as R.

11.4 For free-flowing liquids (Group A):

11.4.1 Fill the empty container (see 7.1) with sample.

11.4.2 Place the lid on the container, pushing out excess sample through the hole.

11.4.3 Wipe off excess sample.

11.4.4 Weigh the sample-filled container with lid on, and record weight as S.

11.5 For granules, powders, and water reactive materials (Group B):

11.5.1 Add as much of the sample to the weighed container (see 7.1) as possible without exerting pressure, filling the container with sample but not allowing large void spaces (see 6.2). The container may be tapped or lightly tamped.

11.5.2 Place the lid on the container and weigh the sample and bottle and record weight as S.

11.6 For bulk solids such as gravel, paper or wood (Group C):

11.6.1 Add as much of the sample to the weighed container (see 7.1) as possible without exerting pressure. Place the lid on the container and weigh and record weight as S.

11.6.2 Fill remaining space in the container with water and place the lid on the container, taking care that air bubbles are not trapped in the material or the container.

11.6.3 Weigh and record weight of sample and water, in container with the lid on. Record weight as Q.

NOTE 2—The quantity Q-S may be erroneously high due to the water absorbed by the solid sample.

12. Calculation

12.1 Calculate the apparent specific gravity (ASG) or bulk density from the following equations, matching the appropriate equation with the procedure of choice for each group.

12.1.1 For Group A materials:

$$\text{ASG} = \frac{(S-W)}{(R-W)} \tag{1}$$

where:

W = weight of empty container (with lid),
R = weight of water-filled container (with lid), and
S = weight of sample-filled container (with lid).

12.1.2 For Group B materials:

$$\text{Bulk Density (g/mL)} = (Y)\frac{(S-W)}{(R-W)} \tag{2}$$

where Y = 1 g/mL, the conversion of mass/volume at 4°C.

12.1.3 For Group C materials:

$$\text{ASG} = \frac{(S-W)}{(R-W)-(Q-S)} \tag{3}$$

where Q = weight of sample and water-filled container.

13. Report

13.1 Report at a minimum the following information:

13.1.1 Sample identification and group (A, B, or C),

13.1.2 Date of test,

13.1.3 Procedure applied, and

13.1.4 Test results.

14. Quality Assurance

14.1 Instrument performance standards, quality control check samples of appropriate matrices, and duplicates should be performed at an action level specified by the laboratory and at an appropriate frequency.

15. Precision and Bias

15.1 The precision and bias of the procedure in this test method is being determined.

16. Keywords

16.1 density; specific gravity; waste screening

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SAS Number [ ]

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[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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1. General description of analytical service requested:

Determination of percent solids in system process filter cake (solids). Report as percent solids.

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

3 filter cake (solid) sample.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

EPA Method 160.3, Total Residue. Gravimetric, dried at 103–105°C.

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

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

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
% Solids	Not Applicable	Duplicate results must agree within 10%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 10 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± 10%

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 a system process filter cake sample by Toxicity Characteristic Leaching Procedure (TCLP). Samples will be extracted according to Method 1311 of Federal Register June 29, 1990, for the TCLP contaminants (Attachment I). The extracts will be analyzed per current SOWs (SOW/OLM01.9 for organics and SOW/ILM03.0 for inorganics). TCLP metals, VOC, SVOC and pesticides are to be determined. A 14-day turnaround time is required for analysis.

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

1 process filter cake (solid) sample.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

Method 1311 of Federal Register—June 29, 1990, for extraction with the specifics as noted in Attachment II. CLP SOWs are to be followed for the analysis of the TCLP extracts (SOW/OLM01.9 for organics and SOW/ILM03.0 for inorganics).

Glass containers must be used for the organic analyses.

After TCLP extraction, the extracts must be kept at 4°C and preserved as follows; VOAs must be acidified to pH $<$ 2 with 1:1 HCL, metals must be acidified to a pH $<$ 2 with HNO_3.

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

See Attachment II.

Samples will be collected in 1-liter wide-mouth glass jars for metals, SVOAs, and pesticides. Two 4-oz wide-mouth glass jars will be collected for VOA analysis.

The results of all dilutions must be reported.

A TCLP blank must be carried through the extraction procedure as required by the method.

Follow the method of standard additions for the metals analyses.

Detection limits shall not exceed one-tenth of the concentration that would define the sample as a hazardous waste for each parameter. If the concentration of the analyte after correction for the matrix spike recovery is $\geq$ 10 percent of the regulatory level, the TCLP extract must be reextracted using a smaller aliquot and spiked at the regulatory level such that the native analyte is at approximately the regulatory level.

The hydrochloric acid and acetic acid used in Extraction Fluid No. 1 and Extraction Fluid No. 2 must be standardization prior to use.

1. The 1 N HCl can be and will be standardized to 1.0 N HCl $\pm$ 5 percent.

2. The pH of Extraction Fluid No. 1 will be 4.93 $\pm$ 0.05. No Standardization of acetic acid can be done—See Section 5.7.1 of the method.

3. The pH of Extraction Fluid No. 2 will be 2.88 $\pm$ 0.05. Standardization of acetic acid is not mandatory but will be done for informational purposes (Optional) and will be compared to theoretical value of 5.7 mL. glacial acetic acid diluted to 1 liter. Titration of acetic acid normality can not be used for contract compliance purposes if correct pH value is obtained (2.88).

A minimum of a four point calibration curve must be used for all analyses. If sample concentrations exceed the calibration range, sample must be diluted to fall within the calibration range.

Matrix spike and matrix spike duplicate (MS/MSD) recoveries must be determined. MS/MSD recoveries must be performed for each of the analytes.

The Case narrative must discuss any sample problems.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Precision is as required by the appropriate Statement of Works, and detections limits are to be no greater than 1/10 the concentration that would define the sample as a hazardous waste for each parameter.

II. Quality Control Requirements

Audits Required	Frequency of Audits	Limits* (±% or conc)
TCLP Extraction		
Preparation Blank for Extract Fluid No. 1(see section 8.1 of Method 1311)	Each set of solid samples	< 5% of Regulatory levels of Table1. Discuss in case narrative if larger than CRDLs of SOW.
Prep. Blk. for Extract Fluid No. 2, if necessary	Same	Same
Analysis of TCLP Extracts		
Preparation blank for TCLP Extract Determinations	Per appropriate SOW and set up with each TCLP extract batch	CRDL of appropriate SOW for Attachment I constituents.
MS/MSD	See Attachment II	Advisory—used to correct TCLP values recovery.
	1 for each set of 8 sample extracts.	RPD ≤ 20% (MS/MSD)
All other QC audits per OLM01, ILM01 and method 8150	Per OLM01, ILM01 and method 8150	Per OLM01, ILM01, and method 8150

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

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Attachment I

List of Constituents	Regulatory Level (mg/L)
Volatile Organics	
Benzene	0.5
Carbon Tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
Methyl Ethyl Ketone	200.0
Tetrachloroethylene	0.7
Trichloroethylene	0.5
Vinyl Chloride	0.2
Semivolatiles Acid Fraction	
o-Cresol	200.0
m-Cresol	200.0
p-Cresol	200.0
Cresol (total)	200.0
Pentachlorophenol	100.0
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
Semivolatiles Acid Fraction	
1,4-Dichlorobenzene	7.5
2,4-Dichlorobenzene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Nitrobenzene	2.0
Pyridine	5.0
Pesticides	
Chlordane	0.03
Endrin	0.02
Heptachlor (and its epoxide)	0.008
Lindane (gamma BHC)	0.4
Methoxychlor	10.0
Toxaphene	0.5
Metals	
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

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Attachment

TCLP Extraction will be done by Federal Register of June 29, 1990. Samples will be dry, but depending on weather conditions could be wet or moist soils could be submitted; therefore, the filtration procedure (Section 7.1.1.7) may produce interstitial water. Also any water collecting on top of sediment or soil is not to be discarded, but mixed with sample prior to filtration or percent solid determination (Section 7.1.1). Particle size reduction is not expected to be necessary for these soils.

Analysis of TCLP extracts will be done to determine compliance with Regulatory Levels using minimum sample aliquot volumes necessary for this purpose.

Sample aliquot sizes are to be minimized to alleviate interferences from acetic acid/acetate buffer, to provide CRQLs that are 10–20 percent of Regulatory Levels, and to expand the working concentration range of the test procedures.

All constituents of Attachment I are required to be determined and reported for TCLP extracts. Remaining TALs of OLM01, ILM01, and SW-846 Method 8150 are not required. A matrix spike/matrix spike duplicate (MS/MSD) for all constituents in Attachment I will be prepared and determined using one of the TCLP soil extracts.

MS/MSD results are advisory and used for calculation purposes.
TCLP Extraction of June 29, 1990, requires correction of constituent values for matrix spike recoveries. See Section 8.2 of Method 1311 of Federal Register June 29, 1990.

The average MS/MSD recovery developed for 1 of the soil extracts will be applied to all of the soil extracts. It is not expected that the samples will provide TCLP values that will exceed Regulatory Levels; however, there is a finite chance that this will occur.

If any on TCLP analyte in an extract exceeds Regulatory Levels, the extracts reanalysis is unnecessary using a Regulatory Matrix Spike concentration (see Section 8.2 of Method 1311).

MKE10013447.WP5

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SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for total dissolved solids (TDS). Results will be reported as mg/L dissolved solids. A 14-day turnaround time is required for analysis.

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

28 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

EPA Method 160.1, Filterable Residue. This is a gravimetric procedure.

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

Samples will be kept at 4°C until analysis and validation of results are complete. Holding time is 7 days from date of sample collection.

1. Use standard aliquots of 100 mL; 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 is less than 4.0, raise the pH of the aliquot using NaOH titrant to a pH between 4 and 8 and subtract the weight of sodium added from the weight of the residue.

3. Residue will be weighed to a constant weight pursuant to Section 7.6 of Method 160.1, which is the weight to be used for calculations. Constant weight is defined as (a) less than 0.5 mg or less than 4 percent 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.

All procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation, weighing, and analysis logbooks shall be submitted. Submit records of all weighings of samples, blanks, duplicates, and reference samples, including initial, final, and intermediate weighings. Dates and times of (a) determination of tare weights, (b) sample filtration, and (c) determination of residue weights and constant residue weights shall be included in the records. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TDS	20 mg/L	Duplicate results must agree within 10% for concentrations ≥ 200 mg/L or to within 2 mg/L for concentrations < 200 mg/L

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank (100 mL of reagent water)	At least 1 per group of 10 or fewer samples	± 20 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg/L)

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for BOD_5. Results will be reported as mg/L oxygen. A 14-day turnaround time is required.

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

36 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 405.1, BOD_5. BOD Method 507, Biological Oxygen Demand (BOD), "Standard Methods for the Examination of Water and Wastewater." (5 day, 20°C).

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

Set up three or more sample dilutions so that two or more sample dilutions overlap to result in a residual D.O. > = 1 mg/L and a D.O. depletion > = 2 mg/L. Measure the seed BOD using two or more dilutions. BOD results for two dilutions should agree within +/- 15 percent. Analyze unseeded dilution water blanks, and glucose-glutamic acid checks, 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 beginning of sample collection. Dilution water will be seeded so that the calculated D.O. uptake from BOD of seed will be between 0.6 and 1.0 mg/L. Do not use seeded blanks to estimate seed corrections. All procedures defined in the method must be followed precisely.

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. Report results of duplicates, unseeded dilution water blank, BOD of seed, calculated D.O. uptake of seed in seeded dilution water, and glucose-glutamic acid check. Report any reference sample, initial calibration verification sample used and identify as to source, lot number, and sample number. Corresponding "true" or target values and associated 95 percent confidence limits for analysis will be provided for all reference samples used.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
BOD_5	2.0 mg/L	Differences in duplicate series of sample results shall not exceed 2 mg/L for concentrations less than 20 mg/L.

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Glucose-glutamic acid checks	1 pair per set of samples	160–240 mg/L
Duplicate (Full dilution series)	1 per group of 10 or fewer samples	+/-(10% or 2 mg/L)
Unseeded Dilution Water Blanks	1 pair per set of samples, including 1 pair for each lot of dilution water	≤ 0.2 mg/L
DO Uptake of seed in seeded dilution water (calculated)	Calculated for each lot of seeded dilution water	0.6—1.0 mg/L

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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:**

   Perform a paint filter liquids test on a system process filter cake (solid) sample. A 14-day turnaround time is required for analysis.

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

   1 process filter cake (solid) sample.

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

   Superfund Remedial

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

   March/April 1994

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

   Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

SW-846 Method 9095, Paint Filter Liquids Test.

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

Per SW-846 Method 9095.

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 records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Per SW-846 Method 9095.

II. Quality Control Requirements

Per SW-846 Method 9095.

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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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 system process influent and effluent water samples for total suspended solids (TSS). Results will be reported as mg/L total suspended solids. A 14-day turnaround time is required for analysis.

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

70 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

EPA Method 160.2, Nonfilterable Residue. This is a gravimetric procedure.

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

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 are to be held at 4°C until analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

1. Sample aliquot volumes are selected on the basis of the following factors:

   a. During initial sample filtration, filtration rate should not drop rapidly, or require more than 5 minutes of filtration time (Increase the filter area or decrease the sample volume as needed for re-analysis),

   b. The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200 mL in volume, and

   c. Sample aliquots should not exceed 200 mL in volume.

2. Duplicate sample aliquots shall be filtered with two 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 used for calculation), or dried overnight (at least 12 hours drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4 percent weight loss from the previous weight, whichever is smaller.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation, weighing, and analysis logbooks shall be submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples, shall be provided. Dates and times of filtration of initial 100 mL volume, determination of tare weight, sample filtration, and determination of constant residue weights, shall be included.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TSS	3 mg/L	Duplicate results must agree to < 0.5 mg/L for residues ≤ 5 mg or less than 10 percent for residues > 5mg

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank (200 mL aliquots)	At least 1 per group of 10 or fewer samples	± 0.5 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 0.5 mg)

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

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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 system process influent and effluent water samples for ammonia. Results will be reported as mg/L N. A 14-day turnaround time required for analysis.

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

50 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 7 days of receipt of the samples.

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

EPA Method 350.1, Ammonia (as N). Colorimetric, Automated Phenate procedure.

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

Samples will be stored at 4°C until analysis and validation of results. Samples aliquots will be preserved in the field with H_2SO_4. The working concentration range of Method 350.1 Auto Analyzer should be 0.01 to 2 mg/L N (NH_3) or lesser concentration.

Check sample pH with wide range pH paper. If pH > 2, contact SMO for instructions. Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 14 days from sample collection. All solutions should be made with ammonia-free water.

The calibration curve must include at least five standards, one of which shall be a zero concentration standard. All standards, blanks, dilution water, and diluted samples shall be acidified with 1 mL/L H_2SO_4.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number. Results are to be in mg/L - N.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Ammonia	0.01 mg/L	Duplicate results must agree within 10% for concentrations ≥ 1/mg/L or to within 0.1 mg/L for concentrations < 1 mg/L

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
Lab Blank	At least 1 per group of 10 or fewer samples	≤ 0.1 mg/L
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 0.1 mg/L)
Matrix Spike	At least 1 per group of 10 or fewer samples	85–115% recovery
Calibration Verification Standard	At least 1 per group of 10 or fewer samples	90–110% recovery

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013442.WP5

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

SAS Number [ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V/Onalaska Groundwater Treatment Facility Startup Sampling.

B. Regional Representative: Brian Freeman

C. Telephone Number: (312) 353-5210

D. Date of Request: December 27, 1993

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 system process influent and effluent water samples for iron. Samples will be filtered and unfiltered. A 14-day turnaround time is required for analysis.

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

128 process influent/effluent water samples. All samples are low/medium concentration water samples.

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

Superfund Remedial

4. Estimated date(s) of collection:

March/April 1994

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

Method of shipment will be daily shipments by overnight carrier.

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

The laboratory will be required to provide results within 14 days of receipt of the samples.

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

Follow methods per SOW/ILM03.0 for Metals.

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

Samples will be preserved with HNO_3 to pH < 2 and stored at 4°C until analysis and validation of results.

Dilute and rerun samples with concentrations higher than the highest standard. The holding time is not to exceed 14 days from sample collection.

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 procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain-of-custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. All instrument readings shall be recorded and submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

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

Follow protocol as per stated methodology

Data rejection and nonpayment will be recommended if the laboratory does not follow the methods referenced in this SAS. Lab must submit all original field documentation and originals for data to the Region in within 14 days of receipt of the samples.

All original sample tags, chain-of-custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in Section 6 above.

11. Name of sampling/shipping contact:

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Iron	0.10 mg/L	+/- 20%

II. Quality Control Requirements

Audits Required	Frequency of Audit	Limits (± % or conc)
As required by the Statement of Works		

III. Action Required if Limits are Exceeded:

Contact SMO and Region V. Contact Brian Freeman at (312) 353-5210 for problems that might result in the delay of reporting sample results.

MKE10013443.WP5

APPENDIX C
FIELD TESTING PROCEDURES

Appendix C
Field Measurements and Monitoring

pH

Method

Electrometric

References

Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Method 150.1, 1983.

Orion SA250 pH Meter Instruction Manual, 1987, Part No. 205376-001, Orion Research Incorporated, Boston, MA.

Orion Ross pH Electrode Instruction Manual, 1988, Part No. 502700-098, Orion Research Incorporated, Boston, MA.

Sensitivity

0.01 pH unit

Range

1 to 12 pH units

Sample Holding Time

Less than 6 hours

Reagents

- pH buffer solutions for pH 4, 7, and 10
- Deionized water in squirt bottle
- 3 M KCL internal filling solution
- Storage solutions

Apparatus

- pH meter
- Combination pH and reference electrode
- Beakers, plastic or glass
- Spare battery

Calibration (most pH Meters)

1. Select either pH 4 and 7, or pH 7 and 10 buffers, whichever will bracket the expected sample concentration. Note: Mix all buffer solutions before use.

2. Place electrode in pH 7 buffer solution. Wait for the pH to stabilize and adjust CAL until pH display reads 7.0.

3. Rinse electrodes and replace pH 7 buffer with either pH 4 or 10 buffer.

4. Wait for the pH value to stabilize. Adjust SLOPE until pH display reads 4.0 for the pH 4 buffer or 10.0 for the pH 10 buffer. Note: Slope values in the 92 to 102 percent range are acceptable.

5. Rinse electrodes and replace pH 4/10 buffer with pH 7 buffer.

6. If display reading is not 7.0, repeat steps 2 through 4.

Autocalibration (Orion SA 250)

1. Select either pH 4 and 7, or pH 7 and 10 buffers, whichever will bracket the expected sample concentration. Note: Shake all buffer solutions before use.

2. Select pH mode and resolution (pH 0.1).

3. Press ISO and verify that the isopotential point is 7.0.

4. Place electrode and Automatic Temperature Compensation (ATC) probe in pH 7 buffer.

5. Press CAL. The display will alternate between 0.1 and the pH value of the buffer. Wait for the pH value to stabilize. Press ENTER. After a short pause the display will advance to 0.2.

6. Rinse electrodes and ATC probe and replace pH 7 buffer with either pH 4 or 10 buffer.

7. Wait for pH value to stabilize. Press ENTER. The letters pH will be displayed. The pH meter is calibrated and ready for use. Note: Slope values in the 92 to 102 percent range are acceptable.

8. Rinse electrode and ATC probe and place into sample. Read the pH directly.

Calibration Frequency

Daily, at the beginning and end of the day, recheck calibration with pH 7 buffer once every 10 samples and after maintenance.

Operating Procedure

1. Check all connections for tight fit.

2. Inspect electrodes (and ATC probe).

3. Check battery charge.

4. Perform calibration, at the beginning and end of the day.

5. Rinse the electrode with distilled water and then with the sample to be measured.

6. Place electrode (and ATC probe) in previously mixed sample. Immerse electrode such that junction is covered by sample.

7. When the display is stable, record sample pH.

8. Recheck calibration with pH 7 buffer solution once every ten samples.

9. After use store electrode. For short-term storage (up to 1 week) soak electrode in manufacturer's recommended storage solution. For long-term storage, the reference chamber should be filled and the filling hole securely covered.

10. Cover the sensing element and/or reference junction with its protective cap and a few drops of the manufacturer's recommended storage solution.

When calibrating the meter, use pH 4 and 7 buffers for sample with pH <7, and pH 7 and 10 buffers for samples with pH >7. Measurement of pH is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C. This is not applicable for meters equipped with an automatic temperature compensation probe.

Weak organic salts, inorganic salts, and oil and grease interfere with pH measurements. If oil and grease are visible, note on data sheet. Clean electrode as described in manufacturer's instrument manual.

Avoid rubbing or wiping electrode bulb to reduce chance of error from polarization. To ensure a quick response and free-flowing liquid junction, the sensing element and reference junction must not be allowed to dry out.

Quality Control Requirements

Accuracy will be assessed by performing two measurements on two standard buffer solutions that bracket the pH range of the samples. Recheck calibration with ph 7 buffer solution once every ten samples. Each measurement will be within ± 0.05 standard unit of pH selection. Precision will be assessed by duplicate measurements and must be less than or equal to 0.1 standard unit. Duplicates will be run at the rate of one every ten samples.

Preventive Maintenance (Frequency)

1. Check batteries (daily).

2. Perform a two-point calibration (daily and after maintenance).

3. Inspect the electrode for scratches, cracks, salt crystal buildup, or membrane/ junction deposits. Rinse off any salt buildup with deionized water and remove membrane/junction deposits as described in the manufacturer's operators manual (as needed).

4. Clean electrode by soaking in 0.1M HCL or $HN0_3$ for 30 minutes, followed by soaking in storage solution for at least 1 hour (as needed or when slow response is observed).

5. Drain the reference chamber and flush it with the manufacturer's filling solution (weekly).

Specific Conductivity and Temperature

References

Methods for Chemical Analysis of Water and Wastes, U.S. EPA Method 120.1, 1983.

YSI Models 33 and 33M S-C-T Meters, Instructions, November 1987, Item 021470, Yellow Springs Instrument Co., Yellow Springs, Ohio.

Sensitivity

1 μmho/cm @ 25°C.

Range

0.1 to 100,000 μmho/cm.

Sample Holding Time

Determine onsite or within 24 hours.

Reagents

Distilled water in squirt bottle and standard potassium chloride solution.

Reagent Preparation

1. *Stock Potassium Chloride (KCl) Solution (1.00 N):* Dissolve 74.555 g KCl in distilled water and dilute to 1,000 mL in a volumetric flask.

2. *Standard Potassium Chloride Solution (0.01 N):* Dilute 10.0 mL of stock 1.00 N KCl solution to 1,000 mL with distilled water using a volumetric pipet and flask.

Apparatus

Conductivity meter and electrodes. Beakers or jars, plastic, or glass; spare batteries, size D alkaline.

Calibration Procedure

1. Switch mode to OFF and unplug the probe, correct meter zero (if necessary) by turning the adjustment screw so that the meter needle coincides with the zero on the conductivity scale.

2. Switch mode to REDLINE, correct meter redline (if necessary) by turning the adjustment screw so that the meter needle coincides with the redline on the meter face. If this cannot be accomplished, replace the batteries.

3. Plug the probe into the probe jack.

4. Place the probe in the 0.01 N standard potassium chloride solution. Record temperature (°C) and conductance (micromho/cm).

5. Correct conductivity reading for temperature. This value must correspond ($\pm$10 percent) to the expected value in Table 1. If the calibration fails, then appropriate corrective action must be performed and the instrument recalibrated.

Note: The temperature probe should be calibrated against a NBS, an ATSM standard or equivalent thermometer before each sampling event.

Operation Procedure

1. Perform calibration at end and beginning of the day.

2. Switch mode to TEMPERATURE. Allow time for the probe temperature to come to equilibrium with that of the water before reading. Read the temperature on the bottom scale of the meter in degrees Celsius.

3. Switch mode to X100. If the reading is below 50 on the 0 to 500 range (5.0 on the 0 to 50 mS/m range), switch to X10. If the reading is still below 50 (5.0 mS/m), switch to the X1 scale. Read the meter scale and multiply the reading by the mode factor. The answer is expressed in microohms/cm. Measurements are not temperature compensated.

4. When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2 percent; if greater, the probe is fouled and the measurement is in error. Clean the probe and remeasure.

Operating Suggestions

- Obstructions near the probe can disturb readings.
- When the calibration test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oil, and organic matter are the most likely contaminants.
- Caution: Do not touch the electrodes inside the probe. The plating material is soft and can be scraped off.
- If cleaning does not restore the probe performance, replatinizing may be required. Always rinse the probe thoroughly in tap water, then in distilled or deionized water after cleaning and before storage. It is best (not required) to store conductivity cells in deionized water.
- Most problems in obtaining good records with monitoring equipment are related to electrode fouling and to inadequate sample circulation.

Calibration Frequency

At the beginning and end of the day or after maintenance, recharge battery after each use. Factory checkout and calibration shall be yearly or when malfunctioning.

Calculations

Calculate conductivity using the formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

where:

G_{25} = conductivity at 25°C, μmho/cm
T = temperature of sample, °C
G_T = conductivity of sample at temperature T, μmho/cm

Table 1
Conductivity Meter Calibration Table

Temperature (°C)	Conductivity (μmho/cm)
15	1,141.5
16	1,167.5
17	1,193.6
18	1,219.9
19	1,246.4
20	1,273.0
21	1,299.7
22	1,326.6
23	1,353.6
24	1,380.8
25	1,408.1
26	1,436.5
27	1,463.2
28	1,490.9
29	1,518.7
30	1,546.7

Quality Control Requirements

The accuracy of conductivity measurements will be assessed by measurement with a 0.01 N standard KCl solution before sample analysis and at the end of the day. Accuracy of measurements will be ± 5 percent of the standard. Precision will be assessed by analysis of duplicate measurements which will have a relative percent difference of ≤ 15 percent. Duplicates will be run at the rate of one every ten samples. The thermometer on the conductivity meter will be checked before each sampling event for accuracy against an ASTM, NBS standard or equivalent thermometer. Accuracy of the measurement shall be ± 1°C.

Preventive Maintenance

- The only maintenance required in battery replacement (every 200 hours or as needed).
- Recalibration (if necessary) should be done at the factory.

Field Filtering

Reference

EPA 1979, Metals 5.

Sample Handling

Filter as soon as possible after sample collection.

Reagents and Apparatus

1. 10 percent HNO_3 solution in a squirt bottle and in a liter plastic bottle
2. DI water
3. Plastic forceps
4. Filtration apparatus
5. 0.45 μm membrane filters
6. Glass fiber prefilters
7. Peristaltic pump (if filter stand is used or "bottle-to-bottle" filtering is performed)

Reagent Preparation

1. *10 percent HNO_3 solution:* Add about 900 mLs of DI water to a 1 liter Erlenmeyer flask. Using a graduated cylinder, add 100 mLs concentrated HNO_3 to the DI water while stirring.

Procedure—Filter Stand

1. Using plastic forceps, place a 0.45 μm filter on top of filter apparatus.

2. Place a prefilter on top of membrane filter.

3. Place top onto filter apparatus. Screw wing nut bolts down until even and snug. Finish tightening with plastic wrench.

4. Attach end of PVC hosing from pump to filter apparatus.

5. Run 50 to 100 mLs of HNO_3 through apparatus, rinse with 50 to 100 mLs DI water. Do not collect this filtrate.

6. Place sample bottle under outlet.

7. Turn pump on, run sample through filter, and collect filtered sample from bottom of apparatus.

8. Shut off pump.

9. Rinse twice with DI water, remove filter and dispose, proceed as above for next sample.

10. Run a DI water blank every 10 to 20 samples.

Notes

Samples with high sediment can be filtered through several membranes with increasing pore size and several prefilters. The 0.45 μm membrane filter should always be on the grid, and the coarsest filters on the top.

Procedure—Disposable Inline Filter

1. When filtering from a bottle into another bottle, attach tubing from pump outlet to filter inlet (note flow direction on the filter housing).

2. Place sample bottle under outlet.

3. Turn pump on, run sample through filter and collect filtered sample from outlet.

4. Shut off pump.

5. Remove filter and discard.

6. Run 50 to 100 mL 10 percent HNO_3 through pump tubing, discarding rinsate.

7. Run 50 to 100 mL DI water through pump tubing, discarding rinsate. Allow enough time to remove all water from the tubing before turning off the pump.

8. Run a DI water blank every 10 to 20 samples.

Note: To collect filtered sample directly from a sample port or dedicated pump, attach an inline sample field filter directly to the pump or port discharge tube and allow the filtered sample to flow directly into the bottle.

HNu Monitoring

References

HNu Model PI101 Portable Photoionization Analyzer Instruction Manual, December 1985, HNu Systems, Inc., Newton, MA.

HNu Model ISP1 101 Intrinsically Safe Portable Photoionization Analyzer Instruction Manual, January 1986, HNu Systems, Inc., Newton, MA.

Sensitivity

0 to 20 ppm at full-scale detection at span = 9.8 ppm; 10.2 ev Probe.

Range

0.1 to 2,000 ppm.

Calibration Gas

Isobutylene at 100 ppm.

Calibration

By analyzing a gas of known concentration, the HNu is easily calibrated. Isobutylene is typically used as the calibration gas with the instrument calibrated to benzene equivalents. When calibrating the HNu, always remember to deliver the calibration gas at ambient temperature and pressure, handle gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use, all gas cylinders must have proper regulators.

Calibration Procedure

1. Identify the probe by lamp label.

2. Attach the probe to the readout unit. Twist connector clockwise until locked.

3. Affirm the relative photoionization sensitivity (PS) calibration gas. [The required reading for isobutylene to read in benzene equivalents is equal to isobutylene ppm $\times$ PS (Isob.)/PS (benzene).]

4. Turn the function switch to battery check position. The indicator should read within the green arc. If indicator is below the green arc or if red L&D comes on, battery must be charged.

5. Zero the instrument by turning function switch to standby and rotate potentiometer until the meter reads zero.

6. Connect sampling hose to regulator outlet and the other end to sampling probe of HNu.

7. Crack regulator valve.

8. Adjust span potentiometer to obtain proper reading.

9. If calibration can not be achieved, clean the UV light source window using lens paper and HNu cleaning compound.

10. If still unable to calibrate, perform preventive maintenance. Return to factory if those procedures do not work.

Calibration Frequency

Daily or after maintenance. Recharge battery after each use. Factory check out and calibration shall be yearly or when malfunctioning.

Preventive Maintenance (Frequency)

Battery (Daily)

Check the battery charge during each period of operation. When the meter needle falls below the green zone or the low-battery indicator light illuminates recharge battery. Do not use the instrument when light is on. When not operating, leave the analyzer assembled and connected to the battery charger. In case of emergency, the analyzer may be used with a low-battery charge.

Gas Cylinders and Valves (After Installation)

All gas supply lines must be leak tested. Leakage can be determined by testing line connections and valve stems with a commercially available leak test solution. Leaks are generally stopped by tightening the fitted surfaces but may require new hardware.

Air Sampling Stream (Initially, then as Needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. A fan draws gas in through the probe and ion chamber. Small fluctuations in the flow rate will not affect the measurement. A major obstruction to the flow rate will prevent proper operation and lengthen response time. Refer to the manufacturer's instrument manual for specific procedures.

Quality Control Requirements

Precision of ± 30 percent. Daily calibration.

OVA Monitoring

Reference

Model OVA 128 Century Organic Vapor Analyzer, Instruction, December 1985, Foxboro, New Haven, CT.

Sensitivity

0.1 ppm (methane).

Range

0 to 1,000 ppm.

Calibration Gas

Methane gas at 100 ppm.

Calibration

By analyzing a gas of known concentration, the OVA is easily calibrated. Methane in air at a concentration of 100 ppm is typically used as the calibration mixture, although the OVA can be calibrated to many other compounds. Primary calibration of an OVA is performed at the factory. When calibrating the OVA, always remember to deliver the calibration gas at ambient temperature and pressure, handle the gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use, and all gas cylinders must have proper regulators.

Calibration Procedure

1. Connect probe readout assembly to sidepack unit.

2. Check battery condition by moving INSTR Switch to BATT.

3. Turn INSTR to ON and allow 5 minutes to warm up.

4. Use calibration adjust knob to set needle to level desired for activating alarm. If alarm level is not zero, the calibration switch must be set to appropriate level.

5. Turn volume knob fully clockwise.

6. Turn the alarm level adjust knob until the audible alarm is activated.

7. Move calibration switch to 1X and adjust meter reading to zero using zero calibration adjustment.

8. Turn pump switch on.

9. Open hydrogen tank valve and hydrogen supply valve. Wait 1 minute.

10. Depress ignitor button until burner lights (not more than 6 seconds).

11. Set calibration switch to 10X.

12. Connect sampling hose to regulator outlet and the other end to sampling probe of OVA.

13. Crack the regulator valve.

14. Check to see if proper reading is achieved.

15. If reading is ± 10 percent from expected value, return to factory for recalibration.

Calibration Frequency

Daily or after maintenance, recharge battery after each use. Factory check out and calibration shall be yearly or when malfunctioning.

Preventive Maintenance

Battery (Daily)

Check battery condition by moving the INSTR switch to the BATT position. Recharge the battery if a low charge is indicated. Do not use the instrument with a low battery charge. When not operating, leave the analyzer connected to a battery charger. Never recharge battery in a hazardous environment.

Particle Filter (As Needed)

Particle filters (primary and secondary) remove foreign matter (>10 microns) from the sample stream. These filters must be in the sample line whenever the instrument is operating. A decrease in flow rate may indicate a plugged filter.

Sampling Fixtures (As Needed)

Sampling fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particulate matter.

Hydrogen Fuel, Calibration Gas, and Valves (Initially, after Changes)

Use prepurified or zero-grade hydrogen (certified total hydrocarbons as methane <0.5 ppm recommended). All fuel and calibration gas supply lines should be leak tested. Leakage can be determined by testing line connections and valve stems with a commercially available leak test solution. Leaks are usually stopped by tightening the fitted surfaces but may require new washers or hardware.

Air Sampling Pump System (Initially, As Needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. The OVA is equipped with a flow gauge that provides a method to check for air leaks. Refer to the manufacturer's instrument manual for specific procedures.

Contaminated Control (As Needed)

Background readings may be relatively high under normal ambient conditions. The sources of high background are normal methane background, contaminated hydrogen supply gas, and contamination in the air sample line. Background readings less than 1 ppm are generally accepted since sample measurement is additive to that background. However, the low background values are more desirable. High background is commonly corrected by running the OVA for an extended time in a clean area. Refer to the manufacturer's instrument manual for specific analysis and correction procedures.

Quality Control Requirements

Precision of ±30 percent. Daily calibration.

< < Add a section on DO meter > >

MKE10013497.WP5

APPENDIX D
SAMPLE DOCUMENTATION AND PACKING AND SHIPPING INSTRUCTIONS

Appendix D
Sample Documentation and Packing and Shipping Instructions

Sample Documentation Instructions

Sample Identification Matrix (Figure 1)

1. Enter site name.

2. Enter project number.

3. Enter the case number and/or SAS number.

4. Enter the CRL log number.

5. Specify the sample matrix using the two- or three-digit codes described in Section 10 of the Sampling Plan.

6. Enter the sample number.

7. Enter the organic traffic label number or the SAS sample number.

8. Enter the inorganic traffic label number.

9. Enter the chain-of-custody number.

10. Indicate the laboratory to be doing the analysis (abbreviations may be used as they are shown on the current laboratory list).

11. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g., 081292).

12. Enter the shipping date.

13. Enter the airbill number of the shipment.

14. List sample tag numbers corresponding to sample containers shipped under the traffic label number listed in either box 7 or 8.

15. List the QC lot numbers of the containers matching the tag numbers listed in Item 14.

Note: The date recorded on this form must be suitable for computer entry. Each entry must be flush left and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, chain-of-custody, and airbill numbers.

Sample Tag (Figure 2)

1. Enter date of sampling.

2. Enter time of sampling (military time only).

3. Specify "grab" or "composite" sample with an "X."

4. Enter CH2M HILL sample identification code.

5. Obtain signature of sample team leader.

6. Indicate preservative used (if any) with an "X."

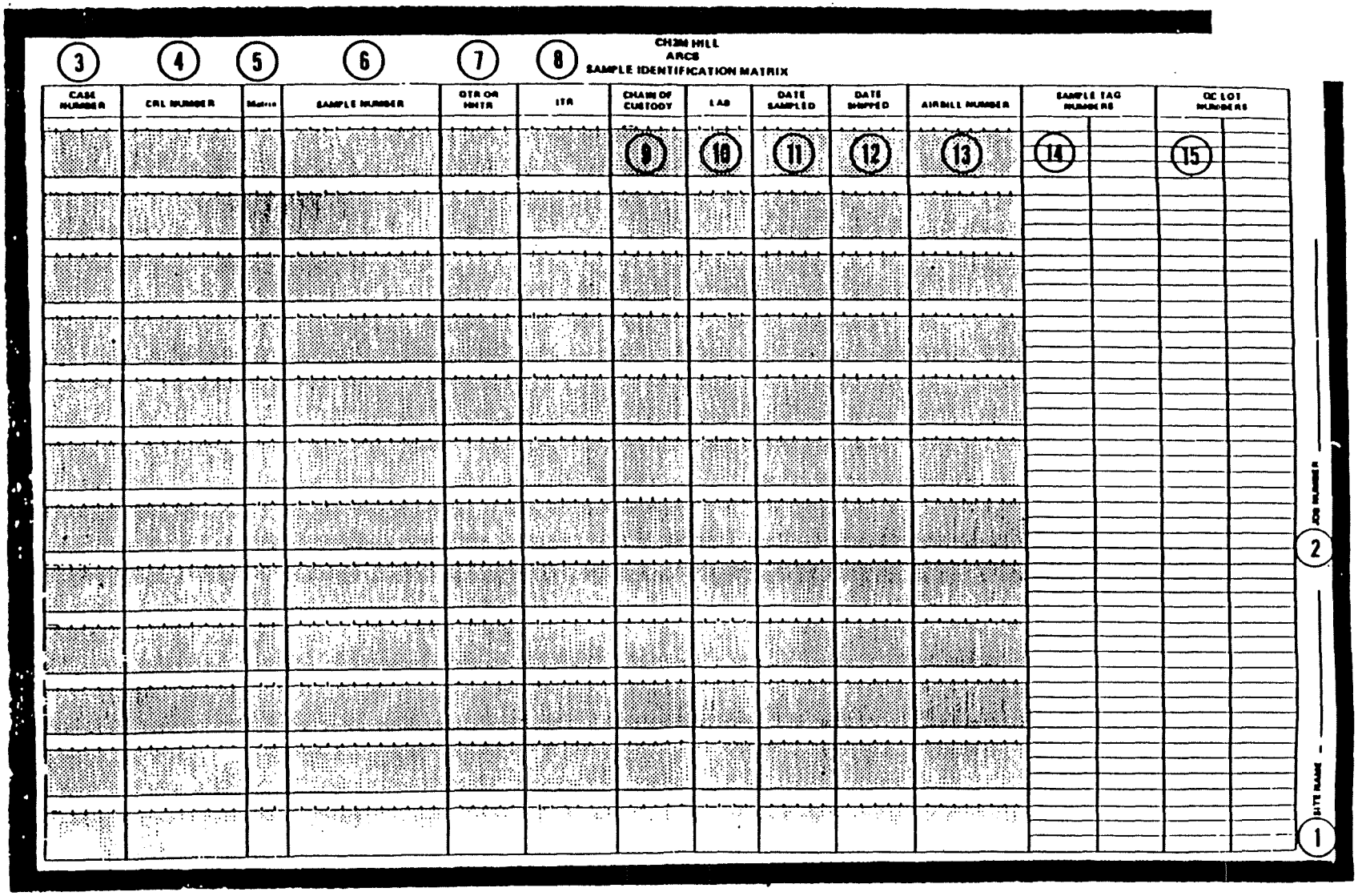
7. Specify all parameters for analysis by placing an "X" to the right of each one.

8. Indicate the sample number (for CLP Lab) or CRL log number (for CRL).

9. Indicate case number and/or SAS number (e.g., Case No. 1234 and/or SAS No. 5678E).

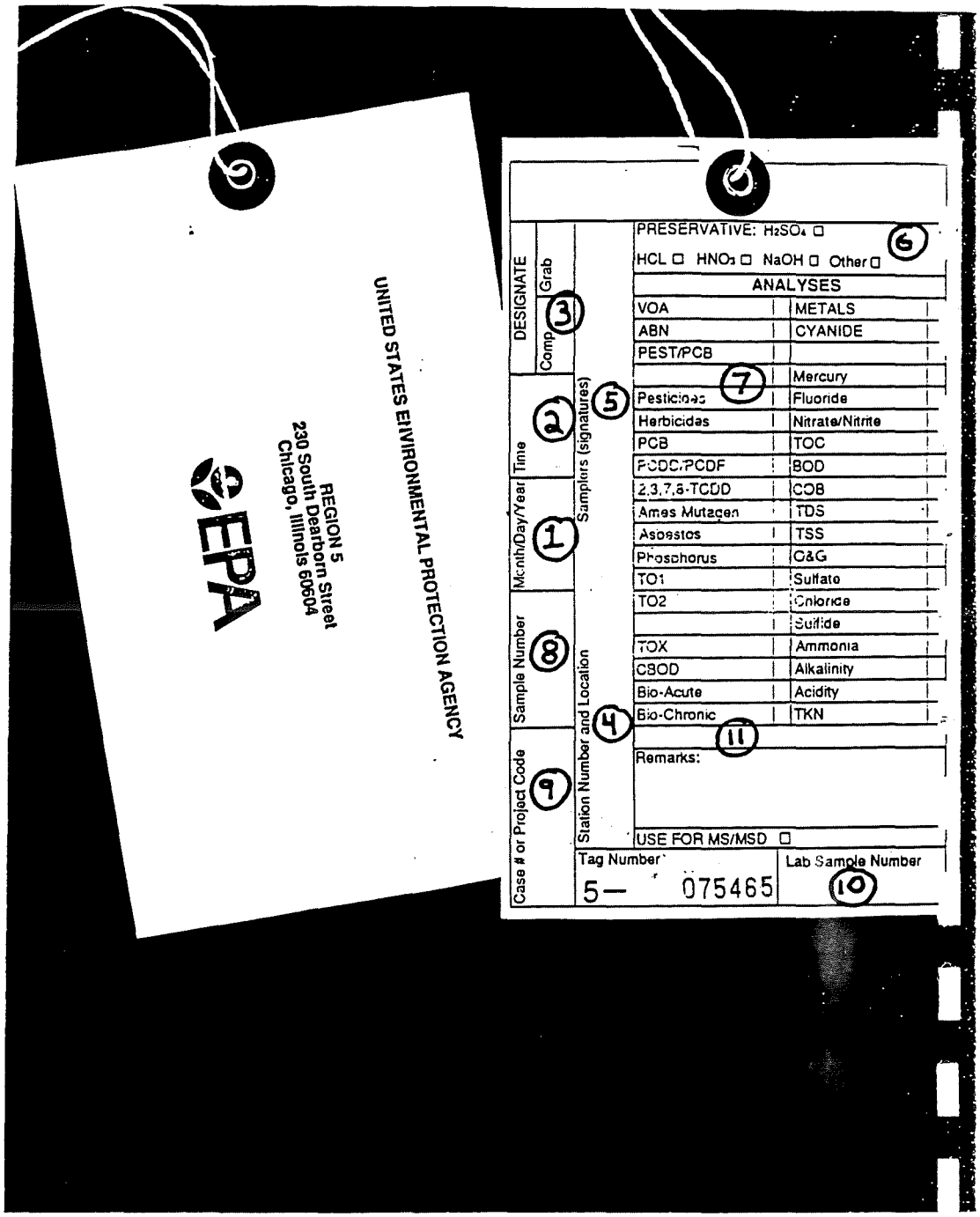
10. Leave BLANK (for laboratory use only).

11. Enter any desired analyses not listed on menu (e.g., PCBs, ammonia, sulfide, etc.) and mark box with an "X."



NOTE: For purposes of illustration this form has been reproduced at 50% of original size.

FIGURE 1



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 2

Inorganic Traffic Report (Figure 3)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

4b. Enter city and state of site.

4c. Enter spill ID No. (obtained from the PM or Data Coordinator).

5. Enter EPA region number (e.g., V).

6. Enter sample team leader's company/office.

7. Enter sample team leader's name.

8. Enter laboratory name and address, and laboratory contact.

9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the ITR Label Number.

13. Indicate sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from box 5 on ITR.

14. Specify sample concentration with an L, M, or H indicating contamination level.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, MS/MSD or field duplicate.

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete**.

Organic Traffic Report (Figure 4)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

4b. Enter site city and state.

4c. Enter spill ID No. (obtained from the PM or Data Coordinator).

5. Enter EPA region number (e.g., V).

6. Enter sample team leader's company/office.

7. Enter sample team leader's name.

8. Enter laboratory name and address and laboratory contact.

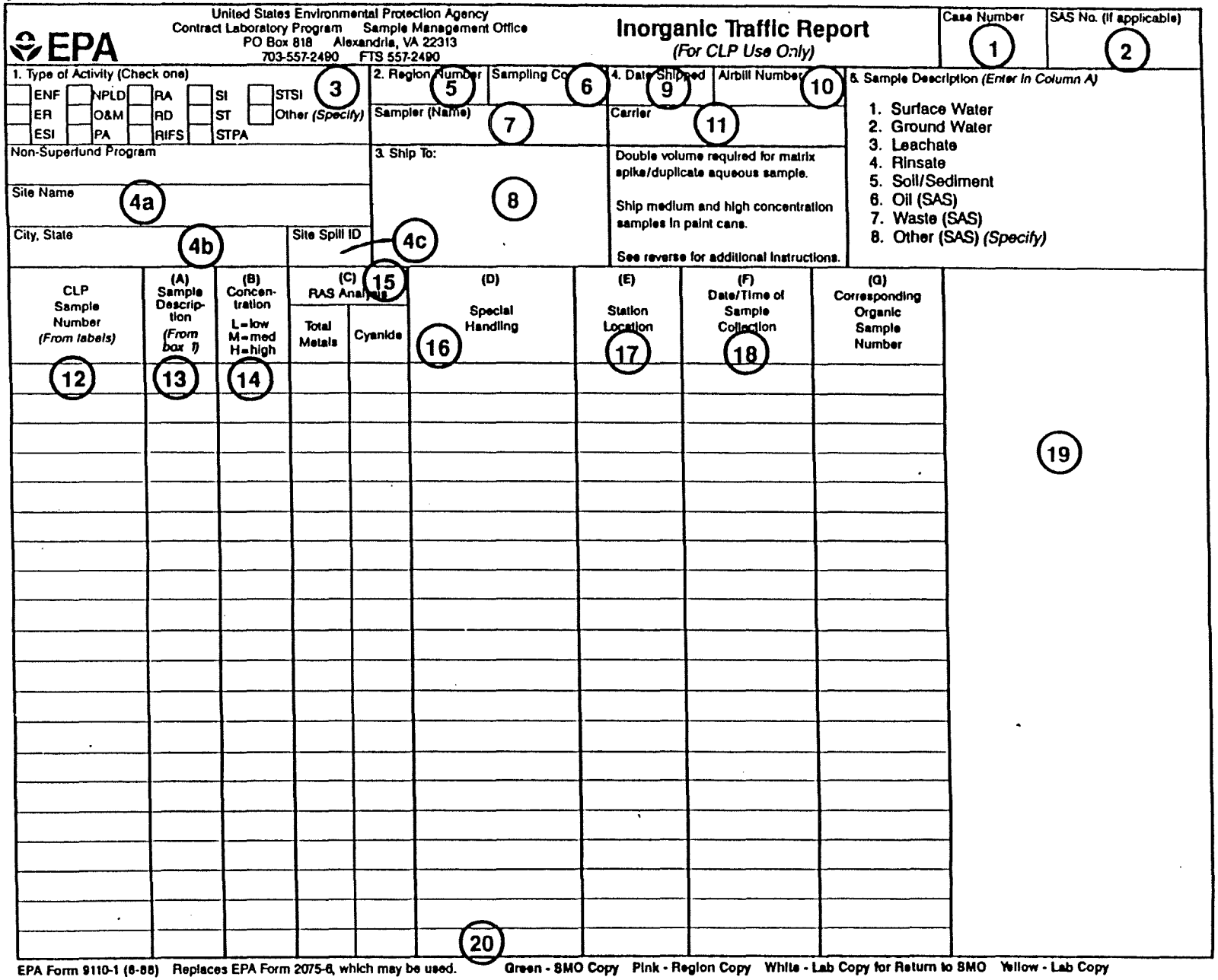
9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the OTR Label Number.

13. Specify sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from box 5 on OTR.



EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report

(For CLP Use Only)

Case Number (1) | SAS No. (If applicable) (2)

1. Type of Activity (Check one) (3)
ENF | NPLD | RA | SI | STSI
ER | O&M | RD | ST | Other *(Specify)*
ESI | PA | RIFS | STPA

2. Region Number (5) | Sampling Co (6)

Sampler (Name) (7)

4. Date Shipped (9) | Airbill Number (10)

Carrier (11)

5. Sample Description *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

Non-Superfund Program

Site Name (4a)

City, State (4b) | Site Spill ID (4c)

3. Ship To: (8)

Double volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

CLP Sample Number *(From labels)* (12)	(A) Sample Description *(From box 1)* (13)	(B) Concentration L=low M=med H=high (14)	(C) RAS Analysis (15) Total Metals	(C) RAS Analysis (15) Cyanide	(D) Special Handling (16)	(E) Station Location (17)	(F) Date/Time of Sample Collection (18)	(G) Corresponding Organic Sample Number	(19)
					(20)				

EPA Form 9110-1 (6-88) Replaces EPA Form 2075-6, which may be used. Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

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FIGURE 3

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report
(For CLP Use Only)

Case Number (1) | SAS No. (if applicable) (2)

1. Type of Activity (Check one) (3)
☐ ENF ☐ NPLD ☐ RA ☐ SI ☐ STSI
☐ ER ☐ O&M ☐ RD ☐ ST ☐ Other *(Specify)*
☐ ESI ☐ PA ☐ RIFS ☐ STPA

2. Region Number (5) | Sampling Co (6)

Sampler (Name) (7)

4. Date Shipped (9) | Airbill Number (10)

Carrier (11)

5. Sample Description *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

Non-Superfund Program

Site Name (4a)

City, State (4b) | Site Spill ID (4c)

3. Ship To: (8)

Triple volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

CLP Sample Number *(From labels)* (12)	(A) Sample Description *(From box 1)* (13)	(B) Concentration L=low M=med H=high (14)	(C) RAS Analysis (15) VOA	BNA	Pest/ PCB	(D) Special Handling (16)	(E) Station Location (17)	(F) Date/Time of Sample Collection (18)	(G) Corresponding CLP Inorganic Sample Number	(19)
						(20)				

EPA Form 9110-2 (8-88) Replaces EPA Form 2075-7, which may be used. Blue - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

For purposes of illustration this form is reproduced at 80% of original size

FIG 4

14. Specify the sample concentration with an L, M, or H, indicating contamination level.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a blank, MS/MSD or field duplicate (replicate).

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete**.

SAS Packing List (Figure 5)

1. Enter assigned SAS case number.

2. Enter EPA region number (e.g., V).

3. Enter sample team leader's name.

4. Enter sample team leader's company/office and phone number.

5. Enter date sample was taken.

6. Enter date of shipment.

7. Enter site name.

8. Enter laboratory name and address.

9. Enter name of laboratory contact.

10. List SAS sample numbers, which should include the SAS number.

11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag No. 5-48246).

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)	Site Name/Code: (7)	Attn: (9)	Received By:
(phone)			

	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

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FIGURE 5

12. Leave BLANK (for laboratory use only).

Chain-of-Custody Record (Figure 6)

1. Enter first six digits of the CRL sample identification code.

2. Leave blank.

3. Obtain full signature of sample team leader and signed initials of active team members (including paperwork person).

4. Enter last three digits of the CRL sample identification code.

5. List sampling dates for all samples.

6. List sampling times for all samples (military time only).

7. Indicate ''grab'' or ''composite'' sample with an ''X.''

8. List CH2M HILL sample numbers.

9. Enter number of containers per sample.

10. List analyses individually.

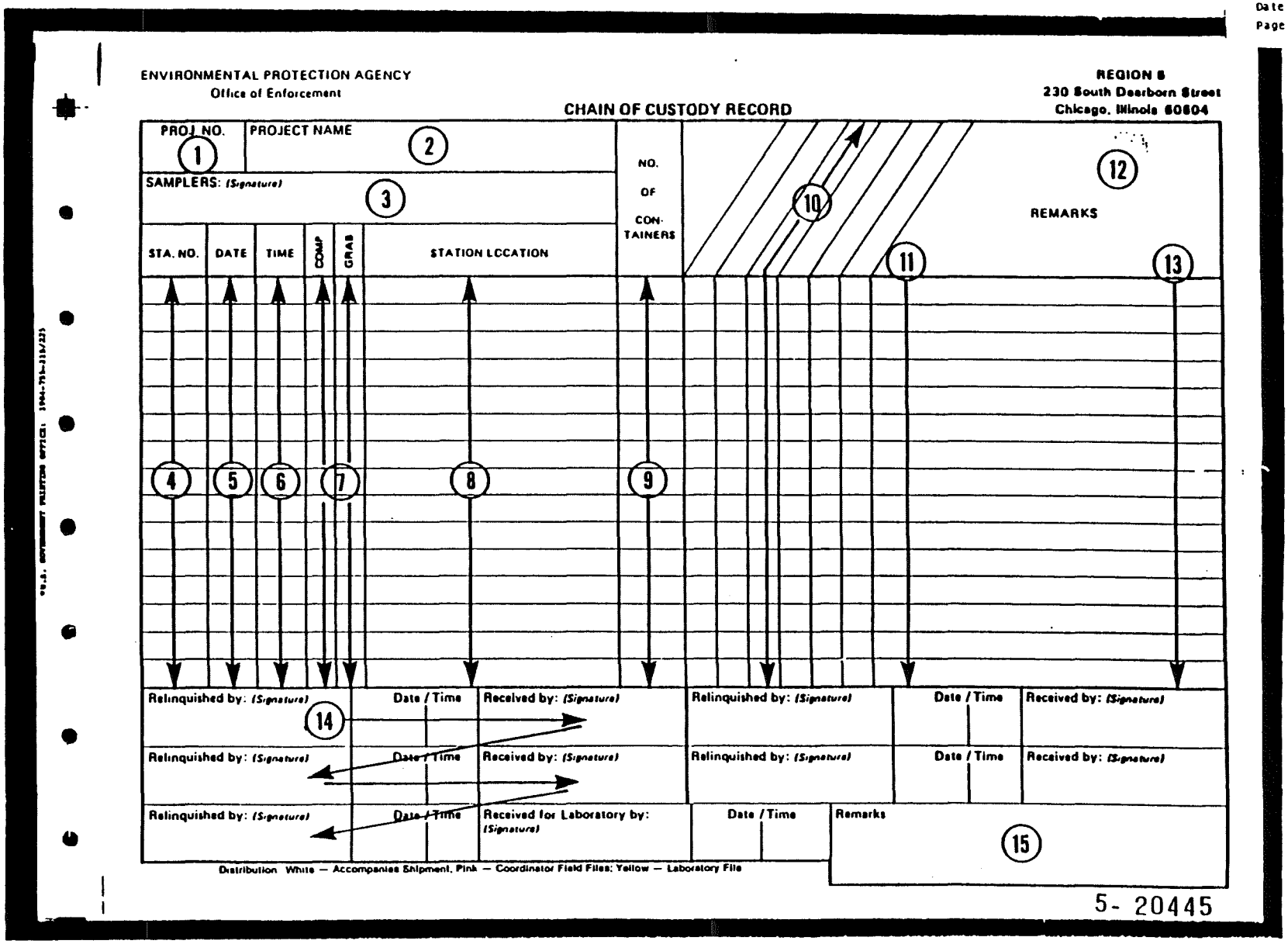
11. Enter column heading for traffic label number and list serial numbers for corresponding sample identification codes.

12. Write in the words ''CASE No. :'' or ''SAS No. :'' and enter the correct number.

13. Enter column heading for ''tag number'' and list tag numbers for each sample container.

14. Obtain signature of sample team leader and carry out chain-of-custody procedures.

15. State carrier service and airbill number, lab service, and custody seal numbers.



ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

CHAIN OF CUSTODY RECORD

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

PROJ. NO. (1) | PROJECT NAME (2)

SAMPLERS: (Signature) (3)

STA. NO.	DATE	TIME	COMP	GRAB	STATION LOCATION	NO. OF CONTAINERS
(4)	(5)	(6)	(7)		(8)	(9)

(10) (11) REMARKS (12) (13)

Relinquished by: (Signature) (14)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks (15)	

Distribution: White — Accompanies Shipment, Pink — Coordinator Field Files; Yellow — Laboratory File

5- 20445

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FIGURE 6

Combined Chain of Custody and Traffic Report Forms (Figure 7)

A. Project Code: Leave blank.

B. Account Code: Leave blank.

C. Regional Information: If sampling is in support of oversight activities, indicate here. If not—leave blank.

D. Non-Superfund Program: If sampling is not done under the Superfund program, enter the name of the program (e.g., RCRA).

E. Site Name, City, State: Complete as instructed.

F. Site Spill ID: Enter ID code provided by the office.

G. Region No.: Enter "Region 5."

H. Sampling Company: Enter "CH2M HILL."

I. Sampler Information: Complete as instructed.

J. Type of Activity:

SF—Superfund lead
PRP—PRP lead
ST—State lead
FED—Federal lead
PA—Preliminary assessment
SSI—Screening site investigation
LSI—Listing site investigation
RIFS—Remedial Investigation/Feasibility Study
RD—Remedial design
O&M—Operation & Maintenance
NPLD—National Priorities List delete
CLEM—Classic emergency
REMA—Removal assessment
REM—Removal
OIL—Oil response
UST—Underground storage tank response

COMBINED CHAIN OF CUSTODY AND TRAFFIC REPORT FORMS

EPA — United States Environmental Protection Agency, Contract Laboratory Program, Sample Management Office, PO Box 818, Alexandria, VA 22313, 703-557-2490, FTS 557-2490

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

SAS No. (if applicable) (M) | Case No. (M)

1. Project Code (A) | Account Code (B)
2. Region No. (G) | Sampling Co. (H)
4. Date Shipped (K) | Carrier (K)
Regional Information (C) | Sampler (Name) (I) | Airbill Number (K)
Non-Superfund Program (D) | Sampler Signature (I) | 5. Ship To (L)
Site Name (E) | 3. Type of Activity (J): SF, PRP, ST, FED; Remedial: PA, SS, LSI, NPLD; Removal: CLEM, REMA, REM, OIL, UST
City, State (F) | Site Spill ID (F)

6. Preservative (Enter in Column D): 1. HCl 2. HNO_3 3. $NaHSO_4$ 4. H_2SO_4 5. Other (SAS) (Specify) 6. Ice only N. Not preserved

7. Sample Description (Enter in Column A): 1. Surface Water 2. Ground Water 3. Leachate 4. Rinsate 5. Soil/Sediment 6. Oil (SAS) 7. Waste (SAS) 8. Other (SAS) (Specify)

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp/ Grab	D Preservative from Box 6	E RAS Analysis: VOA	BNA	Pest/ PCB	High ARO/ TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/ Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg. Samp. No.	K Designated Field QC
(N)	←			(O)	→				(P)	(Q)	(R)	(S)	(T)	(U)

Shipment for Case complete? (V) | Page 1 of (W) | Sample used for a spike and/or duplicate (X) | Additional Sampler Signatures (Y) | Chain of Custody Seal Number (Z)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature) (AA)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	

EPA Form 9110-2 (Rev. 5-91) Replaces EPA Form (2075-7), previous edition which may be used

DISTRIBUTION: Blue - Region Copy Pink - SMO Copy White - Lab Copy Yellow - Lab Copy for Return to SMO

Split Samples ☐ Accepted (Signature) (BB) ☐ Declined

FIGURE 7

K. Shipping Information: Complete as instructed.

L. Ship To: Enter lab name, address and sample recipient/custodian.

M. SAS/Case No.: Complete as instructed.

N. Sample Numbers: For routine organic/inorganic samples, enter the CLP numbers from the "stick-on" labels. For SAS samples, enter the SAS sample numbers (SAS number plus a unique sequential numeric suffix).

O. Sample Information: Complete as instructed.

P. Regional Specific Tracking Number or Tag Number: Enter sample tag number(s).

Q. Station Location Number: Enter sample identifier (as defined in the QAPP).

R. Time/Date: Complete as instructed. Use military time.

S. Sampler Initials: OPTIONAL.

T. Corresponding CLP Organic/Inorganic Sample Number: Enter CLP sample number (from "stick-on" labels) of corresponding sample from same location. Not applicable to SAS forms.

U. Designated Field QC: Indicate QC status when applicable (field blanks, trip blanks, duplicates, MS/MDS, etc.).

V. Sampling Status: Is the sampling for this Case/SAS complete? Circle one.

W. Page 1 of : Record number of documents enclosed in cooler.

X. MS/MSD and/or Duplicate: List samples.

Y. Additional Samplers Signatures: OPTIONAL.

Z. Chain of Custody Seal No.: Enter the numbers that appear on the custody seals to be used to seal the cooler (there should be two).

AA. "Relinquished by" and "Time/Date:" Complete as instructed. Use military time.

BB. Split Samples: PRP representative (PRP contractor) shall sign off here if work is oversight.

Distribution: The **Lab Copy** and **Lab Copy for Return to SMO** are included with the shipment. The **Region Copy** and **SMO Copy** are returned to the office.

Notice of Transmittal (Figure 8)

1. Enter name of team leader.

2. Enter team leader's firm name.

3. Enter CH2M HILL project number.

4. Enter case number.

5. Enter date.

6. Enter number of samples shipped.

7. Enter matrix of samples.

8. Enter the site name in words.

9. Enter the location of the site (city, state).

Central Regional Laboratory Sample Data Report (Figure 9)

The Central Regional Laboratory Sample Data Report is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number or SAS number.

2. Enter the site name.

3. Enter the laboratory name.

4. Enter the date shipped.

Figure 8

Notice of Transmittal

Date:

To:

CH2M HILL
411 E. Wisconsin Avenue, Suite 1600
P.O. Box 2090
Milwaukee, WI 53201

Attn: Cherie Wilson

From: _____ / _____
(name) (firm)

CH2M HILL Project No.: _____

Enclosed are appropriate copies of the sample documentation forms completed under

Case No. _____ for the _____, 19___ shipment of _____ _____ samples
(qty) (matrix)

from the _____ site located in _____, _____.

MKE10013499.WP5

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 or SAS Packing List No.	INORGANIC TRAFFIC REPORT NUMBER	WATER OR LIQUIDS															SEDIMENTS or SOILS												
			ACID/BASE NEUTRAL CPDS ORGANIC SCAN UG/L TDX117S74	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN UG/L TDX117S44	WATER POLYCHLORINATED BIPHENYLS UG/L PES 117144	METHYL CHLORINATED PESTICIDES UG/L PES117134	TOTAL METALS IN WATER UG/L MET1111	WATER CYANIDE UG/L MAN74018	NITRATE-NITRITE MG/L MAN7284	AMMONIA MG/L MAN7284	RESIDUE FILTRABLE TDS MG/L MAN7282	RESIDUE NON-FILT TSS MG/L MAN7272			ACID/BASE NEUTRAL CPDS ORGANIC SCAN MG/KG TDX218772	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN MG/KG TDX218427	SEDIMENTS POLYCHLORINATED BIPHENYLS MG/KG PES211412	SEDIMENT CHLORINATED PESTICIDES MG/KG 211272	TOTAL METALS MG/KG MET413	CYANIDE MG/KG MAN4930	EP TOXICITY METALS MG/KG	AMMONIA MG/KG MAN3925							
(9)	(10)	(10)				(11)																								

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 9

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.

Packaging and Shipping Procedures

Low-Concentration Samples

1. Prepare coolers for shipment.

    - Tape drains shut.
    - Affix ''This Side Up'' labels on all four sides and ''Fragile'' labels on at least two sides of each cooler.
    - Place mailing label with laboratory address on top of coolers.
    - Fill bottom of coolers with about 3 inches of vermiculite or use performed poly-foam liner.
    - Place appropriate traffic reports, SAS packing lists, or regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.

2. Arrange decontaminated sample containers in groups by sample number.

3. Mark volume levels on bottles with a grease pencil.

4. Secure appropriate sample tags around lids of containers with string or wire.

5. Secure container lids with strapping tape.

6. Arrange containers in front of assigned coolers.

7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.

8. Seal each container within a separate plastic bag.

9. Arrange containers in coolers so that they do not touch.

10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags and placed on and around the containers (especially on VOA vials).

11. Fill remaining spaces with vermiculite (or place poly-foam liner cover on top of samples).

12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express.

13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.

14. Close lid and latch.

15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape (Figure 10).

16. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals (see Figure 10).

17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.

18. Telephone the SMO in Alexandria, Virginia.

    (Note: This step should be omitted for samples sent to the CRL).

    Ms. Leslie Braun (subject to change)
    703/557-2490

    Provide the following information:

    - Your name
    - Project name
    - Case number
    - Number of samples sent to each laboratory for analysis
    - Airbill numbers

    This must be done IMMEDIATELY following sample shipment. If the SMO is closed at that time, call in the information first thing the next day.

Medium- and High-Concentration Samples

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required (Figures 10 and 11). Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed in the cooler, as indicated below.

1. Place approximately ½ inch of vermiculite in the bottom of the can.

2. Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.

3. Place the jar in the can and fill the remaining volume with vermiculite.

4. Close the can and secure the lid with metal clips.

5. Write the traffic report number on the lid.

6. Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.

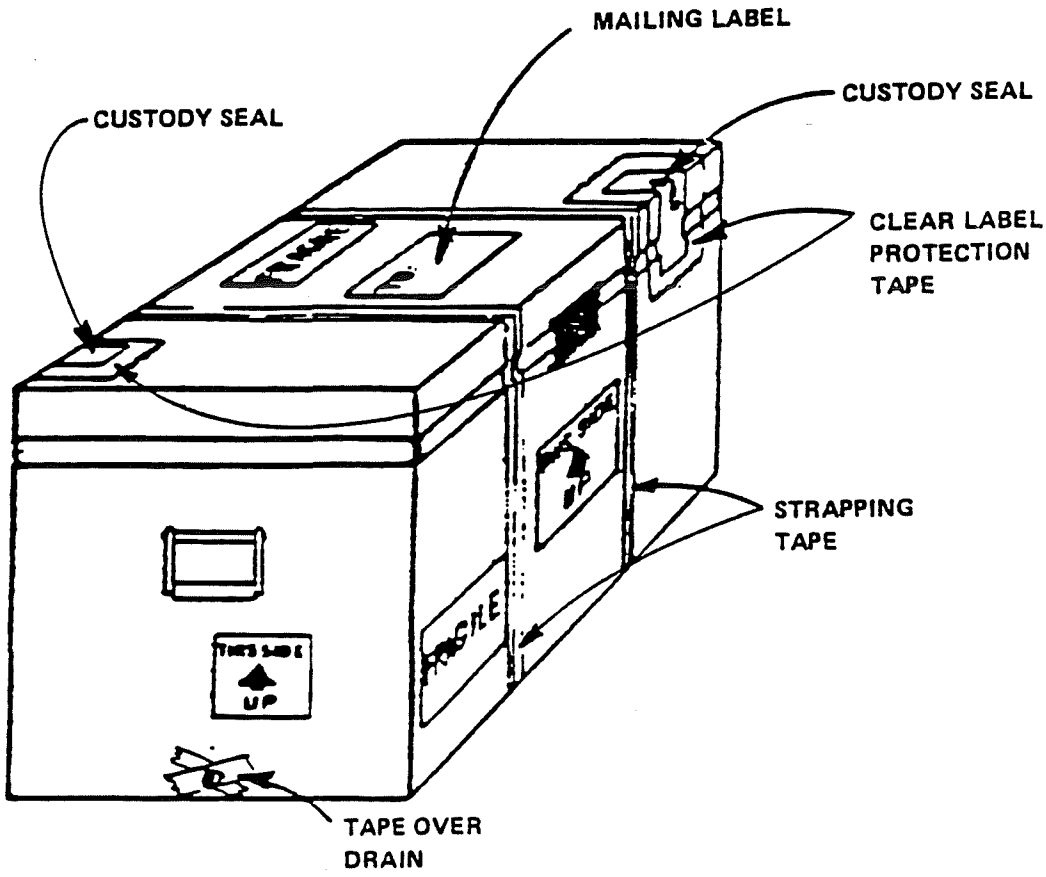
MAILING LABEL
CUSTODY SEAL
CUSTODY SEAL
CLEAR LABEL
PROTECTION
TAPE
STRAPPING
TAPE
UP
TAPE OVER
DRAIN

FIGURE 10

7. Place the cans in the cooler.

Special Instructions for Shipping Samples by Federal Express (Figures 11 and 12)

1. Label cooler as hazardous shipment.

   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker saying "This Side Up" on two adjacent sides.
   - Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NANo. 9188.
   - Label cooler with "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.

2. Complete the special shipping bill for restricted articles (Figures 10 and 11).

   - Under *Proper Shipping Name,* write "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.
   - Under *Class,* write "ORM-E."
   - Under *Identification No.,* write NA No. 9188.

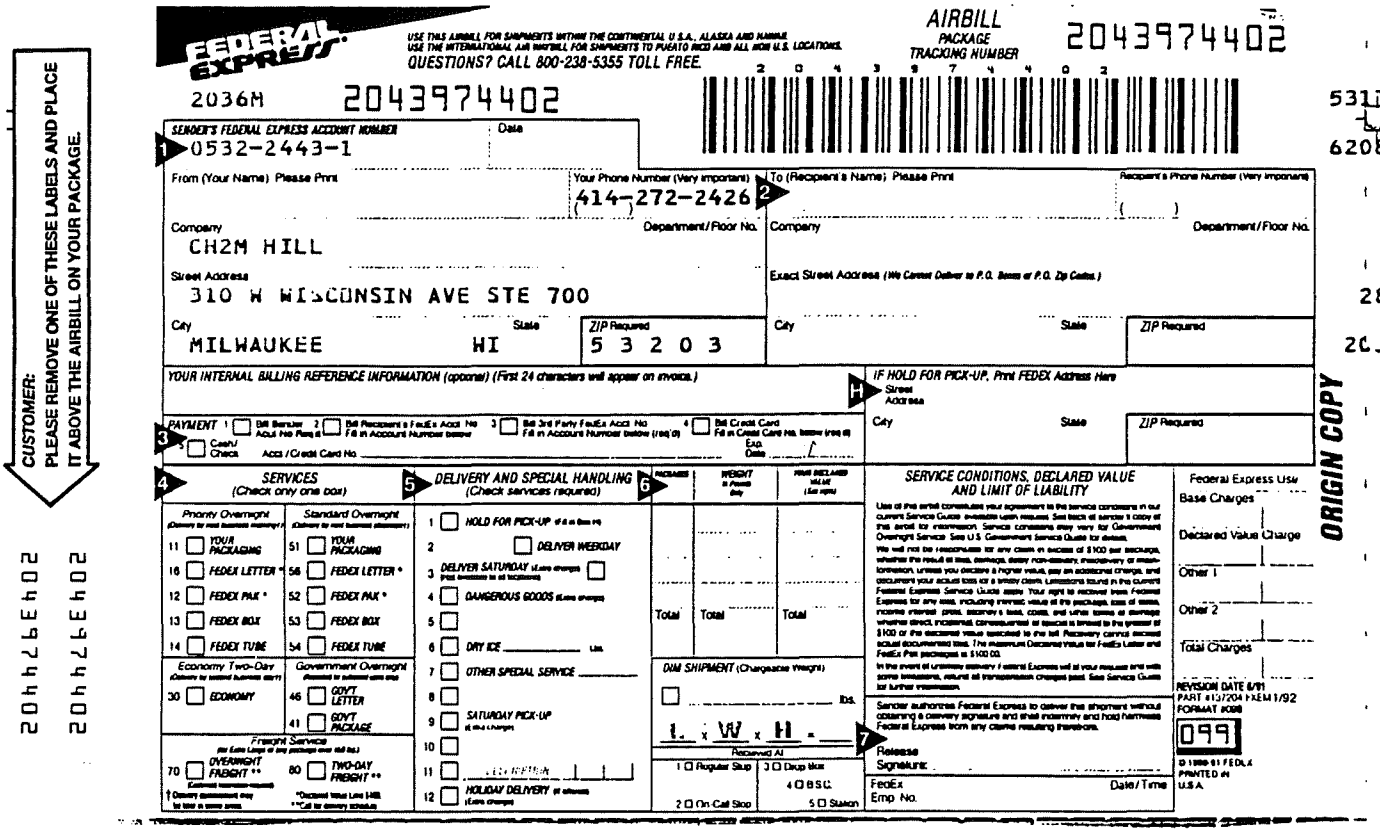

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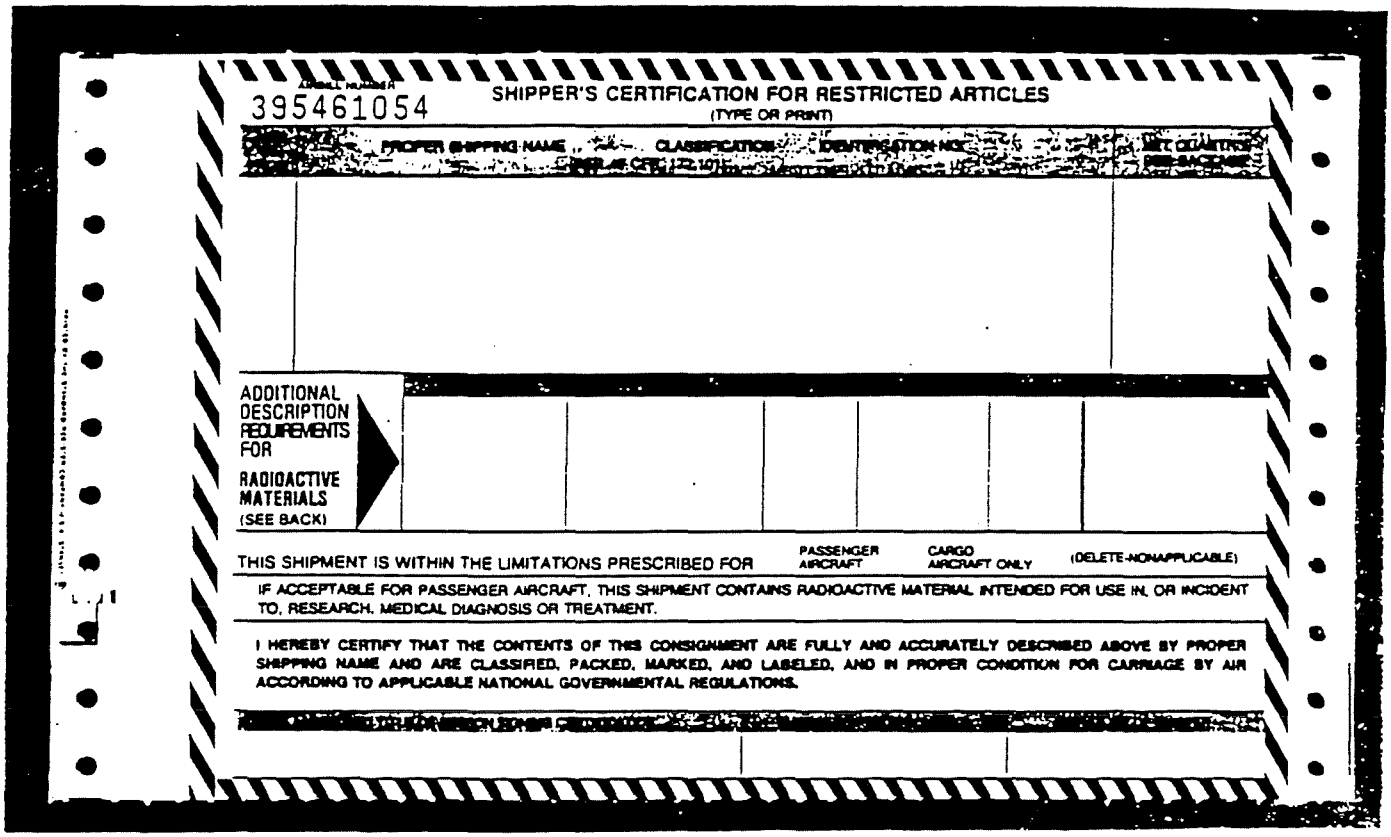
FIGURE 11



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 12

APPENDIX E
BIOASSAY METHODS

Environmental Toxicology

Standard Operating Procedures and Quality Assurance/Quality Control Manual for Wisconsin

Prepared by

CH2M HILL

June 1993

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1.0 Introduction

1.1 CH2M HILL Milwaukee Bioassay Laboratory

CH2M HILL operates three environmental toxicology laboratories designed for specific use in NPDES biomonitoring, toxicity identification/reduction evaluations (TI/RE), and other aquatic and terrestrial toxicology studies.

The address and other information for the Milwaukee laboratory is:

CH2M HILL Bioassay Laboratory
15779 West Ryerson Road
New Berlin, WI 53151

Phone: (414) 784-0448
Fax: (414) 784-0353

Contact: Laboratory Manager, Jim Stark

The Milwaukee laboratory, which began operating in January 1990, is a 6,0007-square-foot state-of-the-art facility designed to conduct bioassays and TI/REs. Figure 1 is the floor plan of the laboratory. Some of the main features of the laboratory include:

- A privately-owned well providing the source of all the laboratory water (i.e., no city or chlorinated water)
- Two 8′ × 10′ walk-in temperature-controlled test incubators
- Two separate temperature-controlled bioassay laboratories for acute and chronic toxicity testing
- In-house culturing of all organisms routinely tested
- Two temperature-controlled culture rooms, separate from the test laboratories
- Separate cleaning, chemical analysis, and treatability laboratories
- An 8′ × 8′ walk-in coldroom for sample storage
- Electronic temperature monitoring of 10 critical points throughout the laboratories

- All material and equipment in direct contact with culture or test media are of approved plastic or metal

- Communication system consisting of three phone lines, 24-hour voice mail, fax machine, computer/laser printer/modem, and copier

- Readily serviced by overnight couriers through Mitchell International Airport, Milwaukee

- Support staff of biologists, chemists, hydrologists, and engineers provided by CH2M HILL's Milwaukee office; additional bioassay personnel/ toxicologists provided by CH2M HILL's two other bioassay laboratories

1.2 Standard Operating Procedures Manual

This manual provides an overview of the standard operating procedures (SOP) and the quality assurance/quality control (QC/QC) policies for all projects and data produced in CH2M HILL's Milwaukee Bioassay Laboratory. It describes the personnel, sampling and handling procedures, standard test methods, test organism culturing, equipment calibration and maintenance, recordkeeping, data analysis, data management, and QA/QC procedures used as part of this testing program. The more detailed SOP manual [1], which also addressed health and safety information and training, is used at the laboratory. It may be reviewed at CH2M HILL's Milwaukee laboratory.

The test methods and QA/QC procedures were developed from the most recent U.S. EPA-approved methodologies. The procedures outlined in this document are followed for all NPDES compliance aquatic toxicity testing conducted for the State of Wisconsin unless there are specific state requirements or client changes. If specific permit language differs from the standard state requirements, procedural modifications will reflect these permit specifications.

2.0 Laboratory Management Organization

2.1 Laboratory Manager

A CH2M HILL laboratory manager is a scientist or professional of adequate education, training, experience, or a combination thereof who is assigned to a project by an office or division manager [2]. The laboratory manager is responsible for the initiation, technical conduct, and completion of a study. His or her responsibilities are to ensure that:

1. Bioassay protocols used, including any changes, are approved by the U.S. EPA and other authorities under whose jurisdiction they may fall and they are followed during the study.

2. Data, including unanticipated phenomena, are accurately recorded and verified.

3. Unexpected circumstances that may affect the quality or integrity of the study are corrected and duly documented.

4. Test systems conform to the protocol specifications.

5. Good laboratory practice regulations applicable to the project are followed.

6. Project data, protocols, documentation, and subsequent reports are transferred to the project file upon completion of the project.

2.2 Task Manager

A CH2M HILL task manager is a scientist or other professional with an adequate education, background, experience, or combination thereof who oversees a project and completes test procedures and documentation of data [2]. His or her responsibilities are to ensure that:

1. Protocols are followed.

2. Complications or problems that may involve a procedural change or decision for which he or she is ultimately responsible are corrected.

3. Technicians will be supervised and assisted in task completion.

4. Equipment and supplies necessary for the completion of a project are available and in working order.

5. The laboratory manager is kept informed of progress and the current status of the project.

6. Proper safety measures are taken and emergency equipment is present and in good working order.

7. Data, notebooks, records, and notes are properly filed at the end of the day.

8. Technicians employed during the project know specifically the tasks they are to perform.

2.3 Technician

A CH2M HILL technician is a person who has adequate education, background, experience, or combination thereof to perform a specific task or procedure assigned to him or her by a task or project manager [2]. Technicians must be fully capable of performing tasks and calling to the attention of the task manager any problems that arise. Technicians may express their opinion about a problem area, but must abide by the final decision or instruction of the task or laboratory manager. Technicians' responsibilities are:

1. To follow specified QA/QC and safety procedures in the laboratory.

2. To have a good knowledge of tasks and procedures they will be performing and be aware of the general policies of the laboratory.

3. To manage their own time in the laboratory and to report specific time spent on each project to the task or laboratory manager.

4. To report any problem encountered in the performance of a task to the task or laboratory manager.

5. To take responsibility for the general cleanup of their work area and maintain the equipment and instruments used.

2.4 Quality Assurance Manager

The quality assurance manager is a person of adequate education, background, experience [2], or a combination thereof who is responsible for monitoring each project from inception to completion and ensures that facilities, protocols, equipment, personnel, methods, practices, records, and controls are in conformance with the procedures presented in CH2M HILL's SOP manual [1] and this QA/QC manual. This person is

separate from and independent of the personnel engaged in the execution of the study. The quality assurance manager's responsibilities are:

1. To maintain a copy of the master schedule log of studies conducted at the test facility indexed by client name and containing the test system, nature of the study, date the study was initiated, status of each study, substance tested, protocols used, task manager's name, and final report status.

2. To maintain copies of protocols pertaining to the studies for which the bioassay laboratory is responsible.

3. To periodically inspect ongoing studies and maintain written and signed records of inspections complete with pertinent information about the study, personnel, findings, problems, and action taken to correct problems.

4. To periodically submit to the laboratory manager and CH2M HILL management written status reports noting specific problems and actions taken.

5. To ensure that no deviations are made from approved protocols without proper documentation and authorization.

6. To review final reports to ensure that procedures and practices are accurately described in the methods and that the results accurately reflect the raw data.

7. To prepare and sign a statement when audits are performed to be included in the final report that specifies when inspections were made and the findings reported to management.

8. To ensure that data records are kept in one location at the test facility.

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3.0 Control/Dilution Water

Three types of water may be used as the internal control or test dilution water: laboratory culture water, prepared dilute mineral water (DMW), or reconstituted laboratory water. Laboratory culture water, which is from the laboratory's private well, is pretreated with a reverse osmosis (RO) system and mixed with well water to provide moderately hard (100–150 mg/L $CaCO_3$) water. This product water is treated with Culligan granular activated carbon filters before laboratory use. Logbooks are maintained to document water quality for each type of water (Figures 2 and 3).

General water quality data recorded include date prepared (synthetic media only), technician, total hardness, total alkalinity, pH, dissolved oxygen, total iron, and conductivity. Each prepared media batch is assigned an identification number to track its usage during culturing or testing.

Specifics on dilution water used depend on the species tested, regulatory requirements for NPDES biomonitoring, or case-by-case requirements.

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4.0 Sample Handling Procedures

4.1 Sampling Procedures

Sample collection procedures are normally dictated by the NPDES permit and are generally the responsibility of the permit holder. If not, the procedures followed are those outlined by the EPA [3,4] unless state or other local agency guidelines require otherwise. The type of sample collected (grab or 24-hour composite) is confirmed with the appropriate regulatory agency before initiating any sample collection for situations not dictated by the NPDES permit.

Sample collection is performed at the designated NPDES sampling point unless specific tests require otherwise (e.g., nonchlorinated effluent). Samples are collected at the subsurface using either glass or polyethylene sample bottles. Composite samples are iced during shipment to maintain a preferred temperature of 4°C and a maximum of 10°C upon arrival at the laboratory. Sample holding times follow specific permit requirements and are typically 36 to 48 hours for effluents and a maximum holding time of 72 hours for all samples before the initiation of the test.

4.2 Chain-of-Custody Document

Chemical and effluent samples are required to have a chain-of-custody sheet to document and ensure proper handling. These records include date and time of sampling, technicians performing the sampling, any sample characteristics measured in the field (DO, pH, salinity/conductivity, temperature, etc.), and any other relevant information needed. These forms then become part of the permanent record of the project file and serve as a future reference for sample documentation (Figure 4).

4.3 Sample Logbook

Samples received by the laboratory are entered into a bound sample logbook (Figure 5). Each sample is given its own ID number to facilitate identification and use during testing and sample storage. The logbook includes the sample receiving date, client, project number, sample ID number, description of sample, the technician responsible for sample receiving, and any other pertinent information. The log provides a permanent record of the number of samples tested in the laboratory.

4.4 Sample Handling

Upon sample receipt, physicochemical parameters are measured and recorded (Figure 6). Samples to be used in a *Ceriodaphnia* or larval fathead minnow test are sieved

(approximately 60-micron mesh screen) into labeled containers. When appropriate, sample aliquots are homogenized before testing. Samples not used immediately are refrigerated (4°C).

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5.0 Test Organism Quality Control

5.1 Test Organisms Cultured

CH2M HILL's Milwaukee laboratory cultures the following organisms for toxicity testing:

- *Pimephales promelas* (fathead minnow)
- *Ceriodaphnia dubia* (water flea)
- *Daphnia magna* (water flea)
- *Daphnia pulex* (water flea)
- *Selenastrum capricornutum* (green algae)

Laboratory cultures of these organisms are managed to ensure only the highest quality organisms are used for testing. Other organisms, including *Selenastrum capricornutum*, are cultured at this laboratory, but are not used for Wisconsin's NPDES compliance biomonitoring. Periodically new batches of organisms are obtained from other sources (e.g., U.S. EPA and other CH2M HILL laboratories). Information about these organisms are entered in a logbook (Figure 7) for future reference.

5.2 Test Organism Isolation/Monitoring Log

Test organism isolation and monitoring logbooks are maintained to document the handling of each test species used for bioassays (Figures 8 through 11). Information includes date, species, age, lot number, and any other pertinent information. Each group of test organisms is assigned an identification (lot) number to facilitate tracking of each group through culturing and testing.

5.3 Reference Toxicant Testing

Reference toxicant tests are performed to document the sensitivity of organisms tested. Large amounts of toxicity data available for specific chemicals such as sodium chloride enable CH2M HILL periodically to compare the sensitivity of organisms tested to the expected sensitivity for that species. Reference toxicant tests are conducted on in-house cultures as well as on each new group of organisms obtained from commercial sources. Separate records for reference toxicant tests are maintained and become part of the permanent QA file (Figures 12 through 19). Computer updates/summaries of reference toxicant tests are made routinely (monthly) on each species tested. The files contain test dates, results of statistical analyses, and cumulative averages and standard deviations for the data collected as recommended by the U.S. EPA.

5.4 Performance Criteria

An internal laboratory control water is concurrently tested with every species/bioassay conducted. The following performance criteria are established for test acceptability.

Acute Bioassay

Acute toxicity criteria apply to tests using *Daphnia magna, Daphnia pulex, Ceriodaphnia dubia*, or juvenile fathead minnows *(Pimephales promelas)*. Control organism mortality in acute tests must not exceed 10 percent at the end of the 48- or 96-hour exposure period.

Chronic Bioassay

Chronic toxicity criteria apply to tests using *Ceriodaphnia*, larval fathead minnows, and *Selenastrum capricornutum*. Control organism mortality in the animal tests must not exceed 20 percent at the end of the 7-day exposure period.

Ceriodaphnia

Neonates used to initiate a test must be obtained from a brood of at least 8 offspring. During a chronic test, control organisms must produce at least 15 young per surviving female with at least 60 percent having 3 broods during a nominal 7-day test period.

Larval Fathead Minnow

Control organisms must attain a minimum individual average dry weight of 0.25 mg after a 7-day test period.

Selenastrum

The mean growth of the controls must be a minimum of 2×10^5 cells/mL and their variability should not exceed 20 percent.

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6.0 Test Organism Culture Procedures

CH2M HILL maintains cultures of *Ceriodaphnia dubia, Daphnia magna, Daphnia pulex, Pimephales promelas*, and *Selenastrum capricornutum* for use as test organisms in its biomonitoring program. (The algae culture procedures are provided, but are not required by Wisconsin for testing.) As part of CH2M HILL's quality assurance/quality control program, these guidelines were established to ensure only healthy, high quality organisms are used for bioassay testing purposes. Logbook pages maintained for these organisms are shown in Figures 20 through 42. Detailed culturing procedures for each species are as follows:

6.1 *Ceriodaphnia dubia*

General Maintenance

Ceriodaphnia dubia are cultured in laboratory fish tank-conditioned water or synthetic water (e.g., dilute mineral water). *Ceriodaphnia dubia* are cultured individually and in batches. Individual *Ceriodaphnia* are cultured in 1-ounce cups for at least 7 days. Survival and reproduction are monitored and water changed on Monday, Wednesday, and Friday. Organisms from these cultures are used for chronic or acute tests. Batch cultures are maintained in 1-liter beakers with weekly medium renewal. The batch cultures are monitored for age, general survival, and reproduction. The batch cultures are typically used for acute testing.

Feeding

Ceriodaphnia cultures are fed a mixture of dried yeast, cereal leaves, and trout (salmon) food (YCT) according to U.S. EPA methods [4]. Concentrated green algae *(Selenastrum capricornutum)* are prepared weekly according to EPA procedures and are also fed to *Ceriodaphnia*. New YCT food batches are prepared about once a month. Aliquots are frozen and thawed portions are used for no more than 1 week. New food batches are tested (solids analysis/adjustment and reproduction effects) before general use.

Individual cultures are fed daily and batch cultures are fed every Monday, Wednesday, and Friday. The feeding rate for individual cultures is 0.1 mL each of prepared YCT food and algae per cup. Batch cultures receive 5 mL YCT per beaker per feeding and 1.5 mL algae on the medium renewal day.

6.2 *Daphnia magna* and *Daphnia pulex*

General Maintenance

Daphnia spp. are cultured with laboratory fish tank-conditioned water. Batch cultures are maintained for each species in 1- to 5-liter containers with weekly medium renewal. The batch cultures are monitored for age, survival, reproduction, and general condition. ''Wild stock'' populations are also maintained in 10-gallon aquaria (part of the fish tank-conditioned culture water system).

Feeding

Daphnia spp. cultures are fed with a mixture of trout (salmon) food, cereal leaves, and dried yeast as described in the U.S. EPA methods [3].

New food batches are prepared about once a month. Aliquots are frozen and thawed portions are used for a maximum of 1 week.

Feeding is performed on an every other day basis (Monday, Wednesday, Friday) at a rate of 1.5 mL per liter. On media renewal days, 2.0 mL of concentrated algae per liter are also fed.

6.3 *Pimephales promelas*

Fathead minnow culturing consists of adult breeders, embryos, larvae and juveniles, and future brood stock. Culture procedures follow EPA recommendations and use laboratory well/RO blended water [5].

Breeders

Four pairs of breeders per 15-gallon aquarium are maintained in a 16-hour-light/8-hour-dark photoperiod with a continuous flow of 25°C laboratory water. Egg production and general health are monitored daily. Frozen brine shrimp are fed twice daily. Tanks are cleaned at least weekly.

Embryos

Embryos attached to spawning substrates are incubated in hatching pans at 25°C under continuous water flow. Daily inspections are made for development and general health. Upon hatching, larvae are pooled either for chronic testing or stocking of culture tanks.

Juveniles

The juveniles are maintained in 15-gallon aquaria at a density of 250 fish per tank for about 1 month. The temperature is regulated at 20°C under continuous water flow. The photoperiod is 16 hours light/8 hours dark. The juveniles are fed live brine shrimp nauplii twice daily. Waste and excess food are siphoned daily and tank sides are cleaned weekly. Aquaria and components are thoroughly cleaned at the time when fish are either removed for tests or discarded.

6.4 *Selenastrum capricornutum*

Green algae cultures are maintained at 25°C in an incubator under continuous light. Replicates of 500 mL in 1-liter Erlenmeyer flasks are continuously agitated (100 cpm) on a shaker table. The culture medium is prepared according to EPA recommendations (Table 6.1). The water used for culture media is Type I reagent grade, produced through RO and carbon and deionization filtration.

Each week 0.45 μm filtered media is inoculated with an axenic *Selenastrum capricornutum* culture. These cultures are renewed weekly and maintained for 2 weeks. Each week algae are concentrated by centrifugation and used as food for daphnids.

About two or three times a year, a new algal slant is obtained from an outside source to maintain a pure strain. Glassware sterilization (by autoclave) and aseptic techniques are used for the axenic cultures.

Weekly growth (cell density) is determined using hemacytometer. Algal production and media chemical parameters are recorded in log books.

6.5 Test Organism Isolation

All isolation/pre-test monitoring of the organisms is recorded in appropriate logbooks for age documentation (Figures 8 through 11).

Daphnia spp. and *Ceriodaphnia* Batch Cultures

Gravid adult *Daphnia* or *Ceriodaphnia* from batch cultures are isolated in the culture chambers prepared with filtered media (i.e., sieved to remove young organisms) no more than 24 hours before test initiation. Neonates (less than 24 hours old) produced from these adults are used in the acute toxicity tests.

Table 6.1
Nutrient Stock Solutions for Maintaining
Algal Stock Cultures and Test Control Cultures

Nutrient Stock Solution	Compound	Amount dissolved in 500 mL Reagent Water
1	$MgCl_2 \cdot 6H_2O$	6.08 g
	$CaCl_2 \cdot 2H_2O$	2.20 g
	H_3BO_3	92.8 mg
	$MnCl_2 \cdot 4H_2O$	208.0 mg
	$ZnCl_2$	1.64 mg[a]
	$FeCl_3 \cdot 6H_2O$	79.9 mg
	$CoCl_2 \cdot 6H_2O$	0.714 mg[b]
	$Na_2MoO_4 \cdot 2H_2O$	3.63 mg[c]
	$CuCl_2 \cdot 2H_2O$	0.006 mg[d]
	$Na_2EDTA \cdot 2H_2O$	150.0 mg
2	$NaNO_3$	12.75 g
3	$MgSO_4 \cdot 7H_2O$	7.35 g
4	K_2HPO_4	0.522 g
5	$NaHCO_3$	7.50 g

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[a] $ZnCl_2$—Weigh out 164 mg and dilute to 100 mL. Add 1 mL of this solution to Stock No. 1.

[b] $CoCl_2 \cdot 6H_2O$—Weigh out 71.4 mg and dilute to 100 mL. Add 1 mL of this solution to Stock No. 1.

[c] $Na_2MoO_4 \cdot 2H_2O$—Weigh out 36.6 mg and dilute to 10 mL. Add 1 mL of this solution to Stock No. 1.

[d] $CuCl_2 \cdot 2H_2O$—Weigh out 60.0 mg and dilute to 1,000 mL. Take 1 mL of this solution and dilute to 10 mL. Add 1 mL of the second dilution and add to Stock No. 1.

Juvenile Fathead Minnows

Aquaria containing appropriately aged fish (from the same lot or within 1 day age difference) are designated for acute bioassay usage. Abnormally large or small size fish are rejected before pooling them for test use.

Ceriodaphnia dubia Individual Cultures

Gravid adult *Ceriodaphnia* (6 to 9 days old) are intensively monitored 24 hours before test initiation in their culture containers. For chronic tests, reproduction is recorded or neonates are isolated about every 4 hours to ensure all are within the prescribed age limitations (<24 hours old and within 8 hours age difference). Neonates used for chronic testing are typically from the third or fourth brood and have a minimum brood size of eight. Neonates less than 24 hours old may also be used in acute tests.

Larval Fathead Minnows

Fathead minnow embryos near their hatching stage are intensively monitored or isolated (in a separate container) to ensure newly hatched fish for chronic testing are less than 24 hours old.

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7.0 Standard Operating Procedures for Bioassay Testing

CH2M HILL performs aquatic toxicity testing according to the most recent EPA-approved procedures, and ensures that all methods used conform to the standards of any state or local agency under whose jurisdiction the testing may fall. In this way, CH2M HILL can ensure that data produced from these tests will be acceptable according to the legal guidelines that govern biomonitoring required by NPDES or state permits.

The procedures described were adopted from EPA guidelines and state-specific regulations. They are followed for all testing unless the investigations are noncompliance and specific client needs require otherwise.

7.1 Procedure for Static Acute Testing of Freshwater Species

The procedure described below is adapted from EPA methods [3]. Tables 7.1 through 7.3 summarize the standard test conditions used by CH2M HILL for the common test species required. Specific state or client requirements may necessitate minor changes, which are performed and documented as necessary. Testing is performed in incubators.

Effluent Sampling

Section 4.0 describes the general sampling and documentation procedures followed for effluent collection. The maximum holding time for any sample used for acute testing is 72 hours, unless specified differently in the NPDES permit.

Test Organisms

All organisms tested are cultured at CH2M HILL's Milwaukee laboratory. Organisms used for testing are pooled and randomly selected for addition to test chambers. Fish weights may be determined for appropriate loading rates.

Control and Dilution Water

The internal control water is the laboratory's appropriate culture medium. Bioassay dilution water may be receiving water or appropriate laboratory culture medium, depending on specific permit or test requirements.

Table 7.1
SUMMARY OF TEST CONDITIONS FOR THE
CERIODAPHNIA ACUTE BIOASSAY

1.	Test organism	*Ceriodaphnia dubia* (Crustacea: Cladocera).
2.	Test type	Static renewal.
3.	Age of test organisms	Less than 24 hours.
4.	Test chamber size	30 mL
5.	Test solution volume	25 mL
6.	Renewal of test solutions	Daily.
7.	Number of replicate chambers per treatment	4
8.	Number of test organisms per chamber	5
9.	Internal control	Laboratory culture medium.
10.	Control/dilution water	Receiving water or laboratory culture medium.
11.	Effluent concentrations	Permit dependent.
12.	Temperature	20 ± 1°C
13.	Feeding regime	None.
14.	Aeration	Initially, when DO < 6.0 mg/L.
15.	Test duration	48 hours.
16.	Sampling scheme	Usually one 24-hour composite effluent sample. Maximum holding time of 72 hours between completion of collection and initial test use for each sample. One grab sample of receiving water collected within 72 hours of test initation. Laboratory water used, collected daily. (Some permits may specify 36-hour holding times.)
17.	Effects measured	Survival.
18.	Test acceptability	90% or greater mean survival in the laboratory control.

Table 7.2
SUMMARY OF TEST CONDITIONS FOR THE
DAPHNIA ACUTE BIOASSAY

1.	Test organism	*Daphnia magna* or *Daphnia pulex* (Crustacea: Cladocera).
2.	Test type	Static renewal.
3.	Age of test organisms	Less than 24 hours.
4.	Test chamber size	30 mL
5.	Test solution volume	25 mL
6.	Renewal of test solutions	Daily.
7.	Number of replicate chambers per treatment	4
8.	Number of test organisms per chamber	5
9.	Internal control	Laboratory culture medium.
10.	Control/dilution water	Receiving water or laboratory culture medium.
11.	Effluent concentrations	Permit dependent.
12.	Temperature	20 ± 1°C
13.	Feeding regime	None.
14.	Aeration	Initially, when DO < 6.0 mg/L.
15.	Test duration	48 hours.
16.	Sampling scheme	Usually one 24-hour composite effluent sample. Maximum holding time of 72 hours between completion of collection and initial test use for each sample. One grab sample of receiving water collected within 72 hours of test initiation. Laboratory water used, collected daily. (Some permits may specify 36-hour holding times.)
17.	Effects measured	Survival.
18.	Test acceptability	90% or greater mean survival in the laboratory control.

Table 7.3
SUMMARY OF TEST CONDITIONS FOR THE
FATHEAD MINNOW ACUTE BIOASSAY

1.	Test organism	*Pimephales promelas* (Osteichthyes: Cyprinidae).
2.	Test type	Static renewal.
3.	Age of test organisms	20 to 40 days old.
4.	Test chamber size	500 mL (minimum)
5.	Test solution volume	200 mL (minimum)
6.	Renewal of test solutions	Daily.
7.	Number of replicate chambers per treatment	2
8.	Number of test organisms per chamber	10
9.	Internal control	Laboratory culture medium.
10.	Control/dilution water	Receiving water or laboratory culture medium.
11.	Effluent concentration	Permit dependent.
12.	Temperature	20 ± 1°C
13.	Feeding regime	None.
14.	Aeration	None, unless DO concentration falls below 40% saturation (then, continuous at rate not exceeding 100 bubbles/minute).
15.	Test duration	96 hours.
16.	Loading rate	Less than 0.8 g/L.
17.	Sampling scheme	Usually two separate 24-hour composite effluent samples, each used for a 48-hour exposure period. Maximum holding time of 72 hours between completion of collection and initial test use for each sample. One grab sample of receiving water collected within 72 hours of test initiation. Laboratory water used, collected daily. (Some permits may specify 36-hour holding times.)
18.	Effects measured	Survival.
19.	Test acceptability	90% or greater mean survival in the laboratory control.

Test Start and Duration

Depending upon regulatory agency requirements (specified in the permit), tests are usually initiated within 36 to 48 hours of sample collection. CH2M HILL's routine fish acute tests are monitored for 96 hours; water flea acute tests are monitored for 48 hours.

Test Solutions Preparation

Appropriate sample volumes are added to beakers for temperature (20° ± 1°C) adjustment in a hot water bath. After proper temperature and DO are achieved, exposure concentrations are prepared (if applicable) using designated graduated cylinders. Depending upon regulatory agency requirements or test specifications, either receiving water or laboratory water is used for dilution of the effluent. A serial dilution including 100 percent effluent is usually prepared for LC_{50} tests. Depending upon test specifications, single or other concentrations may be tested for pass/fail criteria. Specific test solution volume requirements are determined and posted in tabular form to facilitate consistent preparation of treatment renewals.

Chemical Determinations

Chemical parameters measured initially and daily thereafter on all treatment renewal solutions (with surviving organisms) include DO, pH, and conductivity. Parameters measured daily on the 24-hour old (final) solutions are DO and pH.

Total alkalinity, hardness, total residual chlorine, and ammonia are determined in each new sample (100 percent effluent or the highest concentration tested, and receiving water). Total alkalinity and hardness are measured once in the laboratory control. Alkalinity is usually measured immediately, but samples may be refrigerated up to 7 days before titration. If hardness is not analyzed upon sample receipt, a labeled aliquot is appropriately preserved and refrigerated. Sample hardness is determined within 1 week. Total residual chlorine and ammonia are measured on receipt of each new sample.

The chemical monitoring is recorded in logbooks and on data forms (Figures 43 through 55).

Temperature Monitoring

The temperature of the test incubator is electronically monitored hourly by a data logger and thermocouple. Minimum, maximum, and average temperatures are calculated daily and recorded in a temperature logbook (Figures 54 and 55) and on the bioassay temperature data form (Figure 56).

Organism Monitoring

The number of live and dead organisms are counted and recorded daily on the test data sheet along with other comments (see Figures 57 through 61). Mortality/immobility is defined as inability to maintain position in the water column for 5 seconds after stimulation.

Data Analysis

When appropriate, survival data are analyzed using a modified EPA computer program that uses Probit, Moving Average, trimmed Spearman Karber, and binomial analyses to generate an LC_{50} value and 95-percent confidence limits. In some cases, pass/fail criteria may be used to analyze test data.

Report

A formal report is typically issued within 2 weeks of test termination. It may be prepared by the Task Manager or Laboratory Manager. The report contains:

- Chain-of-custody forms, which provide specific client and sample information
- A summary of test conditions
- Any deviations from the protocols or test conditions
- A summary table of survival data
- Photocopies of original bench sheets on survival and chemical parameters
- Task Manager's name
- Results of data analyses
- Information on test validity

Before the report is issued, it is reviewed by at least two staff personnel, including the Laboratory Manager, or designee, (Figure 62).

Test Validation

Part of CH2M HILL's laboratory QA/QC plan is to conduct concurrent internal laboratory control tests using the same methods, techniques, and organisms from the same pool. Test validity is defined as a minimum of 90-percent survival in the laboratory control (culture medium) treatment.

7.2 Procedure for Larval Fathead Minnow Chronic Survival and Growth Test

The procedure described below was adapted from EPA methods [4]. Table 7.4 summarizes the standard test conditions used by CH2M HILL. Specific state or client requirements may necessitate minor changes, which are performed and documented as necessary. Testing is performed in incubators.

Effluent Sampling

Section 4.0 describes the general sampling and documentation procedures followed for effluent collection.

Twenty-four-hour composite effluent samples are used to initiate and perform daily renewals during the 7-day test period. Composite samples are usually collected three times over the test period and used to renew test solutions daily. Each sample is used for a minimum of 48 hours, and the maximum holding time for each composite sample is 72 hours. (Some permits may specify a maximum 36-hour holding time.)

Test Organisms

Larval fathead minnows (< 24 hours old), cultured at CH2M HILL's laboratory are used in chronic bioassays.

Test Start and Duration

Depending upon regulatory criteria, tests are usually initiated within 36 to 48 hours of sample collection. All larval fish chronic bioassays are monitored for 7 days.

Control and Dilution Water

The internal control water used in the test is CH2M HILL's laboratory water (fathead minnow culture medium) obtained from its private well. Daily aliquots for test use are collected from the temperature regulated culture headbox. The bioassay dilution water may be receiving water or laboratory water, depending on specific permit or test requirements.

Test Solutions Preparation

Required volumes of laboratory control water, receiving water, and effluent are added to beakers and placed in a hot water bath for temperature adjustment. DO is measured after proper temperature is achieved (25° ± 1°C). Aeration may be necessary to provide the appropriate DO level. Samples that are aerated are noted on the chemical data sheets. Exposure concentrations are prepared using designated graduated cylinders. Depending on the test requirements, receiving water or laboratory media may be used for dilution of

Table 7.4
SUMMARY OF TEST CONDITIONS FOR THE
FATHEAD MINNOW CHRONIC BIOASSAY

1.	Test organism	*Pimephales promelas* (Osteichthyes: Cyprinidae).
2.	Test type	Static renewal.
3.	Age of test organisms	Larval, less than 24 hours.
4.	Test chamber size	500 mL (minimum)
5.	Test solution volume	250 mL (minimum)
6.	Renewal of test solutions	Daily
7.	Number of replicate chambers per treatment	4
8.	Number of test organisms per chamber	10
9.	Internal control	Laboratory culture medium.
10.	Control/dilution water	Receiving water or laboratory culture medium.
11.	Effluent concentrations	Permit dependent.
12.	Temperature	25 ± 1°C
13.	Feeding regime	0.15 mL brine shrimp nauplii (less than 24-hours old) twice daily.
14.	Aeration	None, unless DO concentration falls below 40% saturation (then, continuous at rate not exceeding 100 bubbles/min).
15.	Cleaning	Siphon daily, immediately before test solution renewal.
16.	Test duration	7 days.
17.	Sampling scheme	Three 24-hour composite effluent samples, each used for a minimum of 48 consecutive exposure hours. Maximum holding time of 72 hours between completion of collection and initial test use of each sample. One grab sample of receiving water collected within 72 hours of test initiation. Laboratory water used, collected daily. (Some permits may specify 36-hour sample holding times.)
18.	Effects measured	Survival and growth (dry weight).
19.	Test acceptability	Laboratory control with 80% or greater mean survival and surviving fish with at least 0.25 mg average dry weight.

the effluent. A serial dilution surrounding and including the designated instream wastewater concentration (IWC) is typically prepared for chronic bioassays. Other dilutions or single concentrations may be used if specified. Specific test solution volume requirements are determined and posted in tabular form to facilitate consistent preparation of treatment renewals.

Chemical Determinations

Routine chemical parameters measured initially and daily thereafter on all treatment renewal solutions (with surviving fish) include DO, pH, and conductivity. Parameters measured daily on the 24-hour-old (final) solutions are DO and pH.

Total alkalinity, hardness, total residual chlorine, and ammonia are determined in each new sample (100 percent effluent or highest concentration tested, and receiving water). Total alkalinity and hardness are measured once in the laboratory control. Alkalinity is usually measured immediately, but samples may be refrigerated up to 7 days before titration. If hardness is not analyzed upon sample receipt, a labeled aliquot is appropriately preserved and refrigerated. Sample hardness is determined within 1 week. Total residual chlorine and ammonia are measured on receipt of each new sample.

The chemical monitoring is recorded on data forms (Figures 63 and 64).

Temperature Monitoring

The temperature of the test incubator is electronically monitored hourly by a data logger and thermocouple. Minimum, maximum, and average temperatures are calculated daily and recorded in a temperature logbook (Figures 54 and 55) and on the bioassay temperature data sheet (Figure 56).

Test Chamber Specifics

Test chambers are typically disposable plastic containers. Depending upon test specifics, the test solution volume may range from 250 to 500 mL per chamber. Four replicates totaling up to 2,000 mL of test solution per treatment are used.

Acquisition of Test Organisms

At least 24 hours before a scheduled bioassay, the development of fish embryos in a hatching pan is checked. Any larvae present are removed from the pan, or at least three tiles (with appropriate number of eggs) assigned for a specific test are transferred to a new pan of water. Isolation time and other information are recorded on the Larval Fish Test Isolation Log. When sufficient larvae are present, the required number plus a 10- to 25-percent surplus are carefully transferred to 500 or 1,000 mL of culture water in a 2-liter beaker (referred to as a pool). Transfer is accomplished using a fine-mesh culture net or siphon.

Test Initiation

Transfer tubes are used to randomly introduce 10 larvae from the pool to each test chamber. Test chambers are labeled and placed in racks arranged on a shelf in the incubator according to a chart of randomly determined positions. Bioassay start time is recorded.

Aeration

Treatments that require aeration for maintenance of 40-percent dissolved oxygen receive continuous aeration through disposable glass pipets at a rate of $\leq$ 100 bubbles/minute. Air lines or pipets are appropriately labeled when different treatments are aerated to prevent contamination. Comments on aeration are recorded on the chemical data sheets.

Feeding

Each test chamber receives a 0.15 mL suspension of concentrated live brine shrimp nauplii (< 24 hours old) twice daily at intervals of about 6 hours, at the beginning and end of a workday.

Test Solution Renewal and Chamber Cleaning

Chambers are cleaned and treatment solutions renewed daily within 2 hours of test initiation time. Dead fish are discarded and recorded on the survival data sheet. Old solutions are removed, along with uneaten or dead brine shrimp, cysts, and other debris, to a depth of about 7 to 10 mm (75 to 100 mL) with siphon apparatus.

Observations During the Test

Fish in each test chamber are counted daily, and mortalities are recorded on the survival data sheet (Figure 65). A fish is considered dead if it is unable to maintain its position in the water column for 5 seconds after stimulation (gentle prod). Comments on other conditions (e.g., fish inadvertently siphoned) and fish behavior are also recorded.

Test Termination

Test termination occurs after 7 days of exposure $\pm$2 hours. The larval fish are removed and placed in tared aluminum weigh boats. Weigh boats are then transferred to a 100°C drying oven for a minimum of 2 hours. After drying, weigh boats are cooled in desiccators before being weighed to the nearest 0.01 mg. Weights recorded on the balance printer are transferred onto a fish growth data sheet (Figure 66).

Data Analysis

Analysis of the survival and growth data follow EPA recommendations [4]. Survival data are transformed using the arc sine square root method. When assumptions of normality and homogeneity of variance are met, the Dunnett's Test or Bonferroni t-test is used to determine significant difference. Otherwise, Steel's Many One Rank Test or Wilcoxon Rank Sum Test is used. Additionally, an IC_{25} (the concentration at which 25 percent inhibition occurs) is also calculated using the linear interpolation method [6] (modified EPA program).

Report

A formal report is typically issued within 2 weeks of test termination. It may be prepared by the Task Manager or Laboratory Manager. The report contains:

- Chain-of-custody forms, which provide specific client and sample information
- Any deviations from the protocols or test conditions
- A summary of test conditions
- A summary table of survival and growth data
- Photocopies of original bench sheets on survival, growth, and chemical parameters
- Task Manager's name
- Results of data analyses
- Information on test validity

Before the report is issued, it is reviewed by at least two staff personnel, including the Laboratory Manager, or designee, (Figure 62).

Test Validation

Part of CH2M HILL's laboratory QA/QC plan is to conduct concurrent internal laboratory control tests using the same methods, techniques, and organisms from the same pool. Test validity is defined as a minimum of 80 percent survival in the laboratory control (culture medium) over 7 days and an average dry weight of surviving control fish $\geq$ 0.25 mg.

7.3 Procedure for *Ceriodaphnia dubia* Chronic Survival and Reproduction Test

The procedure described below was adapted from EPA Methods [4]. Table 7.5 summarizes the standard test conditions used by CH2M HILL. Specific state or client requirements may necessitate minor changes, which are performed and documented as necessary. Testing is performed in incubators.

Effluent Sampling

Section 4.0 describes the general sampling and documentation procedures followed for effluent collection. Twenty-four hour composite effluent samples are used to initiate chronic testing and perform daily renewals during the nominal 7-day test period.

Composite samples are usually collected three times over the test period and used to renew test solutions daily. Each sample is used for a minimum of 48 hours, and the maximum holding time for each composite sample is 72 hours. (Some permits may specify a maximum holding time of 36 hours.)

Test Start and Duration

Depending upon regulatory agency requirements, tests are usually initiated within 36 to 48 hours of sample collection. Chronic *Ceriodaphnia* bioassays are monitored for 7 days, however, early termination at 6 days is acceptable when at least 60 percent of the controls have released three broods.

Test Organisms

Ceriodaphnia dubia are obtained from individual monitored brood racks cultured at CH2M HILL's Milwaukee Bioassay Laboratory. Neonates (< 24 hours old) within an 8-hour age difference are used to initiate *Ceriodaphnia* chronic tests. A minimum of eight offspring from a third or fourth brood of a healthy adult (monitored for 6 to 8 days) are preferred.

Control and Dilution Water

The internal control water used in the test is CH2M HILL's laboratory water (*Ceriodaphnia* culture media). Dilute (e.g., 20 percent) mineral water may be used. Single or daily grab samples are taken for test purposes. The bioassay dilution water may be receiving water or laboratory water, depending on specific permit or test requirements.

Table 7.5
SUMMARY OF TEST CONDITIONS FOR THE
CERIODAPHNIA CHRONIC BIOASSAY

1.	Test organism	*Ceriodaphnia dubia* (Crustacea: Cladocera).
2.	Test type	Static renewal.
3.	Age of test organisms	Less than 24 hours, all released within an 8-hour period (same generation from even-aged parents).
4.	Test chamber size	30 mL
5.	Test solution volume	15 mL
6.	Renewal of test solutions	Daily.
7.	Number of replicate chambers per treatment	10
8.	Number of test organisms per chamber	1
9.	Internal control	Laboratory culture medium.
10.	Control/dilution water	Receiving water or laboratory culture medium.
11.	Effluent concentrations	Permit dependent.
12.	Temperature	25 ± 1°C
13.	Feeding regime	0.1 mL of YCT culture food and 0.1 mL algae per test chamber daily.
14.	Aeration	None.
15.	Test duration	7 days (or when ≥ 60 percent of controls have three broods).
16.	Sampling scheme	Three 24-hour composite effluent samples, each used for a minimum of 48 consecutive exposure hours. Maximum holding time of 72 hours between completion of collection and initial test use of each sample. One grab sample of receiving water collected within 72 hours of test initiation. Laboratory water used, collected daily or prepared as one batch. (Some permits may specify a 36-hour sample holding time.)
17.	Effects measured	Survival and reproduction.
18.	Test acceptability	Laboratory control with 80% or greater mean survival, an average of 15 or more young per surviving female, and at least 60% producing 3 broods.

Acquisition of Test Organisms

Approximately 24 hours before test initiation, gravid adults are monitored at 2- to 4-hour intervals and observations are recorded on the monitoring form (Figure 9). On the day of the test, *Ceriodaphnia* cultures are examined again and monitoring may continue. When sufficient reproduction of similar-aged neonates (< 24 hours old, within 8-hour age difference) is estimated to have occurred, the required number of neonates are pooled. Test neonates are taken from at least three different adults.

Test Solutions Preparation

Required volumes of laboratory control, receiving water, and effluent are added to beakers for temperature adjustment (25° ± 1°C) in a hot water bath. After proper temperature and DO are achieved, exposure concentrations are prepared using designated graduated cylinders. Depending on test requirements or test specifications, either receiving water or laboratory water is used for dilution of the effluent. A serial dilution surrounding and including the IWC or other concentrations are prepared for chronic tests according to the work order. Specific test solution volume requirements are determined and posted in tabular form to facilitate consistent preparation of treatment renewals.

Chemical Determinations

Routine chemical parameters measured initially and daily thereafter on all treatment renewal solutions (with organism survival) include DO, pH, and conductivity. Parameters measured daily on the 24-hour old (final) solutions are DO and pH.

Total alkalinity, hardness, total residual chlorine, and ammonia are determined in each new sample (100 percent effluent or highest concentration tested, and receiving water). Total alkalinity and hardness are measured once in the laboratory control. Alkalinity is usually measured immediately, but samples may be refrigerated up to 7 days before titration. If hardness is not analyzed upon sample receipt, a labeled aliquot is appropriately preserved and refrigerated. Sample hardness is determined within 1 week. Total residual chlorine and ammonia are measured on receipt of each new sample.

The chemical monitoring is recorded on the data forms (Figures 63 and 64).

Temperature Monitoring

The temperature of the test incubator is electronically monitored hourly by a data logger and thermocouple. Minimum, maximum, and average temperatures are calculated daily and recorded in a temperature logbook (Figures 54 and 55) and on the bioassay temperature data form (Figure 56).

Test Chamber Specifics

Test chambers are 1-oz disposable plastic cups. The test solution volume is 15 mL per chamber. Ten replicates totaling 150 mL of test solution per treatment are used.

Test Initiation

The test chambers are labeled (as to treatment number) and arranged in designated rows on a tray. Test solutions of 15 mL per chamber are added with separate and labeled fill tubes. Labeled eyedroppers or disposable pipets are used to transfer the pooled *Ceriodaphnia* neonates into the test chambers. Neonates are captured with the aid of a dissection scope and slowly released beneath the water surface with the aid of a light box to avoid stress or injury. A single neonate is introduced into each of 10 replicate chambers per treatment for a total of 10 neonates per treatment.

Where different effluents or test materials are used, separate aliquots of the pooled *Ceriodaphnia* are employed to prevent contamination during organism addition. Test chambers are arranged in a labeled rack and placed in the 25°C test incubator. Bioassay start time is recorded on the data sheet.

Aeration

Test chambers are not aerated during the exposure period. Renewal solutions may be aerated before they are introduced into the test chambers for DO adjustment either to or near saturation.

Feeding

Each test chamber daily receives 0.1 mL of *Ceriodaphnia* culture food (YCT) and 0.1 mL of algae concentrate.

Test Solution Renewal

The test solutions are renewed daily within 2 hours of the test initiation time using newly collected or refrigerated samples. Initial chemical parameters are measured before samples are poured into the new set of test chambers. Original organisms are transferred to new test solutions and chambers with the aid of a dissection scope and light box. Final chemical measurements are made on a composite of several replicate chambers of each 24-hour-old treatment.

Observations During the Test

Conditions of the adults are observed during daily transfer and solution renewal. Adult survival, reproduction, and other observations are recorded daily on the data sheet

(Figure 67) for each chamber. Survival is defined as the ability to maintain position in the water column for 5 seconds after stimulation.

Test Termination

At test termination, adult survival and reproduction in each replicate are determined and recorded on the data sheet, and final DO and pH measurements are taken and recorded on the chemical data sheet.

Data Analysis

Survival data are analyzed by Fisher's Exact Test for significant difference. Effluent concentrations not statistically different from control in survival will be analyzed for statistical differences in reproduction using Dunnett's Test or Bonferroni t-test. If the assumptions of homogeneity of variance and normality cannot be met, Steel's Many One Rank Test or Wilcoxon Rank Sum Test is used to compare the reproduction data. Additionally, an IC_{25} (the concentration at which 25 percent inhibition occurs) is calculated using the linear interpolation method [6].

Report

A formal report is typically issued within 2 weeks of test termination. It may be prepared by the Task Manager or the Laboratory Manager. The report contains:

- Chain-of-custody forms, which provide specific client and sample information
- A summary of test conditions
- Any deviations from test protocols or test conditions
- A tabular summary of survival and reproduction data
- Photocopies of original bench sheets on survival, reproduction, and chemical parameters
- Task Manager's name
- Results of data analyses
- Information on test validity

Before the report is issued, it is reviewed by at least two staff personnel, including the Laboratory Manager, or designee, (Figure 62).

Test Validation

Part of CH2M HILL's laboratory QA/QC plan is to conduct concurrent internal laboratory control tests using the same methods, techniques, and organisms from the same pool. Test validity is defined as a minimum of 80 percent adult survival, a minimum average of 15 offspring per adult, and at least 60 percent of adults producing 3 broods in the laboratory control (culture medium) during a nominal 7-day test period.

7.4 Procedure for Green Algae, *Selenastrum capricornutum*, Growth Test

The procedure described below was adapted from EPA Methods [4]. Table 7.6 summarizes the standard test conditions used by CH2M HILL. Specific state or client requirements may necessitate minor changes, which are performed and documented as necessary. Testing is performed in incubators or temperature controlled rooms.

Effluent Sampling

Section 4.0 describes the general sampling and documentation procedures followed for effluent collection. One 24-hour composite effluent sample used for the test. The maximum holding time for the composite sample is 36 hours.

Test Start and Duration

Tests are initiated within 36 hours of sample collection. All chronic algal bioassays are monitored for 96 hours.

Test Organisms

Axenic cultures of *Selenastrum capricornutum* are maintained at CH2M HILL's Milwaukee Bioassay Laboratory. Cultures 4 to 7 days old are used to initiate algal bioassays.

Control and Dilution Water

The internal control water used is the algal culture medium. The dilution water may be the laboratory culture medium, depending upon permit specifications.

Inoculum Preparation

The inoculum is prepared less than 3 hours before the test initiation, using *Selenastrum* collected from a 4 to 7-day axenic culture. Inoculum cell density is adjusted to provide 10,000 cells/mL ($\pm$10 percent) in each test chamber.

Table 7.6
SUMMARY OF TEST CONDITIONS FOR THE
ALGAL *SELENASTRUM CAPRICORNUTUM* BIOASSAY

1. Test type	Static.
2. Temperature	25 ± 1°C
3. Light quality	"Cool white" fluorescent lighting.
4. Light intensity	400 ± 40 ft-c
5. Photoperiod	Continuous illumination.
6. Test chamber size	250 mL
7. Test solution volume	100 mL
8. Renewal of test solutions	None.
9. Age of test organisms	4 to 7 days.
10. Initial cell density in test chambers	10,000 cells/mL
11. No. replicate chambers/ concentration	3
12. Shaking rate	100 cpm continuous, or twice daily by hand.
13. Dilution water	Algal stock culture medium with or without EDTA.
14. Effluent concentrations	Permit dependent.
15. Test duration	96 hours
16. Endpoint	Growth (cell counts).
17. Test acceptability	2×10^5 cells/mL in the controls; variability of controls should not exceed 20 percent.

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Algal cells in an aliquot from the culture are counted using a hemacytometer. Cell density is determined and the solution volume is adjusted accordingly (1 mL inoculum per test chamber).

Test Solutions Preparation

The effluent is usually not filtered through a 0.45 μm pore filter because the cell density is determined using a hemacytometer. If indigenous algae is present, the sample may either be filtered (0.45 μm) or autoclaved, before testing/preparation. One mL of each nutrient stock solution is added to each liter of effluent to be used in preparing the dilutions. Required volumes of laboratory control and effluent are added to beakers for temperature adjustment (25° $\pm$ 1°C) in a hot water bath. After proper temperatures are achieved, exposure concentrations are prepared using designated graduated cylinders. Depending on test requirements or test specifications, laboratory water may be used for dilution of the effluent. 100 percent effluent (the IWC) or other concentrations are prepared for chronic tests according to the work order.

Chemical Determinations

Routine chemical parameters measured initially on all treatment solutions include pH and conductivity.

Total alkalinity, hardness, total residual chlorine, and ammonia are determined in each sample (100 percent effluent or receiving water). Total alkalinity and hardness are also measured in the laboratory culture medium. Chemical parameters are recorded on a data sheet (Figure 68).

Temperature Monitoring

The temperature of the test incubator is electronically monitored hourly by a data logger and thermocouple. Minimum, maximum, and average temperatures are calculated daily and recorded in a temperature logbook (Figures 54 and 55) and on the bioassay temperature data sheet (Figure 56).

Test Chamber Specifics

Test chambers are 250-mL glass Erlenmeyer flasks. The test solution volume is 100 mL per chamber. Three replicates totaling 300 mL of test solution per treatment are used.

Test Initiation

The test chambers are labeled and 100 mL of test solution is added to each flask. One mL of the algal inoculum is then added to each test chamber.

The flasks are randomly arranged on a shaker table or shelf in the 25°C incubator or algal test room. Bioassay start time is recorded on the data forms.

Test Termination

The test is terminated at 96 ± 2 hours after initiation. The algal growth in each flask is measured by cell counts using a hemacytometer and recorded on data forms (Figures 69 and 70). If necessary, each flask is preserved with Lugol's solution to prevent algal production during the cell counting process.

Data Analysis

Analysis of algae growth data follow EPA recommendations [4]. The effluent concentrations are analyzed for statistical differences in algal growth (cell density) using Dunnett's test. If the assumptions of homogeneity of variance and normality cannot be met, Steel's Many One Rank Test is used to compare the data. Additionally, an IC_{25} (the concentration at which 25 percent inhibition occurs) is calculated using the linear interpolation method [6].

Report

A formal report is typically issued within 2 weeks of test termination. It may be prepared by the Task Manager or the Laboratory Manager. The report contains:

- Chain-of-custody forms, which provide specific client and sample information
- A summary of test conditions
- Any deviations from test protocols or test conditions
- A tabular summary of growth data
- Photocopies of original bench sheets on growth and chemical parameters
- Task Manager's name
- Results of data analyses
- Information on test validity

Before the report is issued, it is reviewed by at least two staff personnel, including the Laboratory Manager, or designee, (Figure 62).

Test Validation

Part of CH2M HILL's laboratory QA/QC plan is to conduct concurrent internal laboratory control tests using the same methods, techniques, and organisms from the same pool.

The test results are acceptable if the algal cell density in the laboratory controls exceeds 2×10^5 cells/mL at the end of the test and do not vary more than 20 percent among replicates.

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8.0 Laboratory Maintenance and Equipment

8.1 Labware Cleaning

All glassware and reusable plasticware used for bioassays are cleaned in the following sequence:

- Hot soap and water wash
- Tap water rinse
- 10 percent Hcl rinse
- Tap water rinse
- Full strength methanol rinse
- Laboratory water (reverse osmosis/deionized) rinse, three times

8.2 General Laboratory Maintenance

Laboratory maintenance consists of monitoring the facility's equipment or systems, inventory and purchasing supplies, equipment repair, and general cleaning. The laboratory water, air, and temperature control systems are monitored daily (Figure 71). Other activities are performed weekly or as needed.

8.3 Instrument Maintenance

Routine instrument calibration or equipment use is recorded in logbooks (Figures 72 through 78). Calibration and maintenance follow manufacturer's specifications.

8.4 Major Laboratory Equipment

Table 8.1 lists the major equipment used at the bioassay laboratory.

8.5 Chemicals

Material Safety Data Sheets are kept on file for chemicals used in the laboratory. A chemical logbook is kept for recording the use of each chemical in the laboratory. Each chemical will have date received, intended use, storage location, amount received, expiration date, and date discarded recorded and will be assigned a chemical ID number in the logbook. This record provides information on all chemicals currently in use by the laboratory (Figure 79).

Table 8.1

QTY	EQUIPMENT	DESCRIPTION/MODEL
3	ROOF-TOP AIR CONDITIONERS	YORK,3 & 7.5 TON MODELS
1	THERMOSTAT CONTROL SYSTEM	HONEYWELL MODEL T7300/Q7300
1	WELL PUMP	STA-RITE 4" SPQ SUBMERSIBLE PUMP
1	WELL PRESSURE TANK	WELL MATE MODEL WM12WB
1	WATER TEMP.FAIL SAFE SYSTEM	CHROMALUX MODEL 4115 AC CONTROLLER, RKC INSTRUMENTS,DIGITAL CONTROLLER
2	LAB WATER CIRCULATION HEATERS	CHROMALUX, 4.5kw AND 9kw
1	FUME HOOD, PROTECTOR 72	LABCONCO, MODEL 72801
2,1	IRON AND CARBON FILTERS	CULLIGAN WATER SYSTEMS
1	FLAMMABLE STORAGE CABINET	VWR 30-gal
1	ACID/CORROSIVE CABINET	EAGLE 30-gal
1	WALK-IN REFRIGERATOR	GIBBCO SCIENTIFIC 8'x 8'x 7.5'
2	WALK-IN INCUBATORS,8'x 10'	VOLLRATH WITH CONVIRON CMP3244 CONTROLLER
1	FREEZER	WESTINGHOUSE,16cu.ft.
2	VACUUM/PRESSURE PUMPS	GAST MODEL 0522
2	PUMPS & CONTROLLERS	MASTER-FLEX MODEL N-07553-30
1	REGENERATIVE BLOWER, 1/3 HP	SWEETWATER,MODEL S-21
1	R/O-ULTRAPURE WATER SYSTEM	ZYZATECH,LAB TWO WITH 30gal COMPANION TANK
1	AUTOCLAVE/DRYER	NAPCO MODEL 9000-D
1	CENTRIFUGE, BENCHTOP	IEC MODEL HNSII
2	CULTURE INCUBATORS	PERCIVAL MODEL I-30BL AND I-30BLL
1	LAB OVEN	QUINCY,MODEL 30-392
1	COMPUTER SYSTEM	COMPAQ MONITOR AND 386 HARD DRIVE
1	LASER PRINTER	HEWLETT PACKARD, LASER JET IIP
1	ORBITAL SHAKER	LABLINE MODEL 3590
1	SHAKER,ORBIT JR.	LABLINE MODEL 3520
1	SEMI-MICRO BALANCE W/PRINTER	SARTORIUS, R200D WITH YDP 02-OD PRINTER
1	CERTIFIED THERMOMETER	ERTCO,INSTRUMENT No. 1647
3	DISSECTION SCOPES	NIKON SMZ-10 TRINOCULAR, SMZ-5 BINOCULAR
1	COMPOUND MICROSCOPE	NIKON, ALPHAPHOT-2
5	FIBER OPTIC ILLUMINATORS	DOLAN JENNER SERIES 180
1	HARVARD TRIP BALANCE,DBL.BEAM	OHAUS 2kg-5lb
2	TEMP. DATA LOGGERS	KANE-MAY, KM1202
1	ION ANALYZER	ORION MODEL EA 940
1	AMPEROMETRIC TITRATOR	FISCHER & PORTER,SERIES 17T2000
1	WATER ANALYZER	COLE-PARMER, MODEL 5566
1	SPECTROPHOTOMETER	SEQUIA TURNER MODEL 340
1	DIGITAL CONDUCTANCE METERS	YSI,MODEL 35
1	FLOURIMETER	TURNER, ID NUMBER 181
1	LIGHT METER	FOOT CANDLE/LUX METER
2	DISSOLVED OXYGEN METER	YSI, MODEL 58
2	pH METERS	BECKMAN PHI32
2	PORTABLE pH METER	CORNING MODEL 103 AND MODEL 106
1	PORTABLE CONDUCTIVITY METER	VWR MODEL 640

9.0 Quality Assurance/Quality Control

9.1 Program Overview

CH2M HILL's Bioassay Laboratory QA/QC program encompasses standardization and calibration of equipment and instruments, in-house culturing of all test organisms, laboratory control bioassays concurrent with every test, reference toxicant testing, facilities monitoring, and data management and review. A key element in all of these measures is the involvement of well-trained and knowledgeable personnel. Every employee with responsibilities in the bioassay program is thoroughly trained by the Laboratory Manager and Task Managers before taking an active role in either laboratory organism culturing or testing. Periodic meetings of all personnel are held to promote communication and to reinforce basic skills in the conduct of the program. Personal professional development is encouraged as a means to strengthen technical skills and expertise.

9.2 Quality Assurance/Quality Control Review

Bioassay Data Review

Bioassay investigators initial each data form as documentation of conducting the laboratory work. All bioassay data and reports are reviewed by the Laboratory Manager (or designee) in addition to at least another staff member (e.g., Task Manager). Depending on who analyzes the data, either the Task Manager or the Laboratory Manager verifies that the original data have been entered correctly on the computer. The Laboratory Manager also verifies that the data and report have been analyzed, written, and reviewed before final printing and distribution. A signoff form (Figure 62) for each client bioassay or test battery is used to provide QA and management of test data and information.

Laboratory Inspection

About once a month, the bioassay laboratory is internally inspected by at least the Laboratory Manager, QA Manager, or Hazard Communication Coordinator. The following items and facilities are evaluated:

- A check on the conditions of laboratory safety equipment and information (e.g., chemical container labeling) is done according to the safety log. Additionally, safety equipment (e.g., protective gear) is checked to verify that it is readily available and being used.

- Culture logbooks are checked for proper data and information entry (i.e., completeness, legibility, frequency, etc.).

- Culture summaries or logbook data are examined to assess organism conditions and quality.

- Laboratory equipment and facilities logbooks are examined for proper data and information entry, and systems are assessed for normal operation.

- Logbooks are checked to see that equipment is receiving maintenance and that apparatus and instruments are calibrated.

- General conditions and operations (e.g., information boards, cleanliness, organization, supplies, etc.) in the culture and test laboratories are assessed.

- Samples, test labware, test racks, and containers are examined for proper identification and labeling.

- The coldroom is checked to see that "old" samples (i.e., samples used in tests that have been terminated) have been disposed of properly.

- Test data, both client and internal, are checked to see that they are properly identified and filed.

- General and specific work activities are assessed to verify that they are in accordance with CH2M HILL's Bioassay Laboratory Standard Operating Procedures [1].

- The laboratory's Standard Operating Procedures [1] are checked and revisions are made to keep them up to date.

Inspection reports are kept on file in the Laboratory Manager's office. Corrective actions are implemented as necessary.

9.3 Calibration and Standardization of Equipment

All analytical instrumentation and test equipment used in the laboratory are routinely calibrated and standardized at scheduled intervals according to manufacturers' specifications and specific standards or as indicated in *Methods for Chemical Analysis of Water and Wastes* (U.S. EPA 600/4-79/020).

Instruments calibrated daily or before use include dissolved oxygen meters, pH meters, ISE analyzer (for ammonia analysis), amperometric titrator (for chlorine analysis), and the analytical balance. Equipment calibrated weekly to monthly include conductivity meters and the hardness and alkalinity titration reagents.

About once per year, QA samples or standards are obtained from external sources (e.g., U.S. EPA), and the analysis results are examined for acceptability. If the data are invalid, instrument maintenance, preparation of new reagents, or retraining are performed to correct the problem.

9.4 Test Organism Culture Status

The quality of the Bioassay Laboratory's test organisms is maintained through a stringent procedure of culture monitoring and maintenance (Section 6) and adherence to a Culture Monitoring Status System.

Various logbooks are used for recordkeeping and data entry with respect to:

- Organism production (e.g., fish egg production and hatching, daphnid reproduction)
- Organism general health and condition
- Organism management (e.g., fish breeder pair replacement; larval and juvenile fish tank stocking; daphnid monitoring, termination, and renewals; and population control)
- Tank/vessel maintenance
- Culture food preparation
- Water quality (chemical) measurements

Daily checks and weekly reviews or summaries are used to evaluate culture performance.

The following three-tiered scheme is used to monitor laboratory culture status and to provide an early warning system for operational problems:

Code	Condition
GREEN	Laboratory biological and physicochemical conditions meet or exceed established criteria for optimal laboratory operations.
YELLOW	Laboratory biological or physicochemical conditions do not meet established criteria (either absolutely or with respect to parameter/condition variability) for optimal operations. The laboratory remains operational with close scrutiny of

potential problem areas and implementation of remedial measures.

RED — Laboratory biological or physicochemical conditions render laboratory or culture system unsuitable for proper conduct of bioassays. The laboratory or culture system is either shut down or made unavailable during major system maintenance or overhaul.

Any deviation from "green status" assessed through continuous routine monitoring requires immediate notification of the Laboratory Manager or appointed supervisor and the initiation of corrective action. Rigid adherence to this system ensures a continuous supply of quality organisms for testing.

9.5 Laboratory Facilities Status

The status of the major laboratory facilities is monitored daily. These include:

- Laboratory well/RO water system
- Laboratory water temperature control system
- Laboratory heating and air conditioning system
- Incubators and coldroom temperature control system
- Temperature monitoring (data logger) system
- Pressurized air system

Logbooks are used to document system checks and data entry. Any facility-related problems assessed through routine monitoring require immediate notification of the Laboratory Manager or appointed supervisor and the initiation of corrective action.

9.6 Internal Laboratory Control Tests

It is a routine and required laboratory practice to include an internal control of CH2M HILL's laboratory culture medium for the organism being tested with every bioassay. This practice demonstrates overall health of the pooled stock for the test and provides a check on test procedural matters (e.g., food, handling, glassware cleanliness, and so on). The results of this internal control are entered on the bioassay data sheets as part of the documentation for the test. The data supplement routine culture monitoring data and observations. Should the laboratory control organisms not demonstrate $\geq$ 90-percent survival for an acute test or $\geq$ 80-percent survival for a chronic test, the bioassay may be considered invalid and will then be rerun at no expense to the client. Exceptions will be in the case of outliers where the problem was an identified contaminant or stress.

9.7 Reference Toxicant Testing

Testing Frequency

Bioassay of a standard or accepted reference toxicant provides a comprehensive expression of organism culture and test procedure quality or consistency. These tests are conducted on the following schedule:

Acute tests:	Approximately every month for all species currently offered for acute toxicity testing
Chronic tests:	Approximately every month for *Ceriodaphnia dubia*, *Pimephales promelas*, and *Selenastrum capricornutum*

Test Methods and Conditions

All test procedures used in reference toxicant testing are in accordance with test protocols listed in U.S. EPA 600/4-90-027, U.S. EPA 600/4-89/001, or as modified by specific regulatory recommended procedures. The Bioassay Laboratory currently uses reagent grade NaCl as a reference toxicant. The use of NaCl as a suitable reference toxicant has been established by the Environmental Research Laboratory–Duluth, EPA.[1]

Specific acute and chronic test methods follow bioassay procedures detailed in Section 7 with the following modifications:

- Acute fish tests are typically conducted using 10-day old fish ($\pm$ 2 days).
- A test medium (NaCl solution) is prepared as a single batch to be used for the duration of the test, or of the test battery when multiple species are tested concurrently. The NaCl solution is not refrigerated, but is stored at the appropriate test temperature (i.e., ambient laboratory).

The current ranges of the five NaCl concentrations used in reference toxicant testing are listed below. The diluent and stock solution preparation water is the appropriate culture medium: treated well water for fish and fish tank-conditioned water for daphnids.

Test Type	Species	NaCl Concentration (g/L)
Acute	Fathead Minnow	0–16
	Ceriodaphnia dubia	0–6
	Daphnia magna	0–10
	Daphnia pulex	0–6

[1]Theresa Norberg-King, personal communication, 1987.

Chronic	Fathead Minnow	0–15
	Ceriodaphnia dubia	0–4
	Selenastrum capricornutum	0–6

Acceptable Data Limits

Results from acute toxicity reference tests are assessed using available EPA and literature values for each test organism and the EPA guide of ± two standard deviations from the cumulative mean LC_{50} of previous tests. If a test value falls outside the EPA guideline, the test conditions, test organisms, and test procedures are audited for any anomalies and a retest is initiated after appropriate steps have been taken to rectify the situation. It should be noted that occasionally, a test value will fall outside the accepted guidelines because of the inherent variability of biological systems.

Little specific guidance is available on acceptable limits for chronic toxicity reference toxicant tests. CH2M HILL's approach to these tests follows the information available from the EPA—Duluth Laboratory.[2] The NOEC (no observable effect concentration) and LOEC (lowest observable effect concentration) are reported for each parameter and each species. The IC_{25} (the concentration at which 25 percent inhibition occurs) is reported for fathead minnow growth and *Ceriodaphnia dubia* reproduction only. A ± 2 standard deviation of the cumulative mean IC_{25} of previous tests is used as an acceptable test guide.

Test Results

All original data and summary tables of test results are kept in the reference toxicant file. CH2M HILL's summary tables of reference toxicant data are updated and submitted to the Wisconsin DNR monthly (and is available on request).

9.8 Data Management

All client bioassays, reference toxicant tests, and other internal work are kept on file at the laboratory. Client files also contain the original and at least one bound copy of the bioassay report, the work order, notes, memorandums, and other information pertaining to the test.

While a test is being performed, the daily chemical and organism monitoring data sheets are kept on a clipboard and stored in the appropriate bioassay laboratory. Wet chemistry analysis data sheets are kept in client folders in the Chemistry Laboratory. After the test is completed, data sheets and other information are placed in the client file in the Computer Room.

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[2]Theresa Norberg-King, personal communication, 1987.

All culture and laboratory data and information that are not bound, filled logbooks, and yearly logbooks are kept on file in the Computer Room or archive files. Currently, used culture and laboratory logbooks are kept in file boxes located throughout the laboratories.

All data entries are recorded in black ink and any corrections are initialed.

Groups of culture organisms are assigned lot numbers. Culture food and prepared media (e.g., mineral water, algae nutrient stocks) are assigned batch numbers. This system provides means to track organism, food, and media performance and to assist in identifying anomalies or problems.

Weekly or monthly culture and laboratory facility performance summaries are generated to assess long-term conditions and changes for management purposes. These summaries are kept on file, both disk and hard copy, in the Computer Room.

10011F4D.GLO

References

1. CH2M HILL. *Bioassay Laboratory Standard Operating Procedures.*© July 1990.

2. CH2M HILL. Toxics Control Statement of Qualifications.

3. Weber, C. I. (ed.). 1991. *Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms* (Fourth Edition). EPA/600/4-90/027. U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio. 293 p.

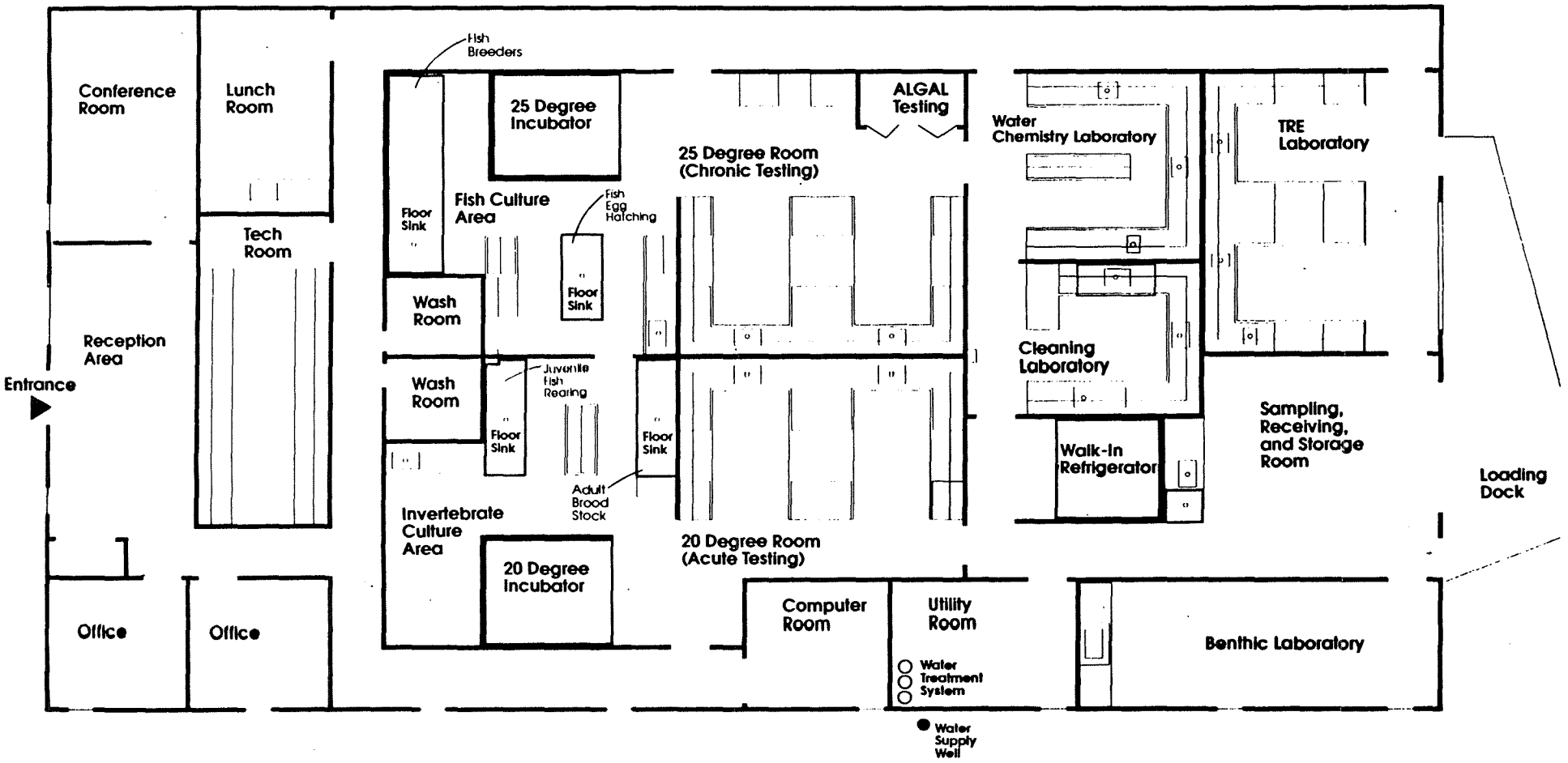
4. Weber, C. I., et al. 1989. *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms* (Second Edition). EPA/600/4-89/001. U.S. EPA, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio. 249 p.

5. Denny, J. S. 1987. *Guidelines for the Culture of Fathead Minnows*, Pimephales promelas, *for Use in Toxicity Tests*. EPA/600/3-87/001. U.S. EPA, Environmental Research Laboratory, Duluth, Minnesota. 42 p.

6. Norberg-King, T. 1988. *An Interpolation Estimate for Chronic Toxicity: The IC_p Approach*. National Effluent Toxicity Assessment Center Tech. Rep. 05-88. U.S. EPA, Environmental Research Laboratory, Duluth, MN. 15 p.

FIGURES—LABORATORY FORMS

JOB NO. FIG NO. DATE



15779 WEST RYERSON ROAD

FIGURE 1

CH2M HILL Milwaukee Environmental Toxicology Laboratory (6,100 sq. ft)

Figure 2

LABORATORY WATER QUALITY LOG

DATE	WATER	DO	COND	pH	ALK	HARD	IRON	COMMENTS
	25–Fish							
	20–Fish							
	25–F.T.							
	20–F.T.							
Initials								
	25–Fish							
	20–Fish							
	25–F.T.							
	20–F.T.							
Initials								
	25–Fish							
	20–Fish							
	25–F.T.							
	20–F.T.							
Initials								
	25–Fish							
	20–Fish							
	25–F.T.							
	20–F.T.							
Initials								
	25–Fish							
	20–Fish							
	25–F.T.							
	20–F.T.							
Initials								

25–Fish = 25 C Headbox Outlet Water
20–Fish = 20 C Headbox Outlet Water
25–F.T. = Ceriodaphnia GAC Water
20–F.T. = Daphnia Flow–Through Tank Water

DO = Dissolved Oxygen (mg/L)
COND = Conductivity (mmho)
ALK = Total Alkalinity (mg/L CaCO3)
HARD = Hardness (mg/L CaCO3)
IRON = Dissolved Iron (mg/L)

Figure 3

DILUTE MINERAL WATER (DMW)/SYNTHETIC WATER (SW) LOG

DATE PREPARED	DMW NO.	SW NO.	DO (mg/L)	pH	COND (mmho)	ALK (mg/L)	HARD (mg/L)	COMMENTS	INITIALS

Figure 4

CH2M HILL

CHAIN OF CUSTODY RECORD FOR NPDES COMPLIANCE BIOMONITORING

Client Name	Client Shipping Address	NPDES Number
Sample Kit Tracking Information No. of Cooler ____ of ____ Total No. of Bottles ____	Method of Shipment (Check One) ☐ Fed X ____ ☐ Pickup ____ ☐ UPS ____ ☐ Other ____	Prepared by/Date: Shipped by/Date:
Ship Samples to: CH2M HILL Bioassay Laboratory 15779 W. Ryerson Road New Berlin, WI 53151 Phone: (414) 784-0448 Fax: (414) 784-0353		

Sample Kit Received By: (Signature) Date Time	Condition of Seal Upon Receipt (Check One) Intact ☐ Other ☐ (Describe) ____

Composite Sample Information	Description of Sampling Site	Sample Container
Flow Proportional ☐ Time Interval ☐ Samples/Hour ____ Volume/Sample ____ Total Hours ____ Total Volume ____ Initiated: Date ____ Time ____ Ended: Date ____ Time ____ Chilled During Collection Yes ☐ No ☐		Glass ☐ Plastic ☐ New ☐ Used ☐ Refrigerant Used For Shipping Wet Ice ☐ Blue Ice ☐ Other ☐ ____ Sample(s) Shipped Via UPS ☐ Fed X ☐ Other ☐ ____

Outfall Number	Date	Time	Sample Type: Composite	Sample Type: Grab	No. of Containers	Volume	Sampled by (Signature)	Analysis Require/Comments	For Lab Use Sample ID No.

Sampled By and Title (Signature) Date Time	Relinquished By: (Signature)	Date Time
Received By: (Signature) Date Time	Relinquished By: (Signature) Date Time	Received By Lab: (Signature) Date Time

DISTRIBUTION: Original - Lab, Yellow - Client

Figure 5

SAMPLE LOG

DATE RECEIVED	PROJECT NUMBER	CLIENT	SAMPLE DESCRIPTION	SAMPLE ID NO.	TEST* A	TEST* C	COMMENTS	INTLS

*A = Acute C = Chronic

CH2M HILL Milwaukee Bioassay Laboratory

Figure 6

BIOASSAY SAMPLE RECEIPT CHARACTERIZATION

CLIENT ______________________________ **PROJECT NO.** ______________

DATE RECVD	SAMPLE NO. DESCRIPTION	TEMP (C)	DO (mg/L)	pH	COND (mmho)	INITIALS

FILTER _____ DECHLORINATE _____ USE: IMMEDIATE _____ STORE (4 C) _____
ALIQUOTS HOMOGENIZED _____ CONTAINER TYPE (G/P) _____ ODOR _____
APPEARANCE : CLEAR _____ CLOUDY _____ SOLIDS _____ COLOR ______________
COMMENTS

DATE RECVD	SAMPLE NO. DESCRIPTION	TEMP (C)	DO (mg/L)	pH	COND (mmho)	INITIALS

FILTER _____ DECHLORINATE _____ USE: IMMEDIATE _____ STORE (4 C) _____
ALIQUOTS HOMOGENIZED _____ CONTAINER TYPE (G/P) _____ ODOR _____
APPEARANCE : CLEAR _____ CLOUDY _____ SOLIDS _____ COLOR ______________
COMMENTS

DATE RECVD	SAMPLE NO. DESCRIPTION	TEMP (C)	DO (mg/L)	pH	COND (mmho)	INITIALS

FILTER _____ DECHLORINATE _____ USE: IMMEDIATE _____ STORE (4 C) _____
ALIQUOTS HOMOGENIZED _____ CONTAINER TYPE (G/P) _____ ODOR _____
APPEARANCE : CLEAR _____ CLOUDY _____ SOLIDS _____ COLOR ______________
COMMENTS

DATE RECVD	SAMPLE NO. DESCRIPTION	TEMP (C)	DO (mg/L)	pH	COND (mmho)	INITIALS

FILTER _____ DECHLORINATE _____ USE: IMMEDIATE _____ STORE (4 C) _____
ALIQUOTS HOMOGENIZED _____ CONTAINER TYPE (G/P) _____ ODOR _____
APPEARANCE : CLEAR _____ CLOUDY _____ SOLIDS _____ COLOR ______________
COMMENTS

Form BL42

Revised 6/93

CH2M HILL Milwaukee Bioassay Laboratory

Figure 7

ORGANISM INTRODUCTION/ACCLIMATION LOG

DATE	INTRODUCTION			ACCLIMATION			COMMENTS	INITIALS	VERIFIED BY
	ORGANISM	SOURCE	CONDITION	ID NO.	TANK NO.	PERIOD			

Form BL53

Revised 7/21/90

Figure 8

LARVAL FISH TEST ISOLATION LOG

DATE	LOT No.	PAN No.	CLIENT	PROJECT No.	TIME ISOLATED	NUMBER USED	COMMENTS

FORM BL84

Revised 7/19/90

Figure 9

AGE MONITORING FOR *CERIODAPHNIA* BIOASSAY

PROJECT NO.	CLIENT	RACK/LOT NO.
1._____	1._____	1._____
2._____	2._____	2._____
3._____	3._____	3._____

SLOT NO.	MONITORING DATE / TIME (HOURS) / RACK / LOT NO. / REPRODUCTIVE CONDITION*						COMMENTS
	/ /	/ /	/ /	/ /	/ /	/ /	
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
TOTAL							
DET. BY							

* G=Gravid E=Eyed R=Releasing young B=Brood Br=Brood just released
Bp=Brood w/posterior eggs in adult

SELECTED ORGANISM SLOT NUMBERS FOR PROJECT	No. YOUNG POOLED FOR PROJECT
1._____ 2._____ 3._____	1._____ 2._____ 3._____

Figure 10

CERIODAPHNIA BATCH CULTURE ISOLATION LOG

DATE	LOT No.	CLIENT	PROJECT No.	PRE-ISOLATE POPULATION	TIME ISOLATED	No. USED FOR TEST	COMMENTS	INITIALS

CH2M HILL Milwaukee Bioassay Laboratory

Figure 11

DAPHNIA TEST ISOLATION LOG

DATE	SPECIES	LOT No.	CLIENT	PROJECT No.	PRE-ISOLATE POPULATION	TIME ISOLATE	No. USED FOR TEST	COMMENTS	INTLS

Form BL82

Revised 7/17/90

Figure 12

48-HOUR ACUTE REFERENCE TOXICANT
CHEMICAL DATA*

TEST NO.:_____ TEST ORGANISM: _____
REFERENCE TOXICANT: _____ DILUENT: _____
STOCK SOLN.: CONC._____ SOLVENT _____ COND._____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____

TREAT. NO.	TEST SOLN	PARAMETER	READING	EXPOSURE DAY 1	EXPOSURE DAY 2	COMMENTS
1	Lab Control	DO	I			
			F			
		pH	I			
			F			
		COND	I			
2		DO	I			
			F			
		pH	I			
			F			
		COND	I			
3		DO	I			
			F			
		pH	I			
			F			
		COND	I			
4		DO	I			
			F			
		pH	I			
			F			
		COND	I			
5		DO	I			
			F			
		pH	I			
			F			
		COND	I			
6		DO	I			
			F			
		pH	I			
			F			
		COND	I			
	INITIAL READING DETERMINED BY:					
	FINAL READING DETERMINED BY:					

*DO as mg/L COND as mmho I = INITIAL F = FINAL

Figure 13

CH2M HILL NEW BERLIN BIOASSAY LAB

48-HOUR ACUTE REFERENCE TOXICANT TEST
SURVIVAL DATA

TEST NO.: _____ TEST ORGANISM: _____ LOT NO.:_____ AGE:_____
TEST START DATE: _____ TIME:_____ TEST END DATE: _____ TIME: _____
REFERENCE TOXICANT:_____ DILUENT:_____
ANALYST(s): _____

TREAT. NO.	TEST SOLN	REP	FATALITIES PER EXPOSURE PERIOD (Hrs) 24	48	TOTAL FATALITIES	MEAN SURVIVAL	COMMENTS
1		A					
		B					
2		A					
		B					
3		A					
		B					
4		A					
		B					
5		A					
		B					
6		A					
		B					
	DATE						
	DETERMINED BY						

Figure 14

96-HR ACUTE REFERENCE TOXICANT TEST

CHEMICAL DATA*

TEST NO.:_____ TEST ORGANISM :_____

REFERENCE TOXICANT: _____ DILUENT: _____

STOCK SOLN CONC: _____ SOLVENT: _____ CONDUCTIVITY: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____TIME: _____

ANALYST(s):_____ CHEMICAL ID.:_____

INITIAL CHEMICAL DATA

TREAT. NO.	TEST CONC	PARAMETER	EXPOSURE HOURS 0	24	48	72	COMMENTS
1	0 (LAB)	DO					
		pH					
		COND					
2		DO					
		pH					
		COND					
3		DO					
		pH					
		COND					
4		DO					
		pH					
		COND					
5		DO					
		pH					
		COND					
6		DO					
		pH					
		COND					
		DET. BY					

FINAL CHEMICAL DATA

TREAT. NO.	TEST CONC	PARAMETER	EXPOSURE HOURS 24	48	72	96	COMMENTS
1	0 (LAB)	DO					
		pH					
2		DO					
		pH					
3		DO					
		pH					
4		DO					
		pH					
5		DO					
		pH					
6		DO					
		pH					
		DET. BY					

*DO as mg/L COND as mmho

Figure 15

96-HR ACUTE REFERENCE TOXICANT TEST
SURVIVAL DATA

TEST NO.: _____

TEST ORGANISM: _____ LOT NO.: _____ AGE: _____

TEST START DATE: _____ TIME:_____ TEST END DATE: _____ TIME: _____

REFERENCE TOXICANT:_____DILUENT:_____

ANALYST(s): _____

TREAT. NO.	TEST CONC g/L	REP	FATALITIES per EXPOSURE PERIOD (HOURS) 24	48	72	96	TOTAL FATALITIES	% SURVIVAL	MEAN SURVIVAL	COMMENTS
1	0	A								
	LAB	B								
2		A								
		B								
3		A								
		B								
4		A								
		B								
5		A								
		B								
6		A								
		B								
	DET. BY									

Figure 16

CH2M HILL NEW BERLIN BIOASSAY LAB

CHRONIC REFERENCE TOXICANT TEST

CHEMICAL DATA*

TEST NO. RTC -_____ TEST ORGANISM: _____ LOT NO.: _____
REFERENCE TOXICANT: _____ DILUENT: _____
STOCK SOLN. CONC._____ SOLVENT _____ COND._____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ Chemical ID.:_____

INITIAL CHEMICAL DATA*

TREAT. NO.	TEST CONC (g/L)	PARAMETER	EXPOSURE DAY 0	1	2	3	4	5	6	COMMENTS
1	0 (LAB)	DO								
		pH								
		COND								
2		DO								
		pH								
		COND								
3		DO								
		pH								
		COND								
4		DO								
		pH								
		COND								
5		DO								
		pH								
		COND								
6		DO								
		pH								
		COND								
		DET. BY								

FINAL CHEMICAL DATA

TREAT. NO.	TEST CONC (g/L)	PARAMETER	EXPOSURE DAY 1	2	3	4	5	6	7	COMMENTS
1	0 (LAB)	DO								
		pH								
2		DO								
		pH								
3		DO								
		pH								
4		DO								
		pH								
5		DO								
		pH								
6		DO								
		pH								
		DET. BY								

*DO as mg/L COND as mmho

Figure 17

CH2M HILL NEW BERLIN BIOASSAY LAB

CERIODAPHNIA CHRONIC REFERENCE TOXICANT SURVIVAL DATA

TEST NO.: RTCC-_____ LOT NO.: _____ ANALYST(s): _____

REFERENCE TOXICANT:_____ DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____ Page_____ of _____

TREAT. NO.	TEST SOLN	DAY	OFFSPRING PER REPLICATE 1	2	3	4	5	6	7	8	9	10	COMMENTS
		1											
		2											
		3											
		4											
		5											
		6											
		7											

	3-BROOD SUMMARY TOTAL	MEAN
NO. YOUNG		
NO. BROODS		
ADULT FATALITIES		% SURVIVAL

TREAT. NO.	TEST SOLN	DAY	OFFSPRING PER REPLICATE 1	2	3	4	5	6	7	8	9	10	COMMENTS
		1											
		2											
		3											
		4											
		5											
		6											
		7											

	3-BROOD SUMMARY TOTAL	MEAN
NO. YOUNG		
NO. BROODS		
ADULT FATALITIES		% SURVIVAL

DAY	1	2	3	4	5	6	7
DATE							
DET./FED BY							

Form BLRTC04

/ = OK G = GRAVID E = EYED R = RELEASING YOUNG AD = ADULT DEAD YD = YOUNG DEAD

Revised 6/93

Figure 18

CH2M HILL NEW BERLIN BIOASSAY LAB

CHRONIC REFERENCE TOXICANT TEST

LARVAL FATHEAD MINNOW SURVIVAL DATA

TEST NO. RTCF-_____ LOT NO.:_____ ANALYST(s): _____

REFERENCE TOXICANT: _____ DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

TREAT. NO.	TEST SOLN (g/L)	REP	FATALITIES PER EXPOSURE DAY 1	2	3	4	5	6	7	TOTAL SURV	FATL	% SURVIVAL	COMMENTS
1		A											
		B											
		C											
		D											
2		A											
		B											
		C											
		D											
3		A											
		B											
		C											
		D											
4		A											
		B											
		C											
		D											
5		A											
		B											
		C											
		D											
6		A											
		B											
		C											
		D											
DETERMINED BY													
FEEDING BY		AM											
		PM											

Figure 19

CHRONIC REFERENCE TOXICANT TEST
LARVAL FATHEAD MINNOW CHRONIC BIOASSAY GROWTH DATA

TEST NO.: RTCF-_____ LOT NO.:____ ____ANALYST(s):_____
REFERENCE TOXICANT:_____ DILUENT: _____
TEST START: DATE _____ TIME_____ TEST END: DATE _____TIME _____

TEST SOLN	TREAT REP	TARE (mg)	TARE + DRY FISH WT (mg)	TOT. FISH WT (mg)	Signif. Difference Test		IC25 Test	
					No. Surv.	Fish Wt.	No. Exposed	Fish Wt.
	1A							
	1B							
	1C							
	1D							
	2A							
	2B							
	2C							
	2D							
	3A							
	3B							
	3C							
	3D							
	4A							
	4B							
	4C							
	4D							
	5A							
	5B							
	5C							
	5D							
	6A							
	6B							
	6C							
	6D							
					Mean 1 =		Mean 1 =	
					Mean 2 =		Mean 2 =	
					Mean 3 =		Mean 3 =	
					Mean 4 =		Mean 4 =	
					Mean 5 =		Mean 5 =	
					Mean 6 =		Mean 6 =	

COMMENTS:

Figure 20

ALGAE CULTURE LOG

PREP. DATE	LOT No.	CULTURE TYPE@	INITIAL pH	pH ADJUST	COND. (mmho)	INOCULUM SOURCE+	INOCULUM DENSITY*	INITIAL CELL DENSITY**	CONC. CELL DENSITY*	mL OF CONC.	INITIALS

@ A = AXENIC
F = FOOD

+ S = SLANT
A = AXENIC
F = FOOD

*DENSITIES RECORDED AS n x 10^6 CELLS/mL

** GRAND MEAN OF INITIAL COUNTS

COMMENTS:

Figure 21

CH2M HILL Milwaukee Bioassay Laboratory

DAPHNIA FLOW-THROUGH SYSTEM LOG

DATE	FED	MAINTENANCE		POPULATION PARAMETER		COMMENTS	INITIALS
		SIPHON	POP. THIN	DENSITY	% GRAVID		

Figure 22

CH2M HILL Milwaukee Bioassay Laboratory

DAPHNIA MAGNA CULTURE LOG

DATE	LOT NO.	MEDIA	FED	TEST ISOLATE	RENEWAL DAY ONLY		FINAL READING		COMMENTS	INITIALS
					ADULT SURVIVAL WEEK 1	*NO. OF OFFSPRING WEEK 2	DO	pH		

Form BL6

*Total of renewal and isolation estimates.

Rev. Date 6/21/90

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Figure 23

DAPHNIA PULEX CULTURE LOG

DATE	LOT NO.	MEDIA	FED	TEST ISOLATE	RENEWAL DAY ONLY		FINAL READING		COMMENTS	INITIALS
					ADULT SURVIVAL WEEK 1	*NO. OF OFFSPRING WEEK 2	DO	pH		

Form BL7

*Total of renewal and isolation estimates.

Rev. Date 6/21/90

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Figure 24

CERIODAPHNIA CULTURE LOG

DATE	LOT NO.	MEDIA	FED	TEST ISOLATE	RENEWAL DAY ONLY		FINAL READING		COMMENTS	INITIALS
					ADULT SURVIVAL WEEK 1	*NO. OF OFFSPRING WEEK 2	DO	pH		

Form BL8

*Total of renewal and isolation estimates.

Rev. Date 6/21/90

Figure 25

CERIODAPHNIA CULTURE MONITORING DATA

W

DATE SET_____ RACK NO._____ LOT NO._____
SET BY_____ MEDIA_____
PARENT LOT NO._____ SLOT NO. PARENT ____ ____ ____ ____ ____ ____
YOUNG 1-5 6-10 11-15 16-20 21-25 26-30

SLOT NO.	NUMBER OF LIVE YOUNG PER DAY							3-BROOD TOTAL	COMMENTS
	1	2	3	4	5	6	7		
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									
Day ---->	THU	FRI	SAT	SUN	MON	TUE	WED		OFFSPRING TOTAL
CHECKED/ FED BY									OFFSPRING MEAN
									ADULT % SURVIVAL
FINAL pH									MEAN NO. BROODS

/ = OK G = GRAVID E = EYED R = RELEASING YOUNG B = BROOD
AD = ADULT DEAD YD = YOUNG DEAD

Figure 26

CERIODAPHNIA CULTURE MONITORING DATA

F

DATE SET_____ RACK NO._____ LOT NO._____
SET BY_____ MEDIA_____
PARENT LOT NO._____ SLOT NO. PARENT ____ ____ ____ ____ ____ ____
YOUNG 1-5 6-10 11-15 16-20 21-25 26-30

SLOT NO.	NUMBER OF LIVE YOUNG PER DAY							3-BROOD TOTAL	COMMENTS
	1	2	3	4	5	6	7		
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									
Day ---->	SAT	SUN	MON	TUE	WED	THU	FRI		OFFSPRING TOTAL
CHECKED/ FED BY									OFFSPRING MEAN
									ADULT % SURVIVAL
FINAL pH									MEAN NO. BROODS

/ = OK G = GRAVID E = EYED R = RELEASING YOUNG B = BROOD
AD = ADULT DEAD YD = YOUNG DEAD

Figure 27

CERIODAPHNIA/DAPHNIA FOOD LOG

DATE	BATCH No.	DISH ID NO.	FOOD C or D	TARE (mg)	TARE + FOOD (mg)	SOLIDS (mg/L)	COMMENTS	INITIALS

Figure 28

FATHEAD MINNOW EGG PRODUCTION LOG
NUMBER OF EGGS SPAWNED PER DATE

Date ---> / FISH PAIR	SUN	MON	TUE	WED	THU	FRI	SAT	WEEKLY TOTAL / PAIR	COMMENTS
1A									
1B									
1C									
1D									
2A									
2B									
2C									
2D									
3A									
3B									
3C									
3D									
4A									
4B									
4C									
4D									
5A									
5B									
5C									
5D									
DAILY SUBTOTAL									WEELKLY SUBTOTAL

D = Discard eggs
CP = Change fish pair
S = Spawn less than 50 eggs

Figure 29

FATHEAD MINNOW EGG PRODUCTION LOG
NUMBER OF EGGS SPAWNED PER DATE

Date ---> / FISH PAIR	SUN	MON	TUE	WED	THU	FRI	SAT	WEEKLY TOTAL / PAIR	COMMENTS
6A									
6B									
6C									
6D									
7A									
7B									
7C									
7D									
8A									
8B									
8C									
8D									
9A									
9B									
9C									
9D									
10A									
10B									
10C									
10D									
DAILY 6-10 SUBTOTAL									WEEKLY 6-10 SUBTOTAL
DAILY 1-5 SUBTOTAL									WEEKLY 1-5 SUBTOTAL
DAILY TOTAL									WEEKLY TOTAL
DET. BY									

FED: A.M.							
P.M.							

D = Discard eggs
CP = Change fish pair
S = Spawn less than 50 eggs

Figure 30

FATHEAD MINNOW BREEDER LOG

	TANK	PAIR		COMMENTS				
DATE	QUADRANT	REPLACED	JUMP OUT	NO EGGS	DEAD	STRESSED	OTHER	INITIALS

Figure 31

FATHEAD MINNOW EGG INCUBATION LOG

	DATE	SPAWN-S HATCH-H	NO. EGGS W/FUNGUS	NO. EGGS UNFERTIL.	TEMP. (C)	TILE ID / COMMENTS	INITIALS
SUN							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
MON							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
TUE							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
WED							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			

Figure 32

FATHEAD MINNOW EGG INCUBATION LOG

	DATE	SPAWN-S HATCH-H	NO. EGGS W/FUNGUS	NO. EGGS UNFERTIL.	TEMP. (C)	TILE ID / COMMENTS	INITIALS
THU							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
FRI							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
SAT							
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			
PAN NO.		S					
LOT NO.							
NO. TILES							
NO. EGGS							
		H	%	%			

Figure 33

FATHEAD MINNOW BREEDER TANK MAINTENANCE LOG

DATE	TANK NO.	SIPHON	CLEAN	FILTER CLEAN	NEEDLE CLEAN	COMMENTS	INITIALS

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Figure 34

FATHEAD MINNOW BREEDER TANK TEMPERATURE

DATE Mo_____ Yr_____	TANK NO. TEMPERATURE (Degrees C) Criteria = 24 to 26 C										INTLS
	1	2	3	4	5	6	7	8	9	10	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											
25											
26											
27											
28											
29											
30											
31											

COMMENTS

RN = Replace Needle CN = Clean Needle

Figure 35

FATHEAD MINNOW LARVAE/JUVENILE LOG

TANK SET-UP DATE	LOT NO.	TANK NO.	HATCH DATES	STOCKING DENSITY	FISH USAGE: DATE			COMMENTS	INITIALS
					TEST	DISCARD	STOCK		

Figure 36

FATHEAD MINNOW JUVENILE TANK MAINTENANCE LOG

DATE	TANK NO.	SIPHON	CLEAN	FILTER CLEAN	COMMENTS	INITIALS

Figure 37

FATHEAD MINNOW ADULT STOCK LOG

DATE FISH INTRODUCED:_____STOCKING DENSITY:_____LOT No.:_____

BASKET			
DATE	No. OF DEAD	COMMENTS	INITIALS

DISCARD DATE:_____

Figure 38

FATHEAD MINNOW ADULT STOCK TANK MAINTENANCE LOG

DATE	BASKET ID.	SIPHON	CLEAN	FILTER CLEAN	COMMENTS	INITIALS

Figure 39

CULTURE
BRINE SHRIMP HATCHERY LOG

DATE Mo___Yr___	FUNNEL ID	TIME	TEMP. (C)	COMMENTS	INITIALS AM | PM
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					

Figure 40

TEST
BRINE SHRIMP HATCHERY LOG

DATE Mo___Yr___	FUNNEL ID	TIME	TEMP. (C)	COMMENTS	INITIALS AM | PM
1	|	|			|
2	|	|			|
3	|	|			|
4	|	|			|
5	|	|			|
6	|	|			|
7	|	|			|
8	|	|			|
9	|	|			|
10	|	|			|
11	|	|			|
12	|	|			|
13	|	|			|
14	|	|			|
15	|	|			|
16	|	|			|
17	|	|			|
18	|	|			|
19	|	|			|
20	|	|			|
21	|	|			|
22	|	|			|
23	|	|			|
24	|	|			|
25	|	|			|
26	|	|			|
27	|	|			|
28	|	|			|
29	|	|			|
30	|	|			|
31	|	|			|

Figure 41

FISH CULTURE QC SURVIVAL DATA

			1-WEEK OLD			4-WEEK OLD				
DATE	TANK No.	LOT No.	No. REMOVED	ESTIMATED* POPULATION	% SURVIVAL°	No. REMOVED	FINAL* COUNT	% SURVIVAL °	INITIALS	COMMENTS

*** Estimated Population = 250 - No. of fish removed.**

° % Survival 100 - [(No. dead / 250) x 100]

FISHSURV.XLS

Figure 42

Ceriodaphnia Individual Culture

WEDNESDAY				FRIDAY			
Date	% Surv.	% Males*	No. in 3 Broods	Date	% Surv.	% Males*	No. in 3 Broods

SURVIVAL QC PREFERENCE = 90%
SURVIVAL MINIMUM CRITERIA = 80%
*NON-PRODUCING ORGANISMS: MAX = 20%

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Figure 43

TOTAL ALKALINITY LOG

ANALYZED DATE _____ pH CALIBRATION: 4 ____ 7 ____ TITRANT CALIBRATION DATE _____

CORRECTION FACTOR _____

ANALYSIS TIME	QA	SAMPLE NO.	SAMPLE pH	SAMPLE VOL mL	CONVERT FACTOR	mL of TITRANT to pH 4.5	ALKALINITY mL CaCO3	COMMENTS	INITIALS

QA : 1 Duplicate for 1–10 samples; 2 duplicates for 11–20 samples; etc...

Figure 44

HARDNESS LOG

CALIBRATE DATE	CORRECT FACTOR	ANALYZED DATE	QA	SAMPLE NO.	SAMPLE VOL mL	CONVERT FACTOR	mL of TITRANT	HARDNESS mL CaCO3	COMMENTS	INITIALS

QA : 1 Duplicate for 1 – 10 samples; 2 duplicates for 11 – 20 samples; etc.

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Figure 45

TOTAL RESIDUAL CHLORINE LOG

DATE	STANDARD SOLN mg/L	STANDARD READING mg/L	SAMPLE NO.	POINTER INITIAL READING	POINTER FINAL READING	mL of TITRANT	TOT. RES. CHLORINE mg/L	COMMENTS	INITIALS

Form BL73

Revised 6/25/90

Figure 46

TOTAL AMMONIA LOG

DATE	CALIBRATE SLOPE	SAMPLE NO.	TOTAL AMMONIA mg/L	COMMENTS	INITIALS

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Figure 47

SUPPLEMENTAL CHEMICAL DATA SUMMARY

PROJECT NO. ______________________ CLIENT ______________________________

TEST DATE ________________________ SUMMARIZED BY ____________________

LABORATORY CONTROL				
	CONTROL I.D.			
TOTAL ALKALINITY mg/L CaCO3				
HARDNESS mg/L CaCO3				

SAMPLE DESCRIPTION				
	SAMPLE NO.			
TOTAL ALKALINITY mg/L CaCO3				
HARDNESS mg/L CaCO3				
TOTAL RESIDUAL CHLORINE mg/L				
TOTAL AMMONIA mg/L				

SAMPLE DESCRIPTION				
	SAMPLE NO.			
TOTAL ALKALINITY mg/L CaCO3				
HARDNESS mg/L CaCO3				
TOTAL RESIDUAL CHLORINE mg/L				
TOTAL AMMONIA mg/L				

SAMPLE DESCRIPTION				
	SAMPLE NO.			
TOTAL ALKALINITY mg/L CaCO3				
HARDNESS mg/L CaCO3				
TOTAL RESIDUAL CHLORINE mg/L				
TOTAL AMMONIA mg/L				

NA = Not Analyzed

* = Duplicate for QA

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Figure 48

48-HOUR ACUTE TEST INITIAL CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

INITIAL CHEMICAL MEASUREMENT

TREAT. NO.	TEST SOLN	PARAMETER	EXPOSURE PERIOD (HR) 0	24	COMMENTS
1		DO			
		pH			
		COND			
2		DO			
		pH			
		COND			
3		DO			
		pH			
		COND			
4		DO			
		pH			
		COND			
5		DO			
		pH			
		COND			
6		DO			
		pH			
		COND			
7		DO			
		pH			
		COND			
	DATE				
	SAMPLE No.				
	DETERMINED BY				

*DO as mg/L COND as mmho

Figure 49

48-HOUR ACUTE TEST FINAL CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

FINAL MEASUREMENT

TREAT. NO.	TEST SOLN	PARAMETER	EXPOSURE PERIOD (HRS) 24	48	COMMENTS
1		DO			
		pH			
2		DO			
		pH			
3		DO			
		pH			
4		DO			
		pH			
5		DO			
		pH			
6		DO			
		pH			
7		DO			
		pH			
	DATE				
	DETERMINED BY				

*DO as mg/L COND as mmho

Figure 50

48-HOUR ACUTE NONRENEWAL TEST CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ LAB MEDIA /No.: _____

SAMPLE No.(s): _____ CONTROL/DILUENT: _____

SAMPLE DESCRIPTION: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLUTION	PARA-METER	INITIAL	FINAL		COMMENTS
			0H	24H	48H	
1		DO				
		pH				
		COND				
2		DO				
		pH				
		COND				
3		DO				
		pH				
		COND				
4		DO				
		pH				
		COND				
5		DO				
		pH				
		COND				
6		DO				
		pH				
		COND				
7		DO				
		pH				
		COND				
INITIAL DETERMINED BY / DATE						
FINAL DETERMINED BY / DATE						

*DO as mg/L COND as mmho

Figure 51

96-HOUR ACUTE TEST INITIAL CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

INITIAL CHEMICAL MEASUREMENT

TREAT. NO.	TEST SOLN	PARAMETER	EXPOSURE PERIOD (HRS) 0	24	48	72	COMMENTS
1		DO					
		pH					
		COND					
2		DO					
		pH					
		COND					
3		DO					
		pH					
		COND					
4		DO					
		pH					
		COND					
5		DO					
		pH					
		COND					
6		DO					
		pH					
		COND					
7		DO					
		pH					
		COND					
	DATE						
	SAMPLE No.						
	DETERMINED BY						

*DO as mg/L COND as mmho

Figure 52

96-HOUR ACUTE TEST FINAL CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ LAB MEDIA /No.: _____

SAMPLE No.(s): _____ CONTROL/DILUENT: _____

SAMPLE DESCRIPTION: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____

FINAL MEASUREMENT

TREAT. NO.	TEST SOLN	PARAMETER	EXPOSURE PERIOD (HRS) 24	48	72	96	COMMENTS
1		DO					
		pH					
2		DO					
		pH					
3		DO					
		pH					
4		DO					
		pH					
5		DO					
		pH					
6		DO					
		pH					
7		DO					
		pH					
	DATE						
	DETERMINED BY						

*DO as mg/L

Figure 53

96-HOUR ACUTE NONRENEWAL TEST CHEMICAL DATA*

(1-7 TREATMENTS)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLUTION	PARA-METER	INITIAL	FINAL				COMMENTS
			0H	24H	48H	72H	96H	
1		DO						
		pH						
		COND						
2		DO						
		pH						
		COND						
3		DO						
		pH						
		COND						
4		DO						
		pH						
		COND						
5		DO						
		pH						
		COND						
6		DO						
		pH						
		COND						
7		DO						
		pH						
		COND						
INITIAL DETERMINED BY / DATE								
FINAL DETERMINED BY / DATE								

*DO as mg/L COND as mmho

Figure 54

DATA LOGGER A

TEMPERATURE SUMMARY LOG

DATE	CH 1: 24–26 C 25° WALK-IN			CH 2: 24–27 C 25° LAB			CH 3: 27–30 C 25° HEAD BOX			CH 4: 24–26 C ALGAE TEST			CH 5: 24–26 C CERIO INCUBATOR				
MO__ YR__	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN	COMMENTS	INITIALS
1																	
2																	
3																	
4																	
5																	
6																	
7																	
8																	
9																	
10																	
11																	
12																	
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21																	
22																	
23																	
24																	
25																	
26																	
27																	
28																	
29																	
30																	
31																	
MO SUM																	

Form BL51A

Revised 7/11/90

Figure 55

DATA LOGGER B

TEMPERATURE SUMMARY LOG

DATE	CH 1: 18–22 C 20° WALK-IN			CH 2: 19–22 C 20° LAB			CH 3: 20–23 C 20° HEAD BOX			CH 4: 24–26 C ALGAE CULTURE			CH 5: 3–5 C COLD ROOM			COMMENTS	INITIALS
MO__YR__	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN	AVG	MAX	MIN		
1																	
2																	
3																	
4																	
5																	
6																	
7																	
8																	
9																	
10																	
11																	
12																	
13																	
14																	
15																	
16																	
17																	
18																	
19																	
20																	
21																	
22																	
23																	
24																	
25																	
26																	
27																	
28																	
29																	
30																	
31																	
MO SUM																	

Form BL51B

Revised 7/11/90

Figure 56

TEST TEMPERATURE SUMMARY

PROJECT No.:_____ CLIENT:_____

ACUTE TEST TEMPERATURE

	DATE								
AVG									
MAX									
MIN									
SUM. BY:									

CHRONIC TEST TEMPERATURE

	DATE								
AVG									
MAX									
MIN									
SUM. BY:									

COMMENTS:

Figure 57

48-HOUR ACUTE BIOASSAY SURVIVAL DATA

(4 Reps. 1-7 Treatments)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ AGE: _____ LOT No.: _____
SAMPLE DESCRIPTION: _____
SAMPLE No.(s): _____
LAB MEDIA/No.: _____ CONTROL/DILUENT: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLN	REP	FATALITIES PER EXPOSURE PERIOD (Hrs)		TOTAL FATAL.	MEAN SURV.	COMMENTS
			24	48			
1		A					
		B					
		C					
		D					
2		A					
		B					
		C					
		D					
3		A					
		B					
		C					
		D					
4		A					
		B					
		C					
		D					
5		A					
		B					
		C					
		D					
6		A					
		B					
		C					
		D					
7		A					
		B					
		C					
		D					
	DATE						
	DETERMINED BY						

Figure 58

48-HOUR ACUTE BIOASSAY SURVIVAL DATA
(2 Replicates)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ AGE: _____ LOT No.: _____
SAMPLE DESCRIPTION: _____
SAMPLE No.(s): _____
LAB MEDIA/No.: _____ CONTROL/DILUENT: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLUTION	REP	FATALITIES PER EXPOSURE PERIOD (Hrs)		TOTAL FATALITIES	MEAN SURVIVAL	COMMENTS
			24	48			
1		A					
		B					
2		A					
		B					
3		A					
		B					
4		A					
		B					
5		A					
		B					
6		A					
		B					
7		A					
		B					
	DATE						
	DETERMINED BY						

Figure 59

96-HOUR ACUTE BIOASSAY SURVIVAL DATA

(1 – 7 Treatments)

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ AGE: _____ LOT No.: _____

SAMPLE DESCRIPTION: _____

SAMPLE No.(s): _____

LAB MEDIA/No.: _____ CONTROL/DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLN	REP	FATALITIES PER EXPOSURE PERIOD (Hrs)				TOTAL FATALITIES	MEAN SURVIVAL
			24	48	72	96		
1		A						
		B						
2		A						
		B						
3		A						
		B						
4		A						
		B						
5		A						
		B						
6		A						
		B						
7		A						
		B						
	DATE							
	DETERMINED BY							

COMMENTS:

Figure 60

ACUTE BIOASSAY SURVIVAL DATA

(2 Reps, 1–7 Treatments, Optional Exposure Period)

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ AGE: _____ LOT No.: _____
SAMPLE DESCRIPTION: _____
SAMPLE No.(s): _____
LAB MEDIA/No.: _____ CONTROL/DILUENT: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLUTION	REP	FATALITIES PER EXPOSURE PERIOD (Hrs)					TOTAL FATAL.	MEAN SURVIVAL
1		A							
		B							
2		A							
		B							
3		A							
		B							
4		A							
		B							
5		A							
		B							
6		A							
		B							
7		A							
		B							
	DATE								
	DETERMINED BY								

COMMENTS:

Figure 61

ACUTE BIOASSAY SURVIVAL DATA

(4 Reps, 1–7 Treatments, Optional Exposure Period)

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ AGE: _____ LOT No.: _____

SAMPLE DESCRIPTION: _____

SAMPLE No.(s): _____

LAB MEDIA/No.: _____ CONTROL/DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLN	REP	FATALITIES PER EXPOSURE PERIOD (Hrs)					TOTAL FATAL.	MEAN SURV.	COMMENTS
1		A								
		B								
		C								
		D								
2		A								
		B								
		C								
		D								
3		A								
		B								
		C								
		D								
4		A								
		B								
		C								
		D								
5		A								
		B								
		C								
		D								
6		A								
		B								
		C								
		D								
7		A								
		B								
		C								
		D								
	DATE									
	DETERMINED BY									

Figure 62

CH2M HILL MILWAUKEE BIOASSAY LABORATORY

CLIENT:_____

TEST DATE:_____

To the best of our knowledge, the laboratory data reported, is true and accurate.

Report and Data:

Reviewed by:

_____Date:_____

_____Date:_____

Approved by:

_____Date:_____

RPTQA

Figure 63

CHRONIC TEST
INITIAL CHEMICAL DATA*

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLN	PARAMETER	EXPOSURE DAY 0	1	2	3	4	5	6	COMMENTS
1		DO								
		pH								
		COND								
2		DO								
		pH								
		COND								
3		DO								
		pH								
		COND								
4		DO								
		pH								
		COND								
5		DO								
		pH								
		COND								
6		DO								
		pH								
		COND								
7		DO								
		pH								
		COND								
	DATE									
	SAMPLE No.									
	DETERMINED BY									

*DO = DISSOLVED OXYGEN (mg/L) COND = CONDUCTIVITY (mmho)

Figure 64

CHRONIC TEST
FINAL CHEMICAL DATA*

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ LAB MEDIA /No.: _____
SAMPLE No.(s): _____ CONTROL/DILUENT: _____
SAMPLE DESCRIPTION: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLN	PARA-METER	EXPOSURE DAY 1	2	3	4	5	6	7
1		DO							
		pH							
2		DO							
		pH							
3		DO							
		pH							
4		DO							
		pH							
5		DO							
		pH							
6		DO							
		pH							
7		DO							
		pH							
	DATE								
	DETERMINED BY								

*DO = Dissolved Oxygen (mg/L) COND = Conductivity (mmho)

COMMENTS:

Figure 65

CHRONIC BIOASSAY SURVIVAL DATA

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ AGE: _____ LOT No.: _____

SAMPLE DESCRIPTION: _____

SAMPLE No.(s): _____

LAB MEDIA/No.: _____ CONTROL/DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____

TREAT. NO.	TEST SOLUTION	REP	NUMBER OF FATALITIES PER DAY 1	2	3	4	5	6	7	TOTAL NUMBER SURVIVORS	FATALITIES	% SURVIVAL	MEAN SURVIVAL	COMMENTS
1		A												
		B												
		C												
		D												
2		A												
		B												
		C												
		D												
3		A												
		B												
		C												
		D												
4		A												
		B												
		C												
		D												
5		A												
		B												
		C												
		D												
6		A												
		B												
		C												
		D												
7		A												
		B												
		C												
		D												
DATE														
DETERMINED BY														

	DAY	0	1	2	3	4	5	6
FEEDING	AM							
	PM							

Figure 66

CHRONIC BIOASSAY GROWTH DATA

PROJECT No.: _____ CLIENT: _____
TEST ORGANISM: _____ AGE: _____ LOT No.: _____
SAMPLE DESCRIPTION: _____
SAMPLE No.(s): _____
LAB MEDIA/No.: _____ CONTROL/DILUENT: _____
TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____
ANALYST(s): _____ CODE: _____

TEST SOLN	TREAT REP	TARE (mg)	TARE + DRY WT (mg)	TOTAL WT (mg)	Signif. Difference Test		IC25 Test	
					No. Surv.	Organism Wt.	No. Exposed	Organism Wt.
	1A							
	1B							
	1C							
	1D							
	2A							
	2B							
	2C							
	2D							
	3A							
	3B							
	3C							
	3D							
	4A							
	4B							
	4C							
	4D							
	5A							
	5B							
	5C							
	5D							
	6A							
	6B							
	6C							
	6D							
	7A							
	7B							
	7C							
	7D							

COMMENTS:

Mean 1 =		Mean 1 =	
Mean 2 =		Mean 2 =	
Mean 3 =		Mean 3 =	
Mean 4 =		Mean 4 =	
Mean 5 =		Mean 5 =	
Mean 6 =		Mean 6 =	
Mean 7 =		Mean 7 =	

Figure 67

CHRONIC BIOASSAY REPRODUCTION AND SURVIVAL DATA

PROJECT No.: _____ CLIENT: _____

TEST ORGANISM: _____ AGE: _____ LOT No.: _____

SAMPLE DESCRIPTION: _____

SAMPLE No.(s): _____

LAB MEDIA/No.: _____ CONTROL/DILUENT: _____

TEST START DATE: _____ TIME: _____ TEST END DATE: _____ TIME: _____

ANALYST(s): _____ CODE: _____ PAGE _____ OF _____

TREAT. NO.	TEST SOLN	DAY	OFFSPRING PER REPLICATE 1	2	3	4	5	6	7	8	9	10	COMMENTS
		1											
		2											
		3											
		4											
		5											
		6											
		7											

											SUMMARY TOTAL	SUMMARY MEAN
NO. of YOUNG												
NO. of BROODS												
ADULT FATALITIES												% SURVIVAL

TREAT. NO.	TEST SOLN	DAY	OFFSPRING PER REPLICATE 1	2	3	4	5	6	7	8	9	10	COMMENTS
		1											
		2											
		3											
		4											
		5											
		6											
		7											

											SUMMARY TOTAL	SUMMARY MEAN
NO. of YOUNG												
NO. of BROODS												
ADULT FATALITIES												% SURVIVAL

EXPOSURE DAY	0	1	2	3	4	5	6	7
DATE								
DETERMINED / FED BY								

G = GRAVID E = EYED R = RELEASING YOUNG AD = ADULT DEAD YD = YOUNG DEAD

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Figure 68

ALGAL BIOASSAY DATA SHEET

Project No. _____ **Client:** _____

Sample Description : _____ **Control/Diluent:** _____

Sample No.(s): _____

Test Organism: _____ **Age:** _____ **Lot No.:** _____

Test Start Date: _____ **Time:** _____ **Test End Date:** _____ **Time:** _____

Task Manager: _____ **Analyst:** _____

TEST INITIATION

TREAT. NO.	TEST CONC.	pH	COND
1			
2			
3			
4			
5			
6			
7			

CHEMISTRY*

	LAB CONTROL	100% EFFLUENT			
ALKALINITY					
HARDNESS					
TRC					
AMMONIA					

*COND = Conductivity (mmho) Total Alkalinity as mg/L CaCO3 Hardness as mg/L CaCO3
TRC = Total Residual Chlorine (mg/L) Total Ammonia (mg/L)

FILTRATION:	60 um screen	
	0.45 um filter	

TEST VOL / REP	
NO. of REPS	
NUTRIENT DOSAGE	

INOCULUM (Axenic Culture)	
# FIELDS	# CELLS
MEAN	

Formulas:

Culture Density = Mean No. Cells/Field x 250,000

Test Start Density*	x	Test Volume	/	Culture Density*	=	Inoculum Volume
0.01	x	100 mL	/	_____	=	_____ mL

*** Densities as 10^6**

TEST TERMINATION

PRESERVATIVE & DOSAGE _____

COLOR Green ____ Light Green ____ Dark Green ____ Other _____

APPEARANCE Clumping ___ Other _____

COMMENTS

CH2M HILL Milwaukee Bioassay Laboratory

Figure 69

ALGAE CELL COUNT DATA

Project No. ______________________ Client: ______________________

Sample Description: Effluent: ______________________ Control/Diluent: ______________

Sample No.(s): ______________________ Age: __________ Lot No.: ______________

Test Organism: __________ Time: __________ Test End Date: __________ Time: __________

Test Start Date: __________

Task Manager: ______________________

No./TEST SOLN		
REPLICATE A		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE B		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE C		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE A		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE B		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE C		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE A		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE B		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

No./TEST SOLN		
REPLICATE C		
	CELLS/ALIQUOT	
FIELD	1	2
1		
2		
3		
4		
5		
DET.BY		
TOTAL		
MEAN		

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Figure 70

ALGAE CELL COUNT WORKSHEET

Project No. ____________________ Client: ____________________

Sample Description: Effluent: ____________________ Control/Diluent: ____________

Sample No.(s): ____________________ Age: __________ Lot No.: __________

Test Organism: __________ Time: __________ Test End Date: __________ Time: __________

Test Start Date: __________

Task Manager: ____________________

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

No./TEST CONC.			
CELL COUNT		No. FIELDS	CELLS/mL*
REP A			
REP B			
REP C			
MEAN No. CELLS per mL*			

FORMULA: NUMBER OF CELLS / FIELD x CONVERSION FACTOR = NUMBER OF CELLS / mL

Where: Conversion Factor for Hemacytometer is 250,000
*Cells x 10^6

COMMENTS

CH2M HILL Milwaukee Bioassay Laboratory

Figure 71

LABORATORY UTILITIES LOG

DATE Mo.__ Yr.__	TEMP. SYSTEM CHECK			LAB WATER FLOW (LPM)		WATER SYSTEM CHECK	CULLIGAN FILTER CHANGE	WKLY WELL WATER USAGE(gal)	AIR SYSTEM CHECK	HOT WATER HEATER	COMMENTS	INTLS
	DATA LOGGERS	CONTROLLER 20	25	20 C	25 C							
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Form BL81

Revised 7/25/90

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Figure 72

DISSOLVED OXYGEN METER LOG

Meter No. _____

DATE Mo__Yr__	TEMP (C)	PRESSURE (mm Hg)	PRECAL READING % SATURATION	CAL READING % SATURATION	MEMBRANE/ ELECTROLYTE	MAINTENANCE/COMMENTS	INITIALS
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31							

Form BL47

REV.DATE 6/5/90

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Figure 73

PH METER LOG

METER NO._____ DATE CALIBRATED TO STANDARD_____

DATE Mo___Yr___	TEMP (C)	CALIBRATE BUFFERS	ELECTRODE STORAGE SOLN. RENEWAL	ELECTROLYTE RENEWAL	CALIBRATE BUFFERS RENEWED	MAINTENANCE/COMMENTS	INITIALS
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31							

FORM BL46 Revised 6/25/90

Figure 74

CONDUCTIVITY METER LOG

METER NO._____ DATE CALIBRATED TO QA STANDARD_____

DATE	TEMP (C)	CALIBRATE RESULTS* STD.1	STD. 5	CELL STORAGE SOLN. RENEWAL	MAINTENANCE/COMMENTS	INITIALS
*RANGE= 24–26 C		0.9–1.1	4.9–5.1 (mmho)			

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Figure 75

AMPEROMETRIC TITRATOR LOG (Total Residual Chlorine)

METER NO. ______

DATE	REAGENT RENEWAL			ELECTRODES CLEANED	MAINTENANCE/ COMMENTS	CALIB. to STANDARD	INITIALS
	TITRANT	PH 4	KI				

Figure 76

CH2M HILL Milwaukee Bioassay Laboratory

BALANCE CALIBRATION LOG

Mo.____Yr.____

DATE	AUTO-CAL	STANDARDIZATION		% Diff.	STATUS	SERVICED	INITIALS	COMMENTS
		Standard Wt.	Measured Wt.					
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								
31								

Figure 77

REVERSE OSMOSIS WATER SYSTEM LOG

DATE Mo___Yr___	MEG OHMS	INPUT COND.	OUTPUT COND.	H2O PRESS.	% REJECT.	MAINTENANCE/COMMENTS	INITIALS
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31							

Form BL49

COND. = mmho

(OUTPUT COND./INPUT COND.) x 100 = % Rejection

Figure 78

LABORATORY EQUIPMENT MAINTENANCE LOG

DATE	INSTRUMENT/EQUIPMENT	ID NUMBER	MAINTENANCE	SERVICED BY	INITIALS

Figure 79

CHEMICAL LOG

DATE REC'D	CHEMICAL	AMOUNT REC'D	ID NO.	EXP. DATE	INTENDED USE	STORAGE LOCATION	INITIALS