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Rev. 07/10/02

ROY F. WESTON, INC.

QUALITY ASSURANCE PROJECT PLAN

EVERGREEN MANOR
ROSCOE, ILLINOIS

WESTON
MANAGERS DESIGNERS CONSULTANTS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF
SR-6J

May 17, 2000

Kurt Fischer, Site Manager
Roy F. Weston, Inc.
3 Hawthorn Parkway, Suite 400
Vernon Hills, IL 60061-4055

Re: Approval of the
Quality Assurance Project Plan
Evergreen Manor Site
Roscoe, Illinois

Dear Mr. Fischer:

The United State Environmental Protection Agency has completed its review of the revised Quality Assurance Project Plan (QAPP) dated April 2000, for the Evergreen Manor Groundwater Contamination Site. U.S. EPA approves the revised QAPP. Attached is the U.S. EPA Field Services Section's approval memorandum and a completed signature page.

If you have any questions regarding this letter or the attached comments, please call me at (312) 886-4592.

Sincerely,

Mike Ribordy
Remedial Project Manager
Superfund Division

Enclosures

cc: Richard Byvik, SMF-4J

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF

MEMORANDUM **SMF-4J**

DATE: May 17, 2000

SUBJECT: Approval of the *First Revision* Quality Assurance Project Plan (QAPP) for the Fund-Lead Remedial Investigation/Feasibility Study (RI/FS) Activities for Evergreen Manor in Roscoe, Illinois

FROM: Richard L Byvik Richard L Byvik
Field Services Section (FSS)

TO: Mike Ribordy
Remedial Project Manager (RPM)

I am granting approval of the first revision QAPP for the Fund-Lead RI/FS activities for Evergreen Manor, Roscoe, Illinois. The subject QAPP was received by FSS on February 9, 2000, Log-in #2583. A conference call was held on February 17, 2000, discussing the use of Lockheed Martin/ESAT Mobile Laboratory for analysis. Lockheed Martin/EAST provided method detection limit studies on March 29, 2000, and the standard operating procedure on April 11, 2000. QAPP amendments were receive on May 2, 2000, and May 16, 2000. The Signature page has been signed and returned with the subject QAPP to the RPM. Please return a copy of the completely signed signature page to FSS.

Attachment

cc: Steve Ostrodka, SMF-4J
Bruce Sypniewski, SR-6J

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QUALITY ASSURANCE PROJECT PLAN

EVERGREEN MANOR
ROSCOE, ILLINOIS

April 2000

Prepared By: _____ Date: 4/25/2000
Tonya Balla, WESTON
Project Engineer

Approved By: _____ Date: _____
Kurt Fischer WESTON
Site Manager

Approved By: _____ Date: _____
Dean F. Geers, WESTON
Program Manager

Approved By: _____ Date: 5/17/00
Mike Ribordy, U.S. EPA
Remedial Project Manager

Approved By: _____ Date: 5/17/00
U.S. EPA Quality Assurance Reviewer

Roy F. Weston, Inc.
Suite 400
3 Hawthorn Parkway
Vernon Hills, Illinois 60061-1450
847-918-4000 • Fax 847-918-4055

25 April 2000

Mr. Mike Ribordy
Remedial Project Manager
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604

U.S. EPA Contract No.: 68-W7-0026
Work Assignment No.: 036-RICO-05MZ
Document Control No.: RFW036-2E-AFGO

Re: Quality Assurance Project Plan and Field Sampling Plan (Revision 2.0)
Evergreen Manor Site
Roscoe, Illinois

Dear Mr. Ribordy:

Enclosed please find three copies of the revised Quality Assurance Project Plan and Field Sampling Plan (QAPP/FSP), for the Evergreen Manor site. Revisions include U.S. EPA comments received in a letter dated 28 February 2000 and additional modifications for use of the U.S. EPA mobile laboratory. These modifications include the results of the method detection limit (MDL) study and final standard operating procedure (SOP) for mobile laboratory operations, which we received final approval to use on 19 April 2000. A response to comment section is also included behind the cover page for reference to changed portions of the document. If you have any questions, please contact me (847) 918-4016.

Very truly yours,

ROY F. WESTON, INC.

Kurt T. Fischer, P.G.
Site Manager

Enclosure

CH01\PUBLIC\WO\RAC\036\27580LTR.WPD RFW036-2E-AFGO

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QUALITY ASSURANCE PROJECT PLAN

EVERGREEN MANOR
ROSCOE, ILLINOIS

Prepared for:

U.S. Environmental Protection Agency
Region V
77 West Jackson Boulevard
Chicago, Illinois 60604

April 2000

This document was prepared in accordance with U.S. EPA Contract No. 68-W7-0026, WESTON Region V Response Action Contract (RAC) .

Work Assignment No. 036-RICO-05MZ Document Control No. RFW036-2E-AFGO

QUALITY ASSURANCE PROJECT PLAN

EVERGREEN MANOR
ROSCOE, ILLINOIS

April 2000

Prepared By: _____ Date: 4/25/2000
Tonya Balla, WESTON
Project Engineer

Approved By: _____ Date: _____
Kurt Fischer WESTON
Site Manager

Approved By: _____ Date: _____
Dean F. Geers, WESTON
Program Manager

Approved By: _____ Date: _____
Mike Ribordy, U.S. EPA
Remedial Project Manager

Approved By: _____ Date: _____
U.S. EPA Quality Assurance Reviewer

RESPONSE TO COMMENTS

A ESAT REGION V SOP FOR FIELD ANALYTICAL SUPPORT PROJECTS VOLATILE ORGANIC ANALYSIS FOR WATER, SOIL, AND SOIL GAS FASP-VOA-05

This SOP as presented would not be suitable for the project unless several project required compounds are added to the FASP target compound list (TCL) and the project required detection limits can be achieved. See QAPP Table 4-1. Perhaps, the ESAT Region V SOP **MS-FASP-SOP, FASP-MVOA-00** can meet the project required data quality objectives. The following compounds must be included in the FASP TCL

Chloromethane	Bromomethane	Vinyl Chloride
Chloroethane	Acetone	Carbon Disulfide
2-Butanone	Bromodifluoromethane	4-Methyl-2-Pentanone
2-Hexanone	1,2-Dibromoethane	Styrene
1,3-Dichlorobenzene	1,4-Dichlorobenzene	1,2-Dichlorobenzene

Response:
A new FASP SOP has been submitted. The project required compounds have been reconciled with the FASP SOP.

B QAPP Table 4-1

1) Verify if Bromodifluoromethane is a project parameter. This compound does not appear on the Method 8260B compound list. The compound may be **Dibromofluoromethane.**
1) Verify the project require detection limits, or the units, for Soil. The low soil quantitation limits for the 8260B Method are about 5 ug/kg. The units expressed here are around 1 mg/kg, or 200 times greater than the method can achieve. This remark was not made in the Draft QAPP comments.

Response:
Bromodifluoromethane has been deleted from the project parameter list. The list has been revised to reflect current ESAT project detection limits based on a March 2000 MDL study. The detection limit unit for soil has been changed from mg/kg to ug/kg.

APPENDIX B SAS REQUESTS

In this first revision QAPP **CHEMTECH SOPs** were not provided for the analysis of NITRITE-NITROGEN, NITROGEN-NITRATE, SULFATE, and ORTHOPHOSPHATE. **CHEMTECH** does not have to provide SOPs, if the SAS specified Analytical Protocols from the Method for the Chemical Analysis of Water and Wastes are followed, and the SAS QC REQUIREMENTS are fulfilled. However, **CHEMTECH** must provide any modifications made to these analytical

protocols.

Response:
The above stated SOPs were submitted in the initial draft QAPP. Modifications were not required as the initial differences were rectified in each SAS request. Chemtech will follow the SOPs.

APPENDIX C CHEMTECH LABORATORY SOPS

A SOP FOR THE DETERMINATION OF TOC IN SOIL AND SEDIMENT USING USEPA REGION II METHOD. DATED 7/27/88

This is not the Analytical Protocol that is specified in Section 7. of the SAS. Delete this SOP and use the SAS method. **CHEMTECH** must have a copy of the **"Methods of Soil Analysis", 2nd ed., 1982**. If not, provide **CHEMTECH** with a complete copy of the SOP.

Response:
An incorrect cover sheet was attached to the SOP and has been removed. The SOP follows method 9060 not a USEPA Region II Method as stated.

B VOLATILE ORGANICS BY GC/MS (METHOD 8260B) SOP #GCMSVOA2

1) The SOP provided in the draft QAPP was **VOLATILE ORGANIC COMPOUND ANALYSIS, Analytical Procedure No. MS8260**. This is not the same SOP. The draft QAPP Comment H (APPENDIX C **CHEMTECH** LABORATORY SOPS) was not addressed.
2) Several pages are missing from this SOP. namely. pages 4. 6. 11. 15. 17. and maybe pages 19 through 25. This SOP is not acceptable. and an adequate review of this SOP is not possible. due to the missing pages.
3) SAMPLE HANDLING AND PRESERVATION
Preservation requirements should be established for samples not analyzed within 48 hours.
4) GC/MS TUNING AND PERFORMANCE CHECK:
Describe the scan time and mass range of the MS.
5) GC/MS TUNING AND PERFORMANCE CHECK: pg 7
The calibration check compound Chlorobenzene. should be replaced with the 8260B Method specified **Ethylbenzene**.
6) PROCEDURE:
Describe in more detail the Initial Calibration and Continuing Calibration acceptance criteria, and corrective actions if the criteria are not satisfied.
7) PROCEDURE: pg 9
Please provide the referenced Table E .
8) PROCEDURE: pg 10

Please provide the referenced Table F.

9) DATA INTERPRETATION:
Express the calculations to determine the soil sediment sample concentration in ug/kg.

Response:
This SOP should be deleted from the QAPP. ESAT's mobile laboratory will conduct the soil/sediment VOC analysis. Approximately 10 percent of the soil/sediment samples will be sent to a U.S. EPA CLP lab for confirmation of the mobile laboratory results. Also, all volatile organic water samples will be preserved as stated in the QAPP to ensure comparability of results between the mobile lab and the fixed lab.

C COD - CHEMICAL OXYGEN DEMAND

QC REQUIREMENTS: Preparation Blank:
The SAS specifies 2 Titration Blanks and 2 Reflux (Reagent) Blanks, also.

Response:
The SAS has been modified to adequately reflect the method and laboratory SOP requirements.

D SULFIDE MCAWW Method 376.1 (Titrimetric, Iodine)

Section 8.1
The SAS specifies an Analytical Spike, and does not specify a reference sample, or LCS.

Response:
The SAS has been modified to adequately reflect the method and laboratory SOP requirements.

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A Field Sampling Plan

B SAS Requests

C Chemtech Laboratory SOPS

D SOPS for Field Instruments

E Guidance for Completing CLP/SAS Paperwork

F Mobile Laboratory SOP and MDL Study

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LIST OF ACRONYMS/ABBREVIATIONS

bgs	Below Ground Surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
CLP	Contract Laboratory Program
COC	chain-of-custody
CPT	cone penetration test
CRL	Central Regional Laboratory
CRQL	Contract-Required Quantitation Limits
DO	dissolved oxygen
DQO	Data Quality Objective
EE/CA	Engineering Evaluation/Cost Analysis Report
ESI	Expanded Site Inspection
FSM	Field Sample Manager/Custodian
FSP	field sampling plan
FSS	Field Services Section
FTL	field team leader
GC/MS	Gas Chromatograph/Mass Spectrometer
HRS	Hazard Ranking System
IATA	International Air Transport Association
IDPH	Illinois Department of Public Health
IDW	Investigation-Derived Waste
IEPA	Illinois Environmental Protection Agency
MCL	Maximum Containment Level
MSL	mean sea level
MS/MSD	matrix spike/matrix spike duplicate
NBS	National Bureau of Standards
NEIC	National Enforcement Investigations Center
NPL	National Priorities List
ORP	oxygen reduction potential
PE	performance evaluation
PID	photoionization detector
ppb	parts per billion
QA	quality assurance
QAM	quality assurance manager
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
QC	quality control
RAS	routine analytical services
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RPM	Remedial Project Manager
RSCC	Regional Sample Control Center

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LIST OF ACRONYMS/ABBREVIATIONS (Continued)

SAS	special analytical services
SHSC	Site Health and Safety Coordinator
SMC	sample management coordinator
SOP	standard operating procedure
SOW	Statement of Work
SSI	Site Screening Inspection
TCE	trichloroethene 1,1-dichloroethene (1.1-DCE
TCL	Target Compound List
TM	Technical Operations Manager
U.S. EPA	United States Environmental Protection Agency
USGS	United States Geological Survey
VOA	volatile organic analysis
VOC	volatile organic compound
WAM	Work Assignment Manager
WESTON	Roy F. Weston, Inc.
c-1,2-DCE	cis-1,2-dichloroethene
PCE	tetrachloroethane
1,1-DCA	1,1-dichloroethane
1,1-DCE	1,1-dichloroethene
1,1,1-TCA	1,1,1-trichloroethane
1,1,2-TCA	1,1,2-trichloroethane

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SECTION 1
INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by U.S. EPA participate in a centrally-managed quality assurance (QA) program. Any party generating data under this program has the responsibility to implement procedures to ensure that the precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is to perform.

This QAPP presents the organization, objectives, functional activities, and specific Quality Assurance and Quality Control (QA/QC) activities associated with a Remedial Investigation/Feasibility Study (RI/FS). The objective of this RI/FS is to collect site characterization data including soil, groundwater, surface water and sediment samples at the Evergreen Manor site in Roscoe, Illinois. This QAPP describes the specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody (COC), and laboratory and field analysis.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA, government regulations and guidelines, and specific project goals and requirements. This QAPP was prepared and amended by Roy F. Weston, Inc. (WESTON®) in accordance with all U.S. EPA QAPP guidance documents; in particular, the *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (QAMS-005/80), *Region V Content Requirements for QAPPs* (U.S. EPA, 1989), the *U.S. EPA Region V Model QAPP* (U.S. EPA, Revision 1, May 1996) and the *U.S. EPA Requirements for QAPPs for Environmental Data Operation* (EPA QA/R-5).

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SECTION 2
PROJECT DESCRIPTION

2.1 FACILITY LOCATION AND HISTORY/BACKGROUND INFORMATION

The Evergreen Manor site is located approximately 1.5 miles northwest of Roscoe, Winnebago County, Illinois. The site includes four residential subdivisions and has been defined by the extent of groundwater contamination. A site layout is provided in Figure 2-1. The site extends over Sections 16, 20, 21, 29, and 32 in Township 46 North, Range 2 East, and is found on the South Beloit, Illinois/Wisconsin Quadrangle. The coordinates of the site are latitude 42° 26' 32.0", longitude 89° 01' 36.0".

The site area was used as farmland prior to development. The Hononegah Heights subdivision was developed between 1940 and 1964; the Tresemer subdivision was developed between 1972 and 1974; the Olde Farm subdivision was developed between 1976 and 1979; and the Evergreen Manor subdivision was developed between 1986 and 1988. With the exception of the Evergreen Manor subdivision, most of the development occurred in the late 1970's and the early 1980's.

The Evergreen Manor site is bounded to the south by the Rock River. The Hononegah Forest preserve is located to the west of the site, some agricultural fields are located to the east of the site, and agricultural land is located north of the site (IEPA, 1992).

Kelley Sand and Gravel, a gravel pit and concrete mixing facility, is located approximately 0.5 miles to the northeast of the site, and an industrial park is located approximately 2 miles to the northeast of the site (IEPA, 1992).

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An industrial park is located approximately 2 miles from the site, north of Rockton Road, and east of Route 251. The industrial park contains the following businesses: Inlander-Steindler Paper Company, Regal-Beloit Corporation, McGuire Brothers Auto Body and Sand Blasting, Makerite Manufacturing Compnay, Midwest-Precision Grinding, Rockford Steam Boiler Works, Oscar's Auto and Battery Clinic, Dayles Welding, Armour Specialty, Inc. (industrial painting), RD Systems, Electro Cam Corporation, Area Elevator, DGM, Preston 151 (trucking firm), and Indicon Midwest (IEPA, 1992).

Several industries are located south of Rockton Road and east of Route 251. These include Ecolab and Taylor Design, Inc. Further south along Route 251 are six other facilities: State Line Foundries, Waste Management Transfer Station , Kenny's Cars, Trucks and Equipment, Stateline Printing Company, Roscoe Sand and Gravel, and Stateline Storage. Warner Brake and Clutch is located south of McCurry Road on the east side of Route 251 (IEPA, 1992).

Kelley Sand and Gravel is located approximately 0.5 miles northeast of the site, south of McCurry Road and west of Route 251. A concrete mixing facility, Roscoe ready mix, is located north of Kelley Sand and Gravel on the other side of McCurry Road (IEPA, 1992).

2.2 PAST DATA COLLECTION ACTIVITY/SITE HISTORY

The Evergreen Manor site was first realized in November 1990 when a lending institution required a homeowner to sample and analyze the drinking water. Results of the analyses indicated elevated concentrations of volatile organic compounds (VOCs). The Illinois Department of Public Health (IDPH) undertook further sampling in the area and identified a plume of contamination located beneath the Hononegah Heights and Evergreen Manor subdivisions (IEPA, 1992).

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The results of the IDPH sampling indicated that maximum concentration limits (MCLs) were exceeded for trichloroethene (TCE; MCL of 5 ppb), and 1,1-dichloroethene (1,1-DCE; MCL of 7 ppb) in one or more wells. Other VOCs identified included cis-1,2-dichloroethene (c-1,2-DCE), 1,1-dichloroethane (1,1-DCA), 1,1,1-trichloroethane (1,1,1-TCA), tetrachloroethane (PCE), and 1,1,2-trichloroethane (1,1,2-TCA). Based on these results, the IDPH concluded that at least 130 residences in the Hononegah, Olde Farm, Evergreen Manor, and possibly the Tresemer subdivisions could be contaminated with VOCs (IEPA, 1992).

The Evergreen Manor site was added to the Comprehensive Environmental Response, Compensation and Liability Information System (CERCLIS) on 3 August 1991, based on information received by the Illinois Environmental Protection Agency (IEPA) from the IDPH and the U.S. Environmental Protection Agency (U.S. EPA). The first Comprehensive Environmental Response and Liability Act (CERCLA) evaluation was a Preliminary Assessment (PA) of the Evergreen Manor in January 1992.

CERCLA Screening Site Inspection Report

A Site Screening Inspection (SSI) was performed in June and August 1992 to gather information for potential Hazard Ranking. A total of 39 soil gas samples and 4 groundwater samples were collected and analyzed for 1,1,1-TCA, TCE, and 1,1-DCE. Soil gas samples collected along McCurry Road and along North Second Street (Route 251 frontage road) indicated the presence of the three VOCs. The compounds were not detected on the north side of the Ecolab facility, or along the north side of Rockton Road.

The results of the groundwater analyses indicated that the VOCs were not detected in the samples collected on the north side of the Ecolab facility, or along the frontage road. The three VOCs were detected in a well north of the Waste Management facility.

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The SSI Report assigned a high priority to the Evergreen Manor site based on the results of the SSI and the groundwater samples collected from residential wells by IDPH between 1990 and December 1991.

CERCLA Expanded Site Inspection Report

The Expanded Site Inpection (ESI) was conducted in November 1993 and consisted of the collection of 49 groundwater samples from residential wells. The residential wells sampled are located in the Hononegah Heights, Olde Farm, and Evergreen Manor subdivisions. The purpose of the ESI was to collect information in support of the Hazard Ranking System package preparation.

A total of 49 groundwater samples were collected from 45 residences in the three subdivisions, four of which were duplicate samples. The samples were analyzed for VOCs. Results indicated that in all but one of the samples, and excluding two background samples, at least one VOC was detected. Acetone, 1,1-DCE, 1,1-DCA, 1,2-DCE (total), and PCE were detected at concentrations less than the laboratory detection limits. 1,1,1-TCA and TCE were detected at concentrations of less than 10 ppb to 37 and 40 ppb, respectively. Of the 45 wells sampled, 36 were found to have 1,1,1-TCA concentrations significantly above background, and 40 were found to have TCE concentrations significantly above background. All 40 TCE detections were at concentrations greater than the MCL.

Additionally, this report indicated that results of residential well sampling by IEPA in December 1993 and January 1994 found more that 60 residential wells with concentrations of 1,1,1-TCA and/or TCE above either the MCLs or the Cancer Risk.

Hazard Ranking System Documentation Record, 29 May 1997

Based on the information and data gathered from the PA, the SSI, the ESI, and other sampling by by IEPA and/or IDPH, a Hazard Ranking System (HRS) score was worked out. The Evergreen

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Manor Ground Water Contamination Plume, ILD 984836734, received a score of 100 points, for the groundwater pathway. The air, soil, and surface water pathways were not evaluated. The final HRS site score was 50 points.

Engineering Evaluation/Cost Analysis Report, October 1998

The Engineering Evaluation/Cost Analysis Report (EE/CA) was written with the objective of evaluating removal action objectives and removal action alternatives. Three viable alternatives were identified which would abate the threat posed by drinking the groundwater. These included a point-of-entry carbon filter treatment option, a point-of-use carbon filter treatment option, and an option to connect the affected residences to a public water supply system. The cost ranged from $154,000 for the point-of-use carbon filter option to $1,900,000 for connection to the public water supply option.

The EE/CA also summarized work that has occurred at the Evergreen Manor outside of the scope of the PA, SSI, and ESI:

- IEPA and IDPH sampled 267 drinking water wells, mostly in the four subdivisions, between December 1990 and March 1994. Results indicated that 108 wells exceeded MCLs and 203 were impacted.
- IEPA installed 24 monitoring wells between December 1993 and February 1995. Sample results from March 1994 indicated that 2 out of 20 wells exceeded MCLs for TCE and PCE. Sample results from February 1995 indicated that 3 out of 24 wells exceeded MCLs for TCE and four exceeded for PCE.
- Sample results from 12 wells sampled by U.S. EPA on 22 May 1998 indicated that six wells exceeded the MCLs for TCE and three wells exceeded the MCLs for PCE.

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Action Memorandum, 2 March 1999

This Action Memorandum served as a request for a non-time critical CERCLA removal action and consistency exemption to the $2 million and 12 month statutory limit at the Evergreen Manor site. The U.S. EPA recommended the extension of the public water supply system in order to provide the affected residences with safe drinking water. This decision was based on the permanence of the solution and the public response to the EE/CA during the public comment period, which opened on 10 November 1998.

The Evergreen Manor site contamination was estimated to affect 700 people in approximately 250 residences. A preliminary risk assessment indicated that the continued usage of residential wells would pose a threat to public health and the environment. Since the concentrations of TCE and PCE exceed MCLs, this condition represents an imminent and substantial endangerment to local residents.

The source area was identified in the Action Memorandum as the area at the intersection of Rockton Road and Route 251. Based on the information and data gathered, four Notice Letters were sent to Potentially Responsible Parties (PRPs) on 30 September 1996. All four PRPs declined to participate in the remediation of the Evergreen Manor site. [(IEPA, 1992) - CERCLA Screening Site Inspection Report].

2.3 SITE GEOLOGY

The geology and hydrogeology in the vicinity of Evergreen Manor have been most heavily influenced by fluvial and glacial processes. The preglacial Rock River incised a deep bedrock valley that was subsequently buried during glaciation. As the glacier retreated, vast deposits of sand and gravel with lesser amounts of silt and clay were deposited in the river valley. The site is located in the preglacial Rock River buried valley.

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The sand and gravel deposits have significant permeability and transmissivity and are the predominant local water source in the vicinity of the preglacial valley.

Overburden Geology

The surficial geology in the vicinity of the Evergreen Manor site consists of windblown sand and silt, lacustrine sand, silts and clays, and outwash sand and gravel deposited within the preglacial Rock River valley. Till deposits are found primarily along the valley margins. The valley was primarily filled with deposits from the Quaternary Period, during the Illinoian and Wisconsinan glacial events. The sand and gravel deposits are the most abundant and most extensive deposits in the buried valley, and can reach a thickness of up to 300 ft. in the vicinity of Evergreen Manor (HWRIC, 1986).

Bedrock Geology

The bedrock geology in the vicinity of the Evergreen Manor site is characterized by the Ordovician and Cambrian clastic and carbonate rocks. The Galena and Platteville dolomite, and the Ancell Groups represent the Orodovician Period in this area. The Ancell Group consists of two formations: the Glenwood, which is a sandy shale, and the St. Peter, which is predominantly a well-sorted sandstone up to 400 ft thick. The ancient Rock River eroded the Galena and Platteville dolomite, and the Glenwood, and carved its valley into the St. Peter sandstone (HWRIC, 1986).

The Cambrian rocks are dominated by sandstones with lesser thicknesses of shale and dolomite. The Potosi (dolomite) and Franconia (sandy shale) Formations separate the Ironton-Galesville sandstone from the Ordovician rocks. The Ironton-Galesville sandstone has a thickness of up to 170 ft. The Eau Claire Formation is up to 450 ft thick, and the Mt. Simon sandstone can be up to 1600 ft thick.

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The sedimentary bedrock units in the vicinity of the Evergreen Manor site were deposited on an irregular surface of metamorphic and igneous Precambrian rocks. Beneath the site, the Precambrian consists of a granite (HWRIC, 1986).

Hydrogeology

The unconsolidated outwash sand and gravel, the St. Peter, Ironton-Galesville, and Mt. Simon Formation sandstones are the aquifers underlying the site. Most privately owned wells draw groundwater from the outwash deposits. Larger wells owned or used by municipalities or developments draw groundwater from the bedrock aquifers.

The outwash sand and gravel is an unconfined aquifer with more uniform (i.e. better sorted) deposits at depth. Hydraulic conductivity in the outwash deposits has been estimated at $1x10^{-3}$ cm/sec (HRS Documentation Record). According to well logs, most of the private residential water wells are finished in the outwash sand and gravel deposits at depths of 50 to 80 ft below ground surface (bgs). The water table is approximately 35 ft bgs (USEPA ACTION MEMORANDUM).

The Galena and Platteville dolomite is an aquitards with a hydraulic conductivity estimated to range from $1x10^{-8}$ to $1x10^{-11}$ cm/sec (HRS Documentation Record). Flow in the dolomite is through vertically oriented fracture and joint systems. Although not used for high yield production wells the Galena and Platteville dolomite is used for small-demand, rural domestic and livestock water supply (HWRIC, 1986).

The Glenwood Formation, consisting of shale overlying a poorly sorted sandstone, has an estimated hydraulic conductivity of $1x10^{-4}$ to $1x10^{-7}$ cm/sec (HRS Documentation Record).

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The St. Peter sandstone underlies the Galena and Platteville, and Glenwood Formations, except along the axis of the Rock River buried valley, where the overlying bedrock has been removed by erosion. The St. Peter sandstone has an estimated hydraulic conductivity of 1×10^{-4} cm/sec and is widely used as a water source in Winnebago County (HRS Documentation Record).

The Ironton-Galesville Formation sandstone is a confined unit and a very productive aquifer. However, due to its depth few wells are finished in the Ironton-Galesville Formation (HWRIC, 1986). The same holds true for the Mt. Simon sandstone, which can reach a thickness of 1600 ft, and overlies the Precambrian granite.

2.4 PROJECT OBJECTIVES AND SCOPE

2.4.1 Introduction

The QAPP describes the policy, organization, functional activities, and QA/QC protocols necessary to obtain data of sufficient and known quality for use as intended in the RI/FS for the Evergreen Manor site. The objective of the QAPP is to establish standard procedures so that the integrity, accuracy, precision, completeness, and representativeness of the samples are maintained and the required objectives of the RI/FS are achieved.

2.4.2 Project Objectives

The objective of the RI/FS is to collect sufficient site characterization data, including soil, surface water, sediments, and groundwater sample data in order to support the selection of a site remedy.

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2.4.3 Specific Objectives

The objectives of the field investigations at Evergreen Manor are to determine the type and quantity of hazardous constituents present at the site, to determine their effect on human health and the environment, and to allow evaluation and selection of remedial alternatives.

2.4.4 Data Usage

The data necessary to attain the project objectives fall into the following categories:

- Source Characterization—To determine the nature and concentration of contaminants.
- Pathway Characterization—To determine the absence or presence of contaminants in surface water, groundwater, and sediments and to define the direction and rate of groundwater flow.
- Extent of Contamination—To determine the horizontal and vertical extent of contaminants at the Evergreen Manor site.
- Ecological and Human Health Risk Assessment—To determine the risk of site contaminants on human health and ecological receptors.

The following is a discussion of each category as it pertains to the field investigation, along with a discussion of the tasks to be performed for each category as it pertains to the data needed to meet each objective.

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2.4.4.1 Source Characterization

Analytical data from the field investigation sampling tasks will be evaluated to verify contaminated areas. The data will be evaluated to determine whether the site represents a contaminant source area. This phase of data evaluation will resolve the following issues:

- Whether residual contamination in the groundwater, surface water or sediment suggests past waste disposal activities.
- Whether the current levels of chemical contamination constitutes a source of soil, groundwater, or food chain contamination.
- The vertical distribution of chemical contaminants in the saturated zone underlying the site.

2.4.4.2 Pathway Characterization

Pathway characterization activities during the RI/FS will consist of surface water and sediment sampling; groundwater sampling; and groundwater and surface water elevation measurements. These efforts are designed to assess contaminant transport mechanisms through each of the potential contaminant transport pathways, and to evaluate the level of chemical contamination that may be associated with them. Primary environmental media targeted for pathway characterization are groundwater, surface waters, and sediments. Surface and subsurface soil samples may be collected in future investigation phases as deemed necessary.

Water level information collected from monitoring wells will be used to determine the potentiometric surfaces and directions of groundwater flow. Surface water elevation data will be used to evaluate surface water/groundwater interactions.

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2.4.4.3 Extent of Contamination

Groundwater samples will be collected to determine the vertical and lateral extent of contamination. Site-specific groundwater extent of contamination will be characterized by sampling groundwater at up to three discrete vertical intervals at 13 CPT boring locations, and with the installation and sampling of five monitoring wells. Residential well samples will also be collected to help characterize the extent of groundwater contamination.

Analytical measurements will be used to evaluate the extent of contamination in the groundwater pathway. Specific information to be obtained from the analytical and field monitoring results include:

- The level of contaminants present in groundwater.
- Subsurface (vertical and horizontal) extent of chemical contamination at the site.

2.5 SAMPLE NETWORK DESIGN AND RATIONALE

Section 2 of the Field Sampling Plan (FSP) (Appendix A) describes the sample network design and rationale for sample locations.

2.6 PARAMETERS TO BE TESTED AND FREQUENCY

Table 2-1, in the FSP (Appendix A) presents sample matrices, analytical parameters, and frequencies of sample collection. Groundwater and soil/sediment VOC analysis will be conducted through the Field Analytical Support Projects Mobile Lab. Confirmation groundwater and soil/sediment samples will be conducted through the U.S. EPA Contract Laboratory Program (CLP). All other sampling

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analyses for surface soils, subsurface soils, groundwater, surface water, and sediment will be performed under the Special Analytical Services (SAS) program. Due to the nature and type of contaminant, turnaround time, and detection limits, most sampling must be conducted as a SAS request. These additional parameters include total organic carbon, grain size, nitrate, nitrite, ammonia, orthophosphate, chemical oxygen demand (COD), sulfide and sulfate for groundwater samples.

2.7 DATA QUALITY OBJECTIVES (DQOs)

Data quality objectives (DQOs) are required for all environmental data collection activities. DQOs are statements of the quality of data needed to support a specific decision or an action. Data quality if defined in terms of the study objectives, rather than in terms of equipment or equipment analysis method characteristics. The DQOs must address the hypotheses that are to be proved or disproved and the necessary quality to support or defend the results obtained.

The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application.

DQOs are qualitative and quantitative statements derived from outputs of each step of the DQO process that:

- Clarify the study objective.
- Define the most appropriate type of data to collect.
- Determine the most appropriate conditions from which to collect the data.

The DQOs are then used to develop a scientific and resource-effective sampling design.

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The DQO process allow decision makers to define their data requirements and acceptable levels of decision during planning before any data are collected. DQOs are based on the seven step process described in EPA QA/G-4 (Sept. 1994) document.

The seven-step DQO process was used to establish DQOs. The seven steps are similar for each of the media of interest (soil, groundwater, sediment, and surface water), so the seven-step process only needed to be applied once.

1. STATE THE PROBLEM - a description of the problem(s) and specifications of available resources and relevant deadlines for the study

   Previous investigations at the Evergreen Manor site indicated that the groundwater at the site is contaminated with TCE and PCE above MCLs. The problem is to determine the extent of the groundwater contaminant plume and to assess the effectiveness of natural attenuation to reduce the contaminant impacts to groundwater. WESTON will be conducting the investigation. A project schedule is discussed in Section 2.8.

2. IDENTIFY THE DECISION - a statement of the decision that will use environmental data and the actions that could result from the decision.

   i. *Identify the principal study decision* - What is the extent of the groundwater contaminant plume as defined by the VOC MCLS, and is natural attenuation reducing contaminant impacts to the aquifer? The site must be further characterized to select a remedy to eliminate, reduce, or control risks to human health and the environment and to determine appropriate remedial alternatives.

   ii. *Define alternative actions that could result from resolution of the principle study question* -

      a. Natural attenuation may be chosen as a viable remedy for contamination found in groundwater at the site.

      b. Further study the extent of the groundwater contaminant plume if VOC concentrations exceed their respective MCLs and the natural attenuation evaluation shows that impacts are not reduced.

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iii. *Combine the principal study question and the alternative actions into a decision statement* - Decide whether natural attenuation is effective in reducing the contaminant impacts within the limits of VOC migration at the site or whether further study is warranted.

3. IDENTIFY INPUTS TO THE DECISION - a list of environmental variables or characteristics that will be measured and other information needed to resolve the decision statement.

i. *Identify the information that will be required to resolve the decision statement* - To resolve the decision statement, WESTON needs to obtain measurements of VOC concentrations (soil/sediments, monitoring well, residential well, and surface water), measurements of biological indicator parameters, and field parameters (pH, conductivity, temperature, DO, and ORP).

ii. *Determine the sources for each item of the information identified* - The groundwater, and surface water samples will be tested using CLP procedures for VOCs to determine if VOCs meet their respective MCLs and using the methods listed in Table 7-1 for biological indicator parameters to assess whether natural attenuation is effective in reducing contaminant impacts in the groundwater.

iii. *Identify the information that is needed to establish the action level* - The action level will be based on the VOC MCLs and on the weight of evidence for the constituents used to assess natural attenuation.

iv. *Confirm the appropriate measurement methods exist to provide the necessary data* - VOCs can be measured using CLP procedures. The detection limits are below VOC MCLs. The specified detection limits for other constituents are low enough to allow for natural attenuation evaluation.

4. DEFINE THE STUDY BOUNDARIES - a detailed description of the spatial and temporal boundaries of the problem, characteristics that define the populations of interest, and any practical considerations of interest

i. *Specify the characteristics that define the population of interest* - Monitoring well, residential well, surface water, and soil/sediment samples will be analyzed. Table 2-1 of the FSP details the number of samples. Figure 2-1 of the FSP indicates anticipated sample locations.

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ii. *Define the spatial boundary of the decision statement* -The study boundaries are based on the boundaries of the VOC groundwater plume and its anticipated pathway.

iii. *Define the temporal boundary of the decision statement* - It will be assumed that the RI/FS sampling date will represent current concentrations within the groundwater. Currently, only one round of sample collection is projected (December 1999).

iv. *Define the scale of decision making* - The scale of decision making will be the one sampling round.

v. *Identify practical constraints on data collection* - The most important practical consideration that could interfere with the study is interference in the ability to collect samples in the field due to inclement weather and lack of private property owners' approval to collect residential well samples. Laboratory holiday schedules could also alter the field work schedule.

5. DEVELOP A DECISION RULE - to define the parameter of interest, specify the action level and integrate previous DQO outputs into a single action statement that describes a logical basis for choosing among alternative actions.

i. *Specify the statistical parameter that characterizes the population of interest* - The laboratory results for the sampling event will characterize the population of interest. A statistical parameter is not being used because only one sampling event is scheduled; the small sample size would not result in meaningful sample statistics.

ii. *Specify the action level for the study* - The action level for the study will be the VOC MCLs. There are no specifiable action levels for the evaluation of natural attenuation. IEPA Tier II criteria will be used for the soil VOCs.

iii. *Develop a decision rule* - Analytical data will be used to evaluate the potenital risk to human health and the environment. If an unacceptable rist is present, the U.S. EPA will consider adding the site to the National Priorities List (NPL) triggering a continuation of CERCLA studies and implementation of remedial atlernatives.

6. SPECIFY LIMITS ON DECISION ERRORS - the decision maker's tolerable decision error rates based on a consideration of the consequences of making a decision error.

Data will be collected with the lowest level of uncertainty possible. All laboratory data will be reviewed for compliance with established methods and U.S. EPA guidelines for

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acceptability. Decision error has the most severe consequences near the action level because the risk of jeopardizing human health outweighs the consequences of paying for an extended groundwater investigation. Due to the small sample size, statistical methods cannot be used for data collection design.

7. OPTIMIZE THE DESIGN FOR OBTAINING DATA

Samples will be collected from each migration pathway that could be potentially impacted. Groundwater samples will be collected upgradient, sidegradient, and downgradient. Surface water and sediment will be collected from the Rock River and Dry Creek. Subsurface soil samples may be collected at a future date, if deemed necessary. Data will be collected using locations and methods that will provide quality data for characterizing the site with the lowest possible level of uncertainty. Following the initial sampling, a decision will be made whether it is necessary to expand the investigative activities by conducting an additional confirmatory groundwater sampling round and/or by installing additional monitoring wells.

2.8 PROJECT SCHEDULE

The overall schedule for the Evergreen Manor site activities is presented in Figure 2-2.

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ELEVATOR RD.

N

0 1200'

SCALE

E:
EMAP ADAPTED FROM FIGURE 1 OF THE CONTAMINANT SOURCE
LUATION – EVERGREEN MANOR SITE, DATED JANUARY 1997, BY
IESTOGA–ROVERS & ASSOCIATES.

FIGURE 2–1

NERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

SITE LAYOUT

EVERGREEN MANOR SITE
Roscoe, Illinois

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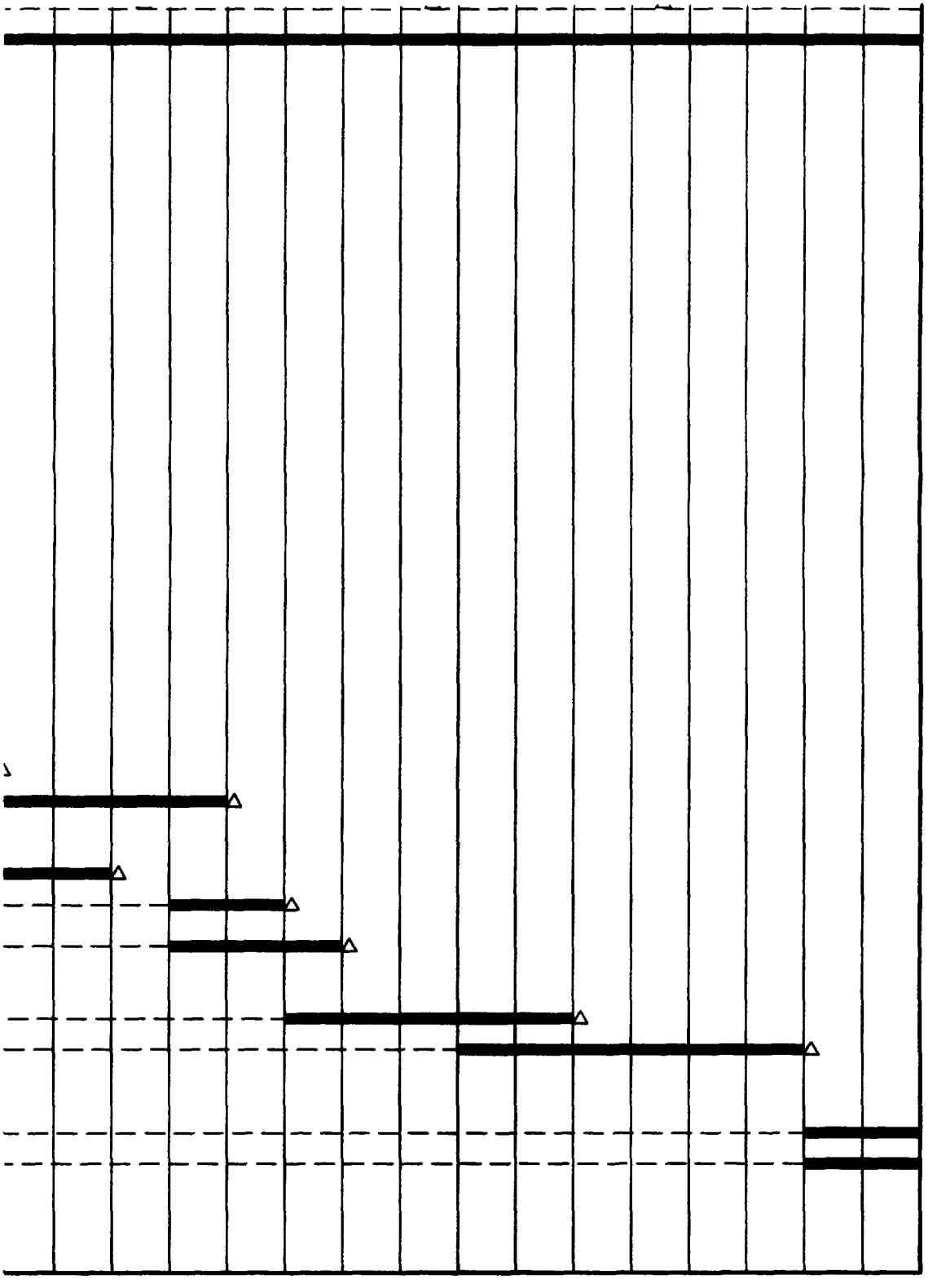

FIGURE 2-2

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

ESTIMATED PROJECT SCHEDULE

EVERGREEN MANOR

Roscoe, Illinois

SECTION 3
PROJECT ORGANIZATION AND RESPONSIBILITY

This section presents overall project organization and responsibilities, lines of communication, responsibilities for specific quality assurance tasks, and responsibilities for field and laboratory operations. The WESTON office location responsible for managing this project is the Vernon Hill, Illinois office.

Key personnel responsibilities in four specific areas (project management, QA, field operations, and laboratory operations) are discussed in the following subsections. Figure 3-1 presents the project organization chart.

3.1 PROJECT MANAGEMENT

This project is a federal-lead project, so the U.S. EPA Remedial Project Manager/Work Assignment Manager has overall responsibility for all phases of the RI/FS.

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

U.S. EPA Remedial Project Manager/Work Assignment Manager—Mr. Mike Ribordy is the U.S. EPA RPM/WAM for this project. Mr. Ribordy has overall responsibility for all phases of the Evergreen Manor RI/FS.

U.S. EPA Field Services Section (FSS) Quality Assurance Reviewer--The U.S. EPA Region V Superfund FSS will be responsible to review and approve all QAPPs.

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Illinois Department of Public Health--Mr. Rodger Ruden is the contact for the Illinois Department of Public Health (IDPH). He will be responsible for reviewing the project plans and data generated for this project.

WESTON Program Manager—Mr. Dean Geers is the WESTON Program Manager. The Program Manager has overall responsibility for the work assignment. The Program Manager is responsible for ensuring that the project meets all U.S. EPA and MDEQ objectives and quality standards. He is also responsible for ensuring that all work is executed in accordance with the U.S. EPA's technical directives. The WESTON Program Manager is responsible for assigning and monitoring the functions and responsibilities of the WESTON Site Manager (SM). In addition, he will commit the necessary resources and personnel to meet the objectives of site investigation.

WESTON Quality Assurance Manager (QAM)—Mr. Jon Olander, is the WESTON QAM. The WESTON QAM has the responsibility to implement and administer the WESTON Quality Assurance Program. He is responsible for coordinating all procedures and tasks pertaining to QA and reporting to the WESTON Program Manager on QA issues. Other duties include:

- Exercise overall responsibility for all audits under the RAC contract.
- Determine projects and activities to be audited.
- Establish audit schedules.
- Notify the audited entity of non-conformances and the need for corrective actions.
- Approve the disposition of non-conformances.

WESTON Technical Manager (TM)—Mr. James M. Burton, P.E., is the TM. The TM is responsible for overall technical review and input for projects. He is also responsible for providing staff for projects.

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WESTON Site Manager—Mr. Kurt Fischer, is the Site Manager. The Site Manager is responsible for implementing the project objectives utilizing the personnel assigned. The Site Managers primary function is to ensure that the technical, financial, and scheduling objectives are achieved successfully. The WESTON Site Manager will coordinate with the WESTON Program Manager, QAM, and TM, and will be the major point of contact and control for matters concerning the project. His other responsibilities include:

- Coordination and management of project personnel.
- Project scheduling.
- Coordination and review of required deliverables.
- General QA of field activities.
- Represent the project team at meetings and public hearings.

WESTON Human Health & Ecological Risk Assessment—Ms. Terry Bosko will perform the human health and ecological risk assessments. She will evaluate site data and assign associated risks.

3.2 QUALITY ASSURANCE

WESTON has procured Chemtech of Englewood, New Jersey to provide SAS analytical services. The ESAT Region V Mobile Laboratory will perform the water and soil/sediment VOC analysis. The U.S. EPA CLP will perform sample analysis on 10 percent of the water and soil VOC samples.

All aspects of the RI/FS at the site are subject to review by the WESTON QAM and approval by the U.S. EPA. Primary responsibility for all QC activities at the site is held by the WESTON site manager. The specific QA tasks and responsibilities are summarized below.

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3.2.1 Final Review/Approval of the Quality Assurance Project Plan

The U.S. EPA Region V Superfund Division, Field Services Section (FSS) Quality Assurance Reviewer reviews and approves all QAPPs.

3.2.2 Validation of Analytical Data

All CLP generated analytical chemical data will be validated by the U.S. EPA Region V Superfund Division, FSS. In addition, WESTON's data reviewers will conduct a systematic review of the data for compliance with established QC criteria. All SAS analytical data will be reviewed and validated by WESTON's data reviewers.

3.2.3 Performance and Systems Audits

Field Audits

- External field audits of Evergreen Manor site activities may be conducted by the U.S. EPA Region V. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the U.S. EPA Region V. External audits will be conducted according to the field activity information presented in the QAPP.

- Internal field audits are the primary responsibility of the WESTON QAM and Site Manager. These audits will verify that all established procedures are being followed. Internal field audits will be conducted at least once at the beginning of the site sample collection activities.

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Laboratory Audits

CLP Laboratories

Technical audits of the CLP laboratories and Region V ESAT laboratory are the responsibility of the U.S EPA Region V Technical Project Officer (TPO).

SAS Laboratories

Technical audits of SAS laboratories or other chemical laboratory are the responsibility of WESTON. These audits may include review of available laboratory analytical procedures, analysis of performance evaluation samples and, if necessary, an on-site system audit at the laboratory.

3.2.4 Scheduling of Special Analytical Services Analyses

WESTON's Sample Management Coordinator will coordinate all site sampling requirements and schedules with the U.S. EPA Region V Regional Sample Control Coordinator (RSCC). All SAS analyses are directly scheduled by WESTON's SMC with the WESTON procured laboratory.

3.2.5 Preparation of Special Analytical Services Requests

For any non-standard sampling methods or parameters a SAS request will be prepared by WESTON for approval by the U.S. EPA Region V FSS and U.S. EPA RPM/WAM. Each SAS request will contain specific methodology, analytical parameters, and detection limits. SAS requests have been prepared for the following parameters:

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Soil/Sediment:

- Volatile organics by FASP Method (mobile laboratory).
- Total Organic Carbon (TOC)
- Grain size

Water:

- Volatile organics by FASP Method (mobile laboratory).
- Ammonia
- Nitrate
- Nitrite
- Orthophosphate
- Sulfate
- Sulfide
- Chemical Oxygen Demand (COD)

A copy of the SAS requests are included in Appendix B.

3.2.6 Final Assessment of Quality Assurance Objectives

WESTON's QAM and Site Manager will jointly assess the validated data to determine whether the QA objectives have been met.

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3.2.7 Internal Quality Assurance Review and Approval of Reports, Standard Operating Procedures (SOPs), and Field Activities

Responsibilities for internal QA review and approval of reports, SOPs and field activities are as follows:

- The WESTON QAM and Site Manager are responsible for reviewing all necessary reports and procedures that can affect the data quality for planned site activities.
- The WESTON QAM or Site Manager are responsible for auditing the implementation of the QA program (as outlined in the QAPP) to ensure conformance with WESTON's and U.S. EPA's project requirements.
- The WESTON Field Team Leader (FTL) shall report the status of the field QA program to the WESTON QAM or Site Manager on a regular basis during field activities.
- The WESTON QAM and site manager shall provide QA technical assistance to the field and project staff during the QA plan's development and field implementation.

3.2.8 Evidence Audits of Field Records

Internal evidence audits of field records shall be the responsibility of the WESTON site manager and/or QAM.

External evidence audits of field records are the responsibility of U.S. EPA Region V.

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3.2.9 Approval of Special Analytical Services (Non-Contract Laboratory Program [CLP]) Laboratory Procedures

The U.S. EPA Region V RSCC and U.S. EPA Region V FSS must approve all SAS laboratory procedures. For the Evergreen Manor site, this pertains to analytical procedures used by the WESTON selected SAS laboratory (Chemtech) handling any chemical analysis.

3.3 FIELD OPERATIONS

The WESTON field team shall operate under the direction of the WESTON site manager. The field team's activities include monitoring well installation, sample collection, field measurements, sample packaging, sample shipment, and sample chain of custody (COC) preparation. Within the field team, there will be a minimum of three specific roles:

- **FTL**—Responsible for the management of the field team and the supervision of all field activities in the absence of the WESTON Site Manager.
- **Site Health and Safety Coordinator (SHSC)**—Responsible for the implementation of the Health and Safety Plan. Will perform health and safety monitoring and ensure compliance with all health and safety requirements for the Evergreen Manor site.
- **Field Sample Manager (FSM)**—Manages the custody of all samples from the time they are collected to when they are shipped. Is responsible for ensuring that all sample management and documentation procedures are implemented correctly.

For health and safety and QA reasons, a minimum of two field personnel will be present at all times during sampling activities. Depending on the schedule for the field sampling activity, the WESTON Site Manager will evaluate the need for additional personnel. When necessary, the FTL may also perform in the capacity of the SHSC. To the extent practicable, the FSM will not be given any additional responsibilities other than field samples. All personnel will be given the title of field

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sampler in order to encourage full utilization of all personnel at all times. The field samplers will collect samples and decontaminate equipment. In the absence of the WESTON Site Manager, the FTL will provide QA of field activities.

3.4 LABORATORY OPERATIONS

All soil/sediment and water VOC analysis will be conducted by the Region V ESAT mobile laboratory. Ten percent of the water and soil/sediment VOC samples will also be analyzed by a U.S. EPA CLP laboratory.

All other analyses for samples collected as part of the activities for the Evergreen Manor site shall be performed by a WESTON selected laboratory capable of SAS analysis. The WESTON SMC is responsible for initiating and scheduling the request for sample data validation with the U.S. EPA Region V RSCC. The WESTON SMC will coordinate with the WESTON Site Manager and FTL in executing all laboratory arrangements. The WESTON SMC solicits and awards contracts for all SAS requests. SAS requests may be reviewed by the U.S. EPA RSCC and U.S. EPA Region V Superfund FSS.

Chemtech

Chemtech located in Englewood, New Jersey has been procured by WESTON to analyze the SAS organic, inorganic, and water quality parameters. WESTON selected Chemtech to provide SAS analysis through a competitive bidding process and the successful analysis of performance evaluation samples for the parameters to be analyzed. Chemtech currently participates in the U.S. EPA CLP. An overview of the Chemtech laboratory organization chart is presented in Figure 3-2.

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Chemtech Laboratory Director- Mr. Divyajit Mehta is the is the laboratory director. Mr. Mehta is responsible for all overall technical performance of the laboratory. He is experienced in the analysis or organic/inorganic soil and water samples according to the requirements of the U.S. EPA Superfund CLP. He is responsible for the quality of the analyses and the validity of the data.

Chemtech Quality Assurance/Quality Control Officer (QA/QC) - Ms. Parveen Hasan supervises the quality of the data generated in the laboratory and the validation of the raw data generated by wet and instrumentation techniques in the inorganic and organic laboratories. She is responsible for reporting the final data in accordance with the requirements of the contracts and projects. The QA/QC officer also reviews program plans for consistency with organizational and contractual requirements and will advise appropriate personal of deficiencies. In addition, the QA/QC officer has the authority to stop work on projects if QC problems arist that affect the quality of the data produced.

Chemtech Organics Manager- Mr. Sam Bestawros is responsible for the supervision of the organic department. He is also in charge of sample flow, analysis, data review, and reporting the final results to the laboratory director.

Chemtech Inorganic/Wet Chemistry Manager - Mr. Mansukh Patel is responsible for the supervision of the inorganic department. He is also in charge of sample flow, analysis, data review, and reporting the final results to the laboratory director.

Chemtech Sample Custodian - Mr. Bob Morovati is responsible for logging in the samples on the computer, signing the Chain of Custody records and preparing the laboratory identification tags for each sample bottle. If the data on the paperwork does not correspond with the submitted

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information, he will inform the laboratory director. Mr. Morovati is in charge of the sample flow within the laboratory.

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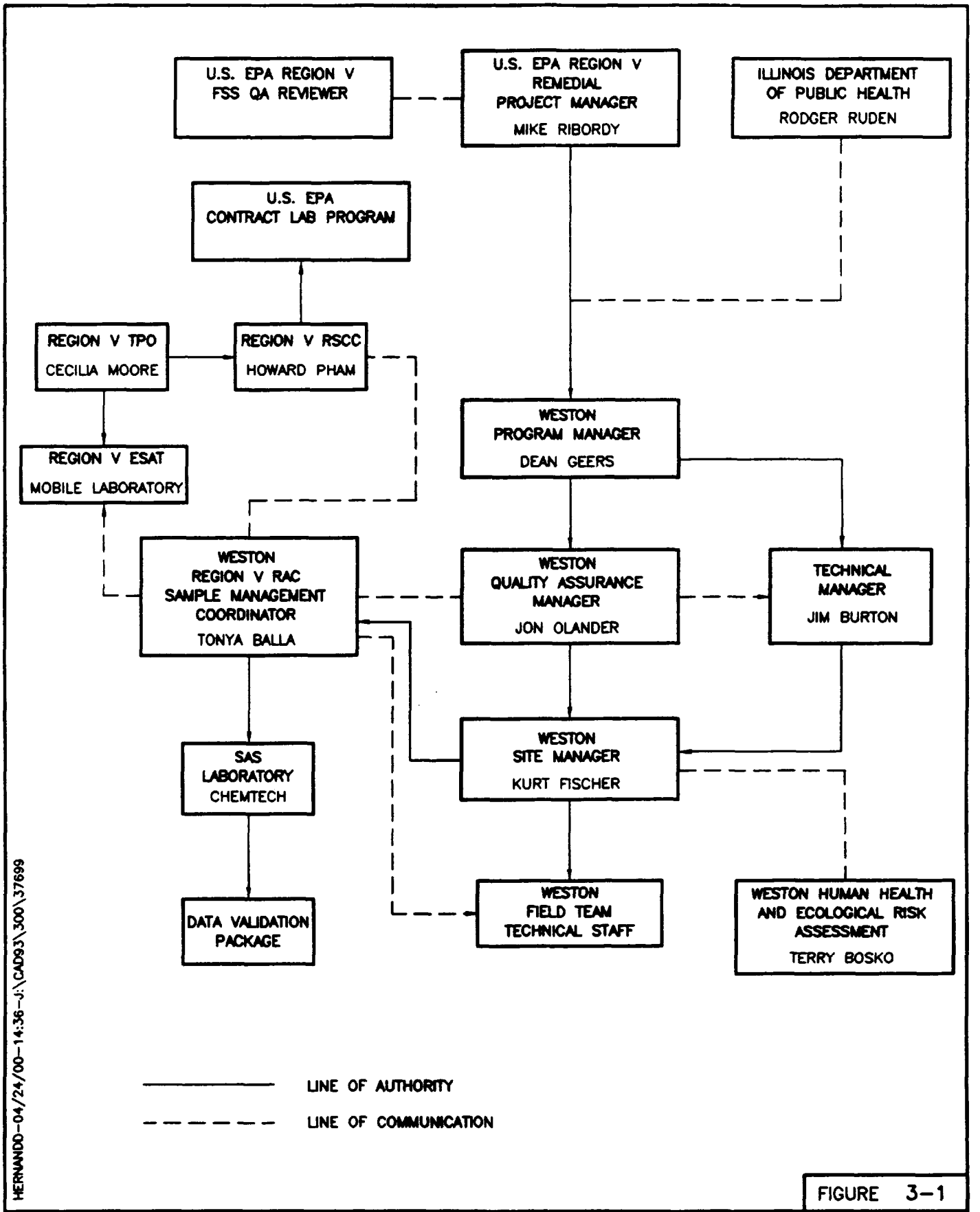

FIGURE 3-1

RESPONSE ACTION CONTRACT U.S. EPA CONTRACT No. 68-W7-0026 WORK ASSIGNMENT No. 036-RICO-05MZ DOCUMENT CONTROL No. RFW036-2E-AFGO	PROJECT ORGANIZATION CHART EVERGREEN MANOR Roscoe, Wisconsin

Chemtech
Laboratory Organizational Chart

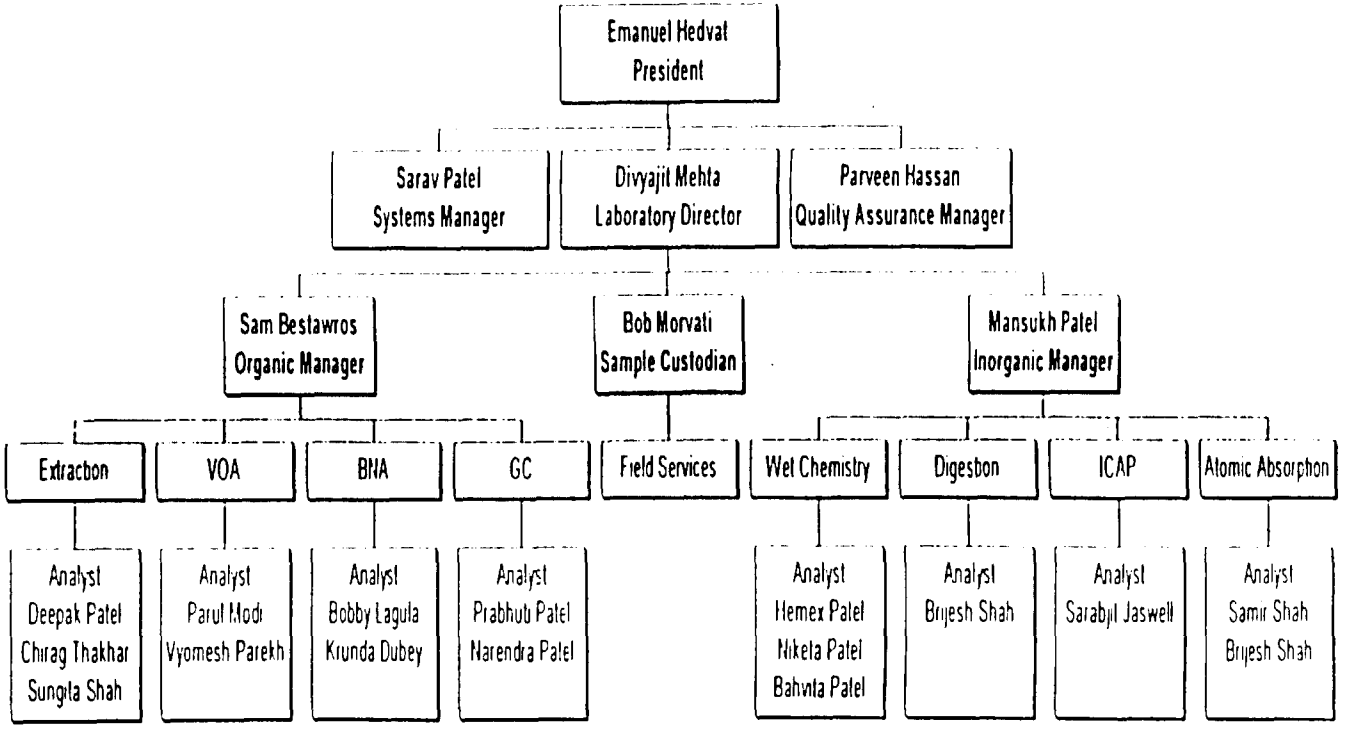

FIGURE 3-2

RESPONSE ACTION CONTRACT U.S. EPA CONTRACT No. 68-W7-0026 WORK ASSIGNMENT No. 036-RICO-05MZ DOCUMENT CONTROL No. RFW036-2E-ADKA	LABORATORY ORGANIZATION CHART EVERGREEN MANOR Roscoe, Illinois

SECTION 4

QUALITY ASSURANCE OBJECTIVE FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that will provide results which are legally defensible. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability of reported data from all analytical laboratories. QA objectives for field measurements are also discussed in this section.

4.1 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 blank and trip samples will be shipped to the analytical laboratory as blind samples. Field and trip blanks consisting of ultra pure water (laboratory grade) will be submitted to the analytical laboratories to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedures at the site that 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 provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are known as matrix spike/matrix spike duplicate (MS/MSD) samples. MS/MSD samples are designated and collected for organic analyses only.

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The general level of the QC effort will be one field duplicate and one field blank for every 10 or fewer investigative samples (i.e., a 10 percent frequency). However, field blanks will only be collected for water samples and will consist of ultra pure water (laboratory grade). No field blanks will be collected for soil or sediment samples because the U.S. EPA Region V discourages the use of water for soil/sediment field blanks. One volatile organic analysis (VOA) trip blank, consisting of ultra pure water (laboratory grade), will be included along with each shipment container of aqueous volatile organic compound (VOC) samples.

MS/MSD and spike/duplicate samples are investigative samples on which additional analyses are performed. One MS/MSD and spike/duplicate sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (e.g., soil, surface water, groundwater). Soil MS/MSD will require additional volume for the volatile fraction due to collection in the Encore® sampler. TOC does not require additional sample volume. Grain size samples with not have an MS/MSD. Aqueous MS/MSD samples must be collected at triple the volume for VOCs. Field blanks, trip blanks, and field duplicate samples will not be used as MS/MSD or spike/duplicate samples.

Accuracy and precision evaluations are also made by the use of performance evaluation (PE) samples. PE samples were sent directly to the WESTON-approved SAS laboratory for analysis as part of the SAS laboratory procurement process.

The specific level of field QC for samples collected as part of the RI/FS for the Evergreen Manor site is summarized by sample matrix and parameter in Table 2-1 of the FSP (Appendix A). Sampling procedures are specified in the FSP.

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4.2 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 and precision requirements for CLP Routine Analytical Services (RAS) are specified in the current Statement of Work (SOW)-OLC02.1 for volatiles organics or in the most recent revision. The sensitivities required for CLP Low Concentration RAS analyses (10 percent of total water samples) will be the Contract-Required Quantitation Limits (CRQL) for volatiles organics. The accuracy and precision requirements for soil volatile organics will use the correct SOW OLM04-2 for volatile organics or in the most recent revision. The water and soil/sediment volatile organic samples (excluding 10 percent analyzed for CLP) will be analyzed by the Region V ESAT mobile laboratory under a SAS request. The accuracy, precision, and sensitivity requirements for all SASs are specified in each individual SAS request in Appendix B. The project required detection limits are presented in Table 4-1.

Equipment to be used for field measurements for pH, conductivity, temperature, and ferrous iron is outlined in the SOPs in Appendix D. Accuracy and precision requirements for field screening analyses are provided in the FSP (Appendix A) and in the SOPs for the field instruments (Appendix D).

The accuracy of field measurements of pH will be assessed through pre-measurement calibrations and post-measurement verifications using at least two different buffer solutions. Each of the two measurements must be within ±0.05 standard unit of buffer solution values. Precision will be assessed with replicate measurements. (The electrode will be withdrawn, rinsed with deionized water, and re-immersed between each replicate.) The calibration and verification will be completed before the first replicate and after the last. The instrument used will be capable of providing measurements of ±0.1 standard unit.

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Table 4-1
Project Required Detection Limits
Mobile Laboratory (FASP Lab)
Evergreen Manor
Roscoe, Illinois

Parameter	Soil ug/kg	Water ug/L
Volatile Organic Compounds		
Chloromethane	5	1
Bromomethane	20	1
Vinyl Chloride	15	1
Chloroethane	10	1
Methylene chloride	10	1
Acetone	25	2
Carbon disulfide	5	1
1,1-Dichloroethene	5	1
1,2-Dichloroethane	5	1
cis-1,2-Dichloroethene	25	4
trans-1,2-dichloroethene	50	2
Chloroform	50	3
1,1-Dichloroethane	10	3
2-Butanone	25	11
Bromodichloromethane	5	1
1,1,1-Trichloroethane	25	2
Carbon tetrachloride	5	2
Dichlorodifluoromethane	5	1
1,2-Dichloropropane	5	1
cis-1,3,-Dichloropropene	5	1
Trichloroethene	5	1
Dibromochloromethane	5	1
1,1,2-Trichloroethane	5	1
Benzene	5	1
trans-1,3-Dichloropropene	5	1
Bromoform	5	1

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Table 4-1
Project Required Detection Limits
Mobile Laboratory (FASP Lab)
Evergreen Manor
Roscoe, Illinois
(Continued)

Parameter	Soil ug/kg	Water ug/L
Trichlorofluoromethane	5	1
1,1,2-Trichloro-1,2,2,trifluoromethane	5	2
Methyl acetate	15	1
Methyl tri-Butyl Ether	25	1
4-Methyl-2-pentanone	25	5
1,2-dichloroethene (total)	---	5
4-Methyl-2-pentanone	25	5
2-Hexanone	25	5
Tetrachloroethene	5	1
1,1,2,2-Tetrachloroethane	5	1
1,2-Dibromoethane	5	1
Toluene	5	1
Chlorobenzene	5	1
Ethylbenzene	5	1
Styrene	5	3
Xylene (total)	5	1
1,3-Dichlorobenzene	5	1
1,4-Dichlorobenzene	5	1
1,2-Dichlorobenzene	5	1

--- Compound not reported for this method.

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Table 4-2
Project Required Detection Limits
Contract Laboratory Program
Evergreen Manor
Roscoe, Illinois

Parameter	Soil ug/kg	Water ug/L
Volatile Organic Compounds		
Chloromethane	10	1
Bromomethane	10	1
Vinyl Chloride	10	1
Chloroethane	10	1
Methylene chloride	10	2
Acetone	10	5
Carbon disulfide	10	1
1,1-Dichloroethene	10	1
1,2-Dichloroethane	10	1
cis-1,2-Dichloroethene	---	4
trans-1,2-dichloroethene	---	2
Chloroform	10	3
1,1-Dichloroethane	10	3
2-Butanone	10	5
Bromodichloromethane	10	1
1... -Trichloroethane	10	1
Carbon tetrachloride	10	1
Dichlorodifluoromethane	---	---
1,2-Dichloropropane	10	1
cis-1,3.-Dichloropropene	10	1
Trichloroethene	10	1
Dibromochloromethane	10	1
1,1,2-Trichloroethane	10	1
Benzene	10	1
trans-1,3-Dichloropropene	10	1
Bromoform	10	1

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Table 4-2
State CLP Limits
Evergreen Manor
Roscoe, Illinois
(Continued)

Parameter	Soil ug/kg	Water ug/L
Trichlorofluoromethane	---	---
1,1,2-Trichloro-1,2,2,trifluoromethane	---	---
Methyl acetate	---	---
Methyl tri-Butyl Ether	---	---
4-Methyl-2-pentanone	10	5
1,2-dichloroethene (total)	10	---
4-Methyl-2-pentanone	10	5
2-Hexanone	10	5
Tetrachloroethene	10	1
1,1,2,2-Tetrachloroethane	10	1
1,2-Dibromoethane	---	1
Toluene	10	1
Chlorobenzene	10	1
Ethylbenzene	10	1
Styrene	10	1
Xylene (total)	10	1
1,3-Dichlorobenzene	10	1
1,4-Dichlorobenzene	10	1
1,2-Dichlorobenzene	10	1

--- Compound not reported for this method.

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Table 4-3
Project Required Detection Limits
SAS Laboratory
Evergreen Manor
Roscoe, Illinois

Parameter	Soil ug/kg	Water ug/L
Other Parameters		
Total Organic Carbon	0.10%	---
Chemical Oxygen Demand	---	5000
Nitrate	---	100
Nitrite	---	100
Ammonia	---	100
Sulfate	---	5000
Sulfide	---	1000
orthophosphate	---	50

--- Compound not reported for this method.

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Specific conductance will be measured by a conductivity meter with temperature compensation. The conductivity meter will be calibrated daily by insertion of the conductivity probe into a 1,000-micromhos standard solution before sample analysis. All field measurements will be to the nearest micromhos (±5 percent).

All monitoring wells will be surveyed for horizontal and vertical reference at the following locations: ground surface, marked reference point on inner stainless steel riser, and outer protective casing. The accuracy of the horizontal and vertical measurements will be ±1.0 foot and ±0.01 foot, respectively. Water levels will be referenced by elevation. The data collection will be referenced to the Illinois State Plane Coordinates.

4.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

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

$$Completeness = \frac{Number\ of\ valid\ data}{Number\ of\ samples\ collected\ for\ each\ parameter\ analyzed} \times 100\%$$

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was

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designed to provide data representative of conditions at the site. During development of this network, consideration was given to past waste disposal practices existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale of the sampling network is discussed in detail in the FSP (Appendix A). Representativeness will be satisfied by ensuring that the FSP is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will also be assessed by the analysis of field duplicate 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 QAPP, are expected to provide comparable data.

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SECTION 5
SAMPLING PROCEDURES

Sampling procedures are specified in the FSP, which is presented as Appendix A.

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SECTION 6
SAMPLE CUSTODY

6.1 INTRODUCTION

It is U.S. EPA's policy to follow U.S. EPA Region V sample custody, or COC protocols as described in *National Enforcement Investigations Center (NEIC) Policies and Procedures*, (U.S. EPA, 1985). This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

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

- In your possession.
- In your view, after being in your possession.
- In your possession, and you place it in a secured location.
- In a designated secure area.

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

WESTON will use the U.S. EPA's field sample custody procedures as required by the U.S. EPA Region V Superfund FSS and outlined in the U.S. EPA Region V CRL Sample Handling Manual and recent revisions. These procedures are outlined in the FSP (Appendix A).

The sample packaging and shipping procedures summarized below will ensure that samples arrive at the laboratory with the COC intact. The protocol for specific sample numbering using case

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numbers and traffic report numbers (if applicable) and other sample designations are included in the FSP (Appendix A).

6.2.1 Field Procedures

Field procedures are as follows:

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

(b) All bottles will be tagged with sample numbers and locations. The FSP defines the site-specific sample numbering system.

(c) Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions.

(d) WESTON's Site Manager (or his designee) will review all field activities to determine whether proper custody procedures were followed during the field work. He or she will notify the WESTON SMC and Site Manager of a breach or irregularity in COC procedures.

6.2.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed at the site. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound, consecutively numbered, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure area when not in use. Each logbook will be identified by the project-specific document number.

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The title page of each logbook will contain the following:

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

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, the level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigative team personnel and the purpose of their visit will also be recorded in the field logbooks.

Measurements taken and samples collected will be recorded. All entries will be made in ink (weather permitting) and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, as well as initialed and dated by the person making the correction. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station (including distance measurements) will be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to take measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the FSP (Appendix A). The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive a separate sample identification number, will be noted under sample description. The sample identification system is described in Section 5 of the FSP.

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6.2.3 Transfer of Custody and Shipment Procedures

Transfer of custody and shipment procedures are as follows:

(a) Samples are accompanied by a properly completed COC form. The sample numbers and locations will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, and to and from a secure storage area.

(b) Samples will be packaged for shipment and dispatched to the appropriate laboratory for analysis. A separate signed COC form will be enclosed in each sample shipment container. Shipping containers will be locked and secured with strapping tape and U.S. EPA custody seals for shipment to the laboratory. A custody seal will be attached to the front right and back left of the shipment container. The custody seals will be covered with clear plastic tape. The shipment container will be strapped shut with strapping tape in at least two locations.

(c) Whenever samples are split with a source or government agency, a separate sample receipt will be 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 will request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this will be noted in the "received by" space.

(d) All shipments will be accompanied by the COC form identifying the contents. The bottom two forms will be retained by the sampler for return to the sampling office, while the white and yellow copies will accompany the shipment.

(e) If the samples are sent by common carrier, a bill of lading will 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 will not be required to sign off on the custody form as long as the custody forms are sealed inside the sample shipment container and the custody seals remain intact.

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(f) Samples will be shipped in accordance with International Air Transport Association (IATA) requirements for air transport and in accordance with Department of Transportation requirements for ground transport.

6.2.4 Summary of Field Chain-of-Custody Procedures

The WESTON site field team will consist mainly of the following:

- The FTL.
- The SHSC.
- The FSM.

There will be a minimum of two people in each field team. All members will be considered to be field samplers and may be involved in the actual sample collection. Depending on the magnitude of the field operations, the WESTON Site Manager will evaluate the need for additional personnel. When necessary, the FTL will also perform in the capacity of the SHSC. To the extent practicable, the FSM will not be given any additional responsibilities other than sometimes performing as a field sampler. If more than two people are in the field team, there may be personnel who are designated as only field samplers.

The FTL will have overall responsibility for ensuring the completion of all field activities in accordance with the QAPP and FSP. The FTL is the overall coordinator of sampling activities at the site and is the communication link between field team members and the WESTON Site Manager. The FTL will assign specific field duties to the team members based on input from the WESTON Site Manager.

The WESTON SMC will give all required sample documentation to the FSM prior to leaving for the site.

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The FSM will be responsible for preparing (and reviewing for accuracy and completeness) all sample paperwork such as Organic CLP COCs, SAS/packing list forms, sample tags and any other paperwork required for sample documentation. The FSM will also prepare all sample shipment information such as airbills. If the FSM does not participate in sampling activities at a site, the FSM will have one or more of the field samplers sign the sample tags. If the FSM requests assistance from other members of the field team in completing sample paperwork, the FSM will be responsible for reviewing and ensuring the accuracy and completeness of this paperwork before he/she encloses it in the sample shipment container. All members of the field team may be involved in the actual sample packaging and shipment. The FSM is responsible for tracking all sample paperwork from the time of receipt until the completed paperwork is given to the WESTON SMC.

The FTL is responsible for maintaining the site logbook. The site logbook will contain notes made by the FTL on-site activities, including the tracking of the samples from the time of sample collection to the delivery of the samples to the shipping carrier. The names and function of all field team members will be listed in the logbook. During the course of sample collection activities, the FTL will document the times and dates of all sampling activities (e.g., who collected the samples, when and where the samples were collected, who delivered the samples to FSM, when the sample coolers were delivered to the shipping carrier) If the FSM was part of the sampling team this will be specifically noted. The FTL will note the names of the actual samplers for each station location along with the time, date, station location identifier and sample identifiers, etc.

The collected samples will be transported to the FSM by a member or members of the field team. If the sample locations are far apart, multiple samples may be collected prior to delivering them to the FSM. The FTL will ensure that any preservation requirements (e.g., keeping the samples cool) are implemented prior to the time that the samples are delivered to the FSM. To the extent practicable, the FSM will be in view of the sampling crew.

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Upon receipt of the samples, the FSM will be responsible for ensuring that custody is transferred. The FSM will require the field team member delivering the samples to sign and date the SAS packing list form associated with the samples as relinquisher of the samples in the "relinquished by" area. The FSM will then sign the forms as the recipient. The signed forms will be the same forms that will accompany the samples to the laboratory. Prior to enclosing the forms in the shipment container, the FSM will sign the SAS packing list to indicate he or she is relinquishing custody to the shipment carrier. If the forms are sealed in the shipment container with COC seals on the outside of the container, the shipment carrier will not sign the forms as the recipient. The FSM will be responsible for completing the remainder of all forms except as noted previously.
The FSM will sign sample tags if he or she participates in sampling activities. Otherwise, the FSM will have members of the sampling team sign the tags when they deliver samples. The team member delivering the samples will also provide the FSM with the individual time of collection for each sample. All sample documentation shipped with the sample to the laboratory will become part of the evidence file for the samples. The field logbook will be maintained in the site file or in the custody of the FTL.

The FSM assumes custody of the samples once the FSM has signed the COC forms. If the FSM must leave the "staging area" (where sample preparation for shipment and documentation completion is performed), the samples will either be locked inside of the sampling team's vehicle/trailer, or will be secured in a sample shipment container with custody seals. The custody seals will be inspected by the FSM upon return to the staging area to ensure they are intact. These practices will be followed whenever necessary to maintain custody of the samples in the field and will be logged into the site logbook. Example COC forms are provided in Appendix E.

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6.3 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

The COC procedures for the CLP are described in the SOWs for RAS; these same procedures apply to all SASs. These custody procedures, along with the holding time requirements are described in the appropriate SOW documents or SAS request. Chemtech laboratory SOPs are presented in Appendix C. Region V ESAT mobile laboratory SOPs are presented in Appendix F.

6.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

WESTON is the custodian of the evidence file and maintains the contents of the evidence files for the Evergreen Manor site activities. WESTON maintains all relevant records, reports, correspondence, logs, field notebooks, pictures, subcontractor reports, and the U.S. EPA Region V Superfund FSS data reviews in a secured, limited access area. WESTON will store the final evidence files in the Vernon Hills Office in a locking cabinet. The files will be kept for a minimum of 5 years or until the end of the contract. All files will be offered to the U.S. EPA prior to disposal.

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SECTION 7
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. For any activity that influences data quality, all instruments and equipment should be calibrated prior to each day's use or on a scheduled periodic basis.

7.1 FIELD INSTRUMENTS/EQUIPMENT

Instrument and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of results are consistent with the manufacturer's specifications. WESTON further requires that field instruments be calibrated and maintained by trained personnel.

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that any prior equipment problem is not overlooked and all necessary repairs to equipment have been made.

Calibration of field instruments is governed by the specific SOP for the applicable field analysis method, and such procedures take precedence over the following general discussion. Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service.

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Field instruments to be used during the Evergreen Manor site field investigation include:

- pH meter.
- Thermometer.
- Conductivity meter.
- Dissolved Oxygen meter.
- Oxygen reduction potential meter.
- Photoionization detector (PID).
- Water level indicator/electronic sounding device.
- HACH Colormetric Ferrous Iron Kit

The calibration and checkout of all field instruments will be performed prior to use each day. The calibration, checkout, and maintenance programs for each instrument are outlined in the respective SOPs presented in Appendix D, along with the procedures for field measurements.

All calibration performed in the field will be documented in the field logbook. A master calibration/maintenance file will be maintained by the WESTON FTL at the site office for each measuring instrument and will include at least the following information:

- Name of device or instrument calibrated.
- Device or instrument serial or identification (I.D.) number.
- Frequency of calibration.
- Results of calibration.
- Name of person performing the calibration.
- Identification of the calibration media (e.g., pH buffer solutions).

Tape measures used to locate sampling stations and to determine depths in boreholes or wells will be examined visually prior to each day of use to check for damage. Damaged tape measures will not be used.

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7.2 LABORATORY INSTRUMENTS

7.2.1 Calibration Procedures and Frequency for Special Analytical Services Analyses

The SAS calibration procedures and frequencies are specified in the various SAS requests. Those SAS analyses for organic parameters that will be performed according to RAS methodology will use the calibration and frequency protocols specified in the applicable CLP organic SOWs.

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SECTION 8
ANALYTICAL PROCEDURES

This section describes the analytical procedures for all analyses to be conducted for the RI/FS at the Evergreen Manor site. Analyses of soil, sediment, groundwater, and surface water samples will be performed by a WESTON procured SAS chemical laboratory that can generate CLP quality data. Table 2-1 in the FSP (Appendix A) identifies the field and laboratory parameters for each media to be sampled at Evergreen Manor site.

8.1 OFF-SITE LABORATORY ANALYTICAL SERVICES

8.1.1 Laboratory Procedures

Groundwater and soil/sediment samples will be analyzed for volatile organics via the Region V ESAT Mobile Laboratory Field Analytical Support Project (FASP) method. Ten percent of the groundwater and soil/sediment samples will be analyzed for organic parameters via the RAS low concentration organic SOW-OLC02.1 and SOW-OLM04.2, respectively (or most recent versions). Analysis will be conducted through the U.S. EPA CLP. The water quality parameters and other soil parameter methods are detailed in Table 8-1. All SAS analyses will be in accordance with the protocols outlined in the respective SAS requests in Appendix B.

8.2 FIELD SCREENING ANALYTICAL PROTOCOLS

The procedures for field measurement of pH, conductivity, temperature, DO, turbidity, ORP, and ferrous iron are described in the SOPs in Appendix D.

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Table 8-1
Analytical Methods
Evergreen Manor
Roscoe, Illinois

Parameter	Soil	Water
Volatile Organic Compounds (mobile laboratory)	Region V ESAT FASP Method	Region V ESAT FASP Method
Volatile Organic Compounds (10 percent)	OLM04.2	OLC02.1
Total Organic Carbon	415.2	---
Grain Size	ASTM D422	---
Chemical Oxygen Demand	---	410.1
Nitrate	---	352.1
Nitrite	---	354.1
Ammonia	---	350.2
Sulfate	---	375.4
Sulfide	---	376.1
Orthophosphate	---	365.2

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SECTION 9

INTERNAL QUALITY CONTROL CHECKS

This section describes the internal QC checks for field sample collection. field measurements. and laboratory analyses.

9.1 FIELD SAMPLE COLLECTION

The assessment of QC for field sampling will be made through the collection of field blank and field duplicate samples, in accordance with the applicable procedures and frequency described in Tables 2-1 of the FSP and Section 5 of the FSP.

9.2 FIELD MEASUREMENT

QC procedures for pH, conductivity, temperature, DO, ORP, and ferrous iron as well as manual and electronic measurement of water levels, are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard, and/or by calibrating the instruments (when appropriate). Assessment of field sampling precision and bias will be made through collection of field duplicates and field blanks in accordance with the applicable procedures described in the FSP at the frequency indicated in the Sampling and Analysis Summary .

9.3 LABORATORY ANALYSIS

9.3.1 Internal Quality Control Checks

QC checks for SAS are identified in the QC requirement section of the SAS requests in Appendix B. QC checks for the CLP RAS organics are identified in the corresponding CLP SOW.

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SECTION 10

DATA REDUCTION, VALIDATION, AND REPORTING

This section identifies responsibilities and procedures for data reduction, validation, and reporting for field measurements, sample collection and laboratory services.

10.1 FIELD MEASUREMENTS AND SAMPLE COLLECTION

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. If the data are to be used in the project reports, they will be reduced or summarized, and the method of reduction will be documented in the report.

10.2 LABORATORY SERVICES

10.2.1 Data Reduction

Data reduction, evaluation, and reporting for water VOCs will be performed in accordance with specifications outlined in the CLP SOW-OLC02.1 (for water) or CLP SOW-OLM04.2 (for soil). Data resulting from SAS analyses will be reduced, evaluated, and reported as described in the actual SAS request presented in Appendix B. Following data evaluation and reduction, the CLP-generated data will be sent to the U.S. EPA Region V Superfund FSS for data validation. All SAS generated data will be sent to WESTON for data validation.

10.2.2 Data Validation

The U.S. EPA Region V Superfund FSS is responsible for CLP data validation. Validation for CLP-generated data will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the CLP methods. Raw data such as chromatograms, mass

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spectra data reports, and data station printouts will be examined to ensure that reported results are accurate. The U.S. EPA Region V Superfund FSS will be responsible for ensuring the accuracy of the reports.

The U.S. EPA Region V validation protocols for the data are based on the following guideline:

- *U.S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* —U.S. EPA, October 1999.

WESTON will be responsible for SAS data validation. This includes the soil VOCs (if collected), TOC, grain size, and water quality parameters. Validation for SAS-generated data will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the SAS requests. Data will also be compared to the U.S. EPA Functional Guidelines or SW-846 protocols as applicable.

10.2.3 Data Reporting

The CLP analytical laboratories will prepare and submit full analytical and QC reports to U.S. EPA Region V Superfund FSS. The SAS laboratory (Chemtech) will prepare and submit full analytical and QC reports to WESTON. All data packages will include the following (as applicable):

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

2. Up to 20 extractable organic compounds not included in the RAS/SAS analytes, tentatively identified and quantified against the nearest internal standard.

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

4. Field and laboratory COC documentation pertaining to each sample delivery group analyzed.

10.3 Date Storage, Retrieval and Presentation

Analytical results, applicable QA/QC data, validation flags, and chain-of-custody information will be entered into a database for storage and query during data evaluation and RI report development. WESTON may enter these items into WESTON's total data management system (TDMS).

This system is a data management system used to manage the collection, entry, verification and reporting of information obtained in hazardous waste site investigations. TDMS is a comprehensive computer program utilizing IBM-compatible software. The general capabilities of TDMS include the following:

- Storage of analytical results.
- Storage of laboratory QC and data validation qualifiers.
- Automated loading of analytical data from WESTON of CLP-formatted electronic deliverables.
- Data entry and edit verification.
- Tracking of samples and analytical data throughout the process.
- Standard report formats as well as ad-hoc query capabilities.

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The benefits of using TDMS offers numerous advantages such as the following:

- Consolidated repository for all site data collected during a project, ultimately serving as the project archive.
- Organized, accurate, systematic, and verifiable method for meeting data management objectives.
- Rapid turnaround in data reporting; data stored in the TDMS repository can be reported in numerous ways, through the use of standard, predefined, reports, ad-hoc queries, on-line searches and views, etc., without duplicating data entry.

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SECTION 11

PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

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 FSP and the QAPP. The audits of field and laboratory activities include two separate, independent parts: internal and external audits.

QA system audits are conducted at least once during activities that may affect the integrity of the sampling program. The objectives of the systems audits are:

- To verify that a system of QC measures, procedures, reviews, and approvals is established for all activities that generate and process environmentally-related data.
- To verify that a system for project documentation (records, COC forms, analytical tags, logbooks, worksheets, etc.) is established.
- To verify documentation of the required QC reviews, approvals, and activity records (logbooks, worksheets, etc.).
- To identify nonconformance with the established system of QC measures, procedures, reviews, approvals, and documentation.
- To recommend corrective actions for identified nonconformance.
- To verify implementation of corrective action.
- To provide written reports of audits.

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11.1 FIELD AUDITS

Internal audits of field activities at the site will be the responsibility of the WESTON QAM and Site Manager. In the absence of both persons, the QA of field activities will be conducted by the designated FTL. Field audits will cover the following:

- Organization and responsibilities—To determine whether the QA organization is operational.
- Collection of samples—To ensure that written procedures are available and being followed.
- COC program—To ensure that the appropriate steps have been followed in the traceability of sample origin.
- Implementation of the operational procedures—To ensure that the appropriate QC checks are being made in the field and records are maintained of these checks.
- Equipment—To determine whether the specified equipment is available, calibrated, and in proper working order.
- Training—To ensure that sampling crews are adequately trained.
- Records—To ensure that record keeping procedures are operational and that field notebooks, log sheets, bench sheets, and tracking forms are properly prepared and maintained.
- Health and Safety Audit

These audits will occur at the outset of the project to verify that all established procedures are followed. Follow-up audits will be conducted to ensure correction of any deficiencies that were previously identified and to verify that QA procedures are maintained throughout the project.

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In addition, constant surveillance of field sampling and testing activities shall be performed by qualified technical personnel, as approved by the site manager. The Site Manager may conduct audits of site work procedures on an unscheduled basis.

External field audits may also be conducted by U.S. EPA Region V. External field audits may be conducted at any time during field operations. These audits may or may not be announced and are at the discretion of the U.S. EPA Region V. External field audits will be conducted according to the field activity information presented in the QAPP.

11.2 LABORATORY AUDITS

Audits of the SAS laboratories are the responsibility of WESTON. Audits of the CLP laboratories are the responsibility of U.S. EPA Region V.

The system audits will include the examination of laboratory documentation for sample receiving, sample log-in, sample storage, COC procedure, sample preparation and analysis, instrument operating records, etc. The performance audits will consist of sending PE samples to the SAS laboratories to evaluate laboratory precision and accuracy. PE results are currently being reviewed for compliance with limits established for the WESTON SAS procurement program. An on-site laboratory audit is tentatively scheduled for November1999.

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SECTION 12

PREVENTIVE MAINTENANCE PROCEDURES

This section describes the specific preventive maintenance procedures to be followed for field equipment and laboratory instruments.

12.1 FIELD EQUIPMENT/INSTRUMENTS

The field equipment for this project includes thermometers, pH meter, conductivity meter, ferrous iron test kit, and photoionization detector. Specific preventive maintenance procedures for this equipment are discussed in the SOPs in Appendix D, and will be conducted in accordance with manufacturer's specifications.

Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook. The FTL will be responsible for implementing and documenting these procedures in the logbook.

Preventive maintenance will normally be conducted by a WESTON Equipment Store representative. Additional maintenance of equipment will be performed at the site, if necessary, by the field sampling personnel on an as-needed or an as-recommended basis. In addition, critical spare parts, such as tape, pH probes, and batteries, will be kept on-site to reduce downtime. Backup instruments and equipment will be available on-site if deemed necessary, or will be within 1-day shipment to avoid delays in the field schedule.

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12.2 LABORATORY INSTRUMENTS

All laboratories participating in the CLP are required under the CLP RAS SOW OLC02.1 and SOW OLM04.2 (or most current) to have SOPs for preventative maintenance for each measurement system and required support activity. Preventive maintenance procedures for SAS laboratories performing CLP analysis are the same as those described above for RAS. All maintenance activities must be documented in logbooks to provide a history of maintenance records.

The preventive maintenance program for Chemtech will be in accordance with the laboratory's preventive maintenance procedures.

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SECTION 13

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

This section describes the specific routine procedures to be followed to assess data precision, accuracy, and completeness for field measurements and laboratory results.

13.1 FIELD MEASUREMENTS

Field data will be checked for compliance with the established QC criteria that are specified in the QAPP and FSP. Data from field measurements of pH, specific conductance, and temperature will be assessed by thorough review of QC data (e.g., calibrations, standards, blanks, replicates), documentation that analytical procedures were followed, and reports from system audits.

Accuracy of field measurements will be assessed using daily instrument calibration, calibration check, and analyses of blanks. Precision will be assessed on the basis of reproducibility by multiple readings of a single sample.

All data will be reviewed for completeness by the FTL or his designee. Data completeness will be calculated as follows:

$$Completeness\ (\%) = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} \times 100\%$$

13.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity as discussed in the following subsections.

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13.2.1 Precision

The degree of agreement between the numerical values of a set of duplicate samples performed in an identical fashion constitutes the precision of the measurement. Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD samples for organic analysis and laboratory duplicate analyses for inorganic analysis. Precision will be reported as a relative percent difference (RPD) and will be calculated for each pair of duplicate analysis as follows:

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100\%$$

Where:

% RPD	=	Relative percent difference.
S	=	First sample value (MS for organics).
D	=	Second sample value (MSD for organics).

13.2.2 Accuracy

Accuracy is the measure of a result to the accepted (or true) value. Accuracy of laboratory results will be assessed for compliance with the QC criteria that are described in Section 4 of the QAPP, using the analytical results of the method blanks, reagent/preparation blank, MS/MSD samples, field blanks, and trip blanks.

Analytical accuracy is expressed as the percent recovery of an analyte that has been added to the sample or standard matrix (i.e., blank) at a known concentration before analysis. The percent recovery of matrix spike samples will be calculated as follows:

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$$\%R = \frac{A - B}{C} \times 100\%$$

Where:

% R	=	Percent recovery
A	=	The total analyte concentration determined experimentally from the spiked sample.
B	=	The background level determined by separate analysis of the unspiked sample.
C	=	Amount of the spike added.

13.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. Data completeness for laboratory data will be calculated using the equation provided earlier in Section 13.1 for completeness of field data.

13.2.4 Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through various means including the analysis of method blank, calibration check sample, and laboratory control samples.

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SECTION 14
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 instrument 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 WESTON Site Manager or his designee if the problem occurs in the field, or the U.S. EPA Region V Superfund TPO if the problem occurs in a CLP laboratory. Problems with the WESTON procured SAS laboratory will be handled by WESTON with guidance from the U.S. EPA RPM/WAM and FSS, as applicable. Information on these problems will be promptly communicated to the U.S. EPA RPM/WAM, the WESTON Site Manager, or QAM. Implementation of corrective actions will be confirmed in writing through the same channels.

Any nonconformance with the established QC procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The WESTON QAM or his designee will issue a Nonconformance Report for each nonconformance condition.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. Corrective actions will be defined by the auditor and implemented to the satisfaction of the QAM. If corrective actions are insufficient, work may be stopped by a stop-work order issued by the U.S. EPA RPM/WAM, the WESTON Site Manager, or the WESTON QAM.

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14.1 SAMPLE COLLECTION/FIELD MEASUREMENTS

Technical staff and project field personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any activity or issued document by reporting the situation to the FTL or his designee. The FTL will be responsible for assessing the suspected problem, consulting with the WESTON Site Manager on the problem and anticipated change, and implementing the change. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the WESTON Site Manager.

The WESTON Site Manager will be responsible for informing the WESTON QAM and the U.S. EPA RPM/WAM of the problem. The WESTON Site Manager will be responsible for ensuring that corrective action for nonconformance is initiated by:

- Evaluating all reported nonconformance.
- Controlling additional work on nonconforming items.
- Determining disposition or action to be taken.
- Maintaining a log of nonconformance.
- Reviewing nonconformance reports and corrective actions to be taken.
- Ensuring nonconformance reports are included in the final site documentation in project files.

If appropriate, the WESTON Site Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

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Corrective action for field measurements may include:

- Repeating the measurement to check the error.
- Checking all proper adjustments for ambient conditions such as temperature.
- Checking instruments' batteries.
- Checking instruments' calibration.
- Re-calibration.
- Replacing the instrument or measurement device.
- Stopping work (if necessary).

All changes will be evaluated based on their potential to affect the quality of the data. The WESTON site manager has ultimate responsibility for all site activities. In this role, the Site Manager at times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible FTL notifies the WESTON Site Manager of the anticipated change and implements the necessary changes. The WESTON Site Manager or his designee must approve all changes verbally or in writing prior to field implementation by the FTL. The WESTON QAM and the U.S. EPA RPM/WAM will be notified when any field changes are made.

All problems and corrective actions will be documented in a field logbook by the FTL. No field team member will start corrective action without prior communication of findings through the proper channels. The action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken. If corrective actions are insufficient, work may be stopped by the FTL following instructions from the U.S. EPA RPM/WAM. WESTON Site Manager, or the WESTON QAM.

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14.2 LABORATORY ANALYSES

14.2.1 Laboratory Corrective Actions - CLP RAS

For the CLP RAS, 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.

The Sample Management Office also may request corrective action for any contractual nonconformance identified by audits or data validation. Corrective action may include:

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

If resampling is deemed necessary due to laboratory problems, the U.S. EPA must be notified and will help identify the necessary approach including cost recovery for the additional sampling effort. The U.S. EPA RPM/WAM must also recognize any delay in the project schedule as a result of the resampling effort. The WESTON QAM must be notified in writing of all decisions.

14.2.2 Laboratory Corrective Actions - SAS

For SAS Requests, corrective action will be implemented as specified in each SAS request (see Appendix B) and in accordance with the procedures described in the previous paragraph.

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SECTION 15

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The WESTON QAM or site manager (or his designee) will audit the implementation of this QAPP. These reviews will include an assessment of data quality, and the results of systems and performance audits as appropriate. These reviews are done to ensure that problems, if any, identified during the sampling and analysis are investigated, and corrective actions are taken properly. The preparation of a QA Report is not anticipated, except as necessitated by problems arising during the execution of project activities. Any QA report prepared by WESTON for the Evergreen Manor site will be submitted to the U.S. EPA RPM/WAM.

The RI/FS site characterization reports for the Evergreen Manor site will contain QA sections that summarize data quality information collected during the project.

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SECTION 16

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APPENDIX A

FIELD SAMPLING PLAN

APPENDIX A

FIELD SAMPLING PLAN
EVERGREEN MANOR SITE
ROSCOE, ILLINOIS

April 2000

Prepared for

U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604

This document was prepared in accordance with U.S. EPA Contract No. 68-W7-0026, WESTON Region V Response Action Contract (RAC) and contains confidential business information.

Document Control No. RFW036-2E-AFGO

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SECTION 1
INTRODUCTION

The field investigation for the Evergreen Manor site will seek to determine the type and quantity of hazardous constituents present at the site. The data collected during the field investigation will be used to provide technical support for the development and evaluation of remedial alternatives during the feasibility study (FS) process. The field investigation is composed of the following activities:

- Site reconnaissance
- Geological investigations
- Hydrogeological investigations
- Hydrological investigations
- Ecological investigations

This Field Sampling Plan (FSP) presents the details of field activities, discusses individual sampling rationale, and provides the field sampling procedures and protocols. Specifically, the FSP is organized as follows and addresses the following:

- Section 2—Sample Network Design and Rationale
- Section 3—Field Investigation Protocols
- Section 4—Field QC Samples
- Section 5—Sample Numbering System
- Section 6—Sample Documentation and Tracking
- Section 7—Sample Handling
- Section 8—Sample Team Organization
- Section 9—Management of Investigation-Derived Wastes
- Section 10—Sample Container Procurement

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SECTION 2

SAMPLE NETWORK DESIGN AND RATIONALE

The objectives of the field investigation at the Evergreen Manor site fall into the following categories:

- Source Characterization—To determine the nature and concentration of contaminants.
- Pathway Characterization—To determine the absence or presence of contaminants in groundwater, surface water, and sediments, and to define the direction and rate of groundwater flow.
- Extent of Contamination—To determine the horizontal and vertical extent of contaminants at the Evergreen Manor site.
- Ecological and Human Health Risk Assessment—To determine the risk of site contaminants on human health and ecological receptors.

The field activities are expected to generate sufficient data to support a risk assessment and FS. Table 2-1 presents a summary of the sampling and analysis program for the Evergreen Manor site. The field activities include an initial sampling program to delineate groundwater contamination, followed by a drilling program to install monitoring wells for subsequent sampling and verification. The following subsections present the rationale behind each field activity planned for the field investigation.

2.1 SITE RECONNAISSANCE

2.1.1 Well Inventory

An inventory of the residential and municipal wells in the vicinity of the site will be conducted to determine if residents near the site obtain their potable water from private wells. The areas to be covered include the Hononegah Heights, Olde Farm and Evergreen Manor residential subdivisions,

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and the stretch of Route 251 between McCurry Road and Rockton Road. The data collected for the well inventory will be used to identify potential receptors of groundwater contamination, if any. Data for this effort will be primarily compiled from previous reports and supplemented with additional information, if available, from state and local agencies.

2.1.2 Fracture Trace Analysis

As part of further detailed review of existing information, a high resolution fracture-trace analysis will be performed to identify potential contaminant migration pathways and target sampling locations that may have a higher potential of detecting contamination in overburden sediments. This study will be focused in the vicinity of Rockton Road and Route 251 where previous studies have not been able to draw conclusive evidence of a contaminant source and/or the upgradient extent of contamination. This specialized fracture trace activity will be performed at scales as low as 1:1000. Typical fracture trace is performed at a scale of 1:40000, which will only identify very large features.

Because the site is located overlying a pre-glacial bedrock valley, the presence of fractured bedrock at great depth is likely to have resulted in observable surface features. These surface features may facilitate the location of important structures, such as fracture networks or sand channels, which often act as preferential contaminant pathways. When located, these features will be targeted for geologic and hydrologic characterization using CPT methods described in subsequent sections.

2.2 GEOLOGICAL INVESTIGATIONS

2.2.1 Geologic Characterization

The geology underlying the Evergreen Manor site will be characterized at up to 8 locations using cone penetration testing (CPT) methods. CPT involves pushing a 2-inch diameter instrumented probe (penetrometer) through the subsurface while continually gathering geotechnical data. The soil end bearing resistance and pore water pressure response to penetration will be measured to provide stratigraphic and hydrogeological information. In addition, natural gamma measurements will be

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recorded to determine the presence of clay layers which may effect contaminant transport. This is of particular importance since a majority of the work will be performed in a residential area. Additionally, this method was chosen after a thorough review of available geologic information and discussions with previous investigators to evaluate its degree of successfulness in the geologic materials present and at the depths to be penetrated. CPT is an applicable approach for subsurface investigations where large cobbles, boulders, or dense gravel pavements are not present. Based on available geologic data, the subsurface at the Evergreen Manor site appears to consist predominantly of sand and gravel, without any significant documented obstructions. In the event that the CPT equipment consistently fails to penetrate to the desired depths, the U.S. EPA WAM will be contacted and alternate approaches discussed.

Potential CPT test locations are shown on Figure 2-1 and are placed in areas where previous geologic/hydrogeologic and groundwater chemistry data were not collected; or where existing wells are not sufficient to spatially evaluate the groundwater chemistry, or well logs were not located. CPT locations are preferentially located in areas along existing roadways to ease access and limit potential landscape damage to private property. Specific CPT locations for geologic characterization will be chosen in the field and will be based on the distribution of existing geologic information, plume delineation progress, and access restrictions.

2.2.2 Subsurface Soil Sampling

At this time, subsurface soil samples are not anticipated to be collected during this phase of the investigation. Previous studies have attempted to define a source of VOC contamination in the area with limited success. Groundwater VOC contamination extends from the Rock River upgradient to the vicinity of a small industrial area near the intersection of Rockton Road and Route 251. Several potential sources are located in this area which have been investigated previously to varying extent, with no specific source or residual source materials located. Taking this into account, combined with the apparent decrease in groundwater VOC concentrations over the years, indicates that a source or residual source may be difficult to determine, or not currently present.

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If, based on the results of this investigation, conditions are found to be different that summarized herein, additional site characterization activities (including soil sampling) may be required and will be conducted as a follow-up phase. In order to expedite this process, if needed, soil sampling procedures and QA/QC requirements are included in this FSP and associated QAPP as discussed in the following paragraphs.

Subsurface soil samples may be collected for analytical testing in support of source characterization and evaluation of natural attenuation characteristics. Soil borings will be advanced using a 4.25-inch hollow stem augers or an equivalent drilling method. Subsurface soil sampling will be conducted using a 2-foot split spoon sampler. The subsurface soil will be logged at regular intervals determined in the field. The sample cores will be screened using field equipment (e.g., photoionization detector [PID]).

At the completion of borehole sampling, the soil cuttings will be placed back in the borehole. Excess space will be filled with bentonite. If excess cuttings are realized, they will be placed in 55-gallon drums for characterization and disposal.

Soil samples, if collected, are anticipated to be analyzed for VOCs, TOC, and grain size distribution.

2.2.3 Sediment Sampling

Sediment samples will be collected from the Rock River and Dry Creek. Sediment samples will be collected to determine if site contaminants have been discharged, or have migrated into the river and creek. The proposed sediment sampling locations are shown in Figure 2-2. Sediment samples will be collected in areas that sediments are readily deposited and within approximately 5 feet of the shore. Three samples will be collected from the Rock River: one sample will be collected upriver of both the site and the confluence with Dry Creek; one sample will be collected upriver of the site, but down river of the confluence with Dry Creek; and one sample will be collected adjacent to the site. Three samples will also be collected from Dry Creek: one sample will be collected north of Rockton Road, upstream of the suspected source area; one sample will be collected approximately

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400 feet west of Route 251; and one sample will be collected approximately 0.25 miles to the southwest of Hononegah Road.

The 6 sediment samples will be analyzed for VOCs and TOC.

2.3 HYDROGEOLOGICAL INVESTIGATIONS

Groundwater quality and flow conditions will be assessed to define the areal and vertical extent of contaminant migration and to determine the concentrations of the contaminants in groundwater. Depth to water in the monitoring wells will be measured to determine the direction of groundwater flow, and hydraulic gradient. Residential wells and monitoring wells will be sampled to determine groundwater quality within the unconfined sand and gravel aquifer. Stratigraphic information obtained during drilling will be used to identify screened depths for proposed monitoring wells.

2.3.1 Residential Well Sampling

A total of ten residential wells have been identified and will be sampled at the site to determine the extent of contamination and the concentrations of contaminants in the aquifer. Figure 2-1 shows the proposed sampling locations, which are located along Straw Lane and Burnside Lane. The locations will be selected such that a reasonably spaced transect is achieved lateral to the expected path of the contaminated plume. Specific addressed will be selected after a survey of the area is completed. Initial contact and permission to sample the residences will be obtained by the Illinois Department of Public Health. If access to the above residences is not obtainable, attempts to obtain access from adjacent residences will be made and the sampling locations will be altered.

Each of the 10 residential well samples will be analyzed for VOCs. Six residential wells will also be analyzed for orthophosphate, ammonia, nitrate, nitrite, COD, sulfide, and sulfate parameters to evaluate natural attenuation characteristics across the lateral extent of the expected plume.

Residential wells will be purged and sampled in accordance with procedures specified in the QAPP and this Field Sampling Plan (FSP.)

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2.3.2 CPT Groundwater Sampling

Following evaluation of fracture trace and stratigraphic data at each of the 8 CPT test locations (discussed in Subsection 2.2.1), groundwater samples will be collected at up to 16 CPT locations to determine if groundwater has been impacted by contamination and to evaluate the lateral and vertical extent of the contamination. Sampling locations are shown on Figure 2-1.

Groundwater samples will be collected at each CPT test location at 10 foot intervals for a maximum total of 160 investigative groundwater samples. Groundwater sampling will commence at the water tables (approximately 30 feet bgs) and continue every 10 feet thereafter to a depth of 120 feet bgs.

Actual sampling depths may be modified in the field based on a review of the results of the CPT stratigraphic log. Potential contaminant pathways or preferential pathways (e.g., coarser horizons) will be targeted. Since well logs are not available for all existing monitoring Wells, field personnel will measure well depths of existing wells when performing CPT testing nearby. CPT groundwater sampling may be reduced based on these measurements. Groundwater samples will not be collected in zones where existing IEPA monitoring wells are directly adjacent to CPT test locations. In these areas, monitoring well sampling data is intended to fulfill the sampling objective.

The samples will be collected by advancing a stainless steel shielded well point and sample barrel to the desired depth at the end of the penetrometer string. The wellpoint can then be sampled by opening the shield and allowing groundwater to enter and then retrieving the sampler. This method is sufficient where only VOCs will be collected and limited volume is required.

Each of the CPT groundwater samples collected will be analyzed for VOCs. All CPT groundwater samples will be collected as unfiltered samples.

The VOC analytical results obtained from CPT groundwater sampling will also be used to evaluate the need and determine the depth at which to install permanent monitoring wells. This is discussed further in Subsection 2.3.3.

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2.3.3 Monitoring Well Installation

Up to five monitoring wells (2-inch diameter stainless steel) may be installed at the site in the aquifer to verify the results of the CPT groundwater sampling and serve as permanent monitoring points. Monitoring wells will be installed at locations where contamination was identified from the CPT groundwater sampling and where existing monitoring wells are not adequate to evaluate spatial distribution of contaminants. The actual location of monitoring wells will be determined from an evaluation of the CPT groundwater data and discussions with U.S. EPA. Samples collected from the monitoring wells will serve to confirm the CPT groundwater sample results, and will be used in long term monitoring of the site conditions.

Upon completion of well installation, each well will be developed. Monitoring wells will be constructed, installed, and developed in accordance with procedures specified in the QAPP and this FSP.

2.3.4 Monitoring Well Sampling (New and Existing Wells)

Each of the 5 newly installed monitoring wells at the site will be sampled to determine groundwater quality and aid in determining the extent of contamination. In addition to the newly installed monitoring wells, 8 existing monitoring wells previously installed at the site by IEPA will also be sampled. The locations of the 8 existing monitoring wells to be sampled is shown on Figure 2-1 and include G101D, G103, G104, G105, G108, G110, G112, and G113. Existing wells were selected such that various depths and locations within the previously delineated plume are selected. Because well logs are not available for all selected locations, field personnel will measure the depth of the selected wells and modifications to the number and locations of sampling may take place such that adequate spatial distribution is obtained.

Each of the monitoring well samples (maximum 13 samples) will be analyzed for VOCs. In addition, the 13 samples may also be analyzed for orthophosphate, ammonia, nitrate, nitrite, COD, sulfide, and sulfate parameters to evaluate natural attenuation characteristics. However, where new or

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existing monitoring wells are located near previously collected samples (CPT groundwater samples, residential wells, etc.), these natural attenuation parameters would be duplicative and will not be collected. Monitoring wells will be purged and sampled in accordance with procedures specified in the QAPP and this FSP.

2.3.5 Groundwater Elevation Measurements

The newly installed monitoring wells will be surveyed for vertical control using ground control equipment (transit, rod, etc.). Each monitoring well will be measured at three locations (ground surface, top of protective casing, and top of inner well casing). The depth to the water surface in each well will then be measured with a water level indicator. Horizontal control of wells will be referenced to easily identifiable structures, roadways, property lines, etc. The depth to the water surface will also be measured in all of the previously installed monitoring wells. Locations of existing monitoring wells will be verified. At this time, it is assumed that previous survey elevation data will be utilized for existing wells and re-surveying these points is not anticipated. The groundwater elevation data will be used to assess the groundwater flow direction in the sand and gravel aquifer.

2.3.6 Hydraulic Testing

Existing data from area testing will be used to the extent it is available and valid (to be determined by U.S. EPA). Additionally, hydraulic tests (slug tests) will be performed in the 5 newly installed monitoring wells after well development and sampling. This information will augment and verify existing data.

2.4 HYDROLOGICAL INVESTIGATIONS

2.4.1 Surface Water Sampling

Surface water samples will be collected from the Rock River and Dry Creek. Water samples will be collected to determine if site contaminants have been discharged, or have flowed into the river

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and creek. The proposed surface water sampling locations are shown in Figure 2-2. Surface water samples are anticipated to be collected within 5 feet of the shore and just below the water surface (i.e. 6 inches). Three samples will be collected from the Rock River: one sample will be collected upriver of both the site and the confluence with Dry Creek; one sample will be collected upriver of the site, but down river of the confluence with Dry Creek; and one sample will be collected adjacent to the site. Three samples will also be collected from Dry Creek: one sample will be collected north of Rockton Road, presumably upstream of the source area; one sample will be collected 400 feet west of Route 251; and one sample will be collected approximately 0.25 miles to the southwest of Hononegah Road.

The 6 surface water samples will be analyzed for VOCs.

2.4.2 Surface Water Elevation Measurements

The surface elevation of the Rock River and Dry Creek will be surveyed for vertical control using staff gauges and ground control equipment (transit, rod, etc.). One staff gauge will be installed in the Rock River and one staff gauge will be installed in Dry Creek. The elevation of the river and creek at the staff gauge locations will be measured using the ground control equipment, and will provide information regarding the elevation of the surface water with respect to the groundwater elevation.

2.5 ECOLOGICAL INVESTIGATIONS

Ecological investigation activities that will be conducted by WESTON personnel as part of the site characterization activities may include the following:

- Wetland and habitat delineation.
- Wildlife observations.
- Community characterization.
- Identification of endangered species.

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2.5.1 Wetland and Habitat Delineation

Appropriate research will be conducted to identify any wetland areas on or adjacent to the site. This will include review of National Wetland Inventory maps and potential site reconnaissance activities by a qualified wetland scientist.

2.5.2 Wildlife Observations

In conjunction with the reconnaissance survey for wetland identification and habitat delineation, qualified personnel will also make note of any wildlife observations at the site.

2.5.3 Community Characterization

A qualitative characterization of aquatic (e.g., fish, reptiles/amphibians, and benthic communities) and terrestrial (e.g., plant and mammal communities) communities present, or that could be present, on the site, will be made during reconnaissance surveys, and through a literature search.

2.5.4 Identification of Endangered Species

As part of the ecological assessment, information on endangered and/or threatened species known to exist on the site or in close proximity, will be requested from the Illinois Department of Conservation (IDOC) and the U.S. Fish and Wildlife Service.

2.6 WASTE DISPOSAL

Investigation derived waste, such as soil cuttings and purge water, generated during the investigation may be stored in 55-gallon D.O.T. approved drums. At the completion of the RI/FS investigations, the waste will be characterized for disposal acceptance. See Section 9 for specific procedures for management of investigative derived waste.

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Table 2-1

Summary of Sampling and Analysis Program for Evergreen Manor Roscoe, Illinois

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			MS/MSD[1]			Matrix Total*
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Cone Penetration Test (CPT) Groundwater Samples	conductivity, temperature, dissolved oxygen, pH, oxidation reduction potential	SAS Volatile Organics*	160	1	160	16	1	16	16	1	16	8	1	8	200
Monitoring Well	conductivity, temperature, dissolved oxygen, pH, oxidation reduction potential, turbidity	SAS Volatile Organics*	13	1	13	2	1	2	2	1	2	1	1	1	17
		orthophosphate	13	1	13	2	1	2	2	1	2	---	---	---	17
		ammonia	13	1	13	2	1	2	2	1	2	---	---	---	17
		nitrate	13	1	13	2	1	2	2	1	2	---	---	---	17
		nitrite	13	1	13	2	1	2	2	1	2	---	---	---	17
		chemical oxygen demand	13	1	13	2	1	2	2	1	2	---	---	---	17
		sulfide	13	1	13	2	1	2	2	1	2	---	---	---	17
		sulfate	13	1	13	2	1	2	2	1	2	---	---	---	17
Residential Well	conductivity, temperature, dissolved oxygen, pH, oxidation reduction potential, turbidity	SAS Volatile Organics*	10	1	10	1	1	1	1	1	1	1	1	1	12
		orthophosphate	6	1	6	1	1	1	1	1	1	---	---	---	8
		ammonia	6	1	6	1	1	1	1	1	1	---	---	---	8
		nitrate	6	1	6	1	1	1	1	1	1	---	---	---	8
		nitrite	6	1	6	1	1	1	1	1	1	---	---	---	8
		chemical oxygen demand	6	1	6	1	1	1	1	1	1	---	---	---	8
		sulfide	6	1	6	1	1	1	1	1	1	---	---	---	8
		sulfate	6	1	6	1	1	1	1	1	1	---	---	---	8
Surface Water	conductivity, temperature, dissolved oxygen, pH, oxidation reduction potential	SAS Volatile Organics*	6	1	6	1	1	1	1	1	1	1	1	1	8
Sediment	PID	SAS Volatile Organics*	6	1	6	1	1	1	1	1	1	1	1	1	8
		Total Organic Carbon	6	1	6	1	1	1	1	1	1	1	1	1	8

* Samples to be analyzed on site by U.S. EPA laboratory. Approximately 10 percent to be collected as additional duplicate samples for confirmation analysis at fixed laboratory.

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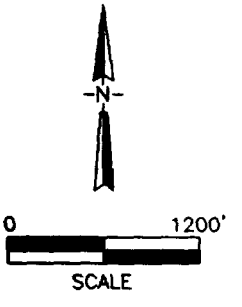
ELEVATOR RD.



:MAP ADAPTED FROM FIGURE 1 OF THE CONTAMINANT SOURCE
UATION – EVERGREEN MANOR SITE, DATED JANUARY 1997, BY
:STOGA-ROVERS & ASSOCIATES.

FIGURE 2-1

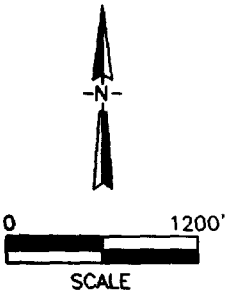
ERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

GROUNDWATER SAMPLING LOCATIONS
(CPT, RESIDENTIAL WELL, AND MONITORING WELLS)
EVERGREEN MANOR SITE
Roscoe, Illinois

HERNANDO-02/01/00-12:50-J:\CAD93\300\32499

ELEVATOR RD.



E:
EMAP ADAPTED FROM FIGURE 1 OF THE CONTAMINANT SOURCE
LUATION – EVERGREEN MANOR SITE, DATED JANUARY 1997, BY
IESTOGA-ROVERS & ASSOCIATES.

FIGURE 2-2

NERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

SEDIMENT AND SURFACE WATER
SAMPLING LOCATIONS
EVERGREEN MANOR SITE
Roscoe, Illinois

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SECTION 3
FIELD INVESTIGATION PROTOCOLS

The following sections detail the procedures that will be followed during the field investigation at the Evergreen Manor site. All sample container preservation and volume requirements are outlined in Section 7.

3.1 SITE RECONNAISSANCE

3.1.1 Well Inventory

The well inventory consists of a desktop survey and no field tasks associated with well inventory are anticipated.

3.2 GEOLOGICAL INVESTIGATIONS

3.2.1 Geologic Characterization

The geology underlying the Evergreen Manor site will be characterized by augmenting existing information with CPT testing at multiple locations as described in Subsection 2.2.1. This method involves pushing a 2-inch diameter instrumented probe (penetrometer) through the subsurface while continually gathering geotechnical data. The soil end bearing resistance and pore water pressure response to penetration will be measured to provide stratigraphic and hydrogeological. Natural gamma data will also be collected to evaluate the presence of clay layers and their potential effect on contaminant transport. Data is gathered as the instrumented probes are advanced at a constant rate of 4 feet per minute. Computerized CPT soil classification charts will be used in identifying and correlating site stratigraphy.

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CPT holes are abandoned by injecting a bentonite grout directly through the rod string as the CPT equipment is withdrawn.

The standard decontamination protocol for drilling equipment will be followed during this procedure and is presented in Subsection 3.7.

3.2.2 Subsurface Soil Sampling

Subsurface soil samples, if collected, will be obtained during drilling of soil borings. Subsurface soil samples will not be collected from CPT test locations. The following paragraphs outline the procedures that will be followed when advancing soil borings and collecting subsurface soil samples.

Soil Boring Drilling Procedures

Soil boring drilling may be required to evaluate site stratigraphy and collect subsurface soil samples. The need for borings, number of samples, and types of analyses for the soil boring samples will be determined based on the results of this investigation.

The drilling will be accomplished using a high-torque, 4.25-inch hollow-stem auger, or equivalent drill rig. A qualified drilling company will be subcontracted to drill the soil borings. All decontamination will be in accordance with Subsection 3.7. All work will be under the direction of a WESTON geologist. The following procedures will be used:

- Before drilling at any location, the driller will decontaminate the working end of the drill rig, all drilling equipment, and tools using a high-pressure steam cleaner. The driller will avoid placing equipment, tools, and materials on the ground during the drilling activities. The standard decontamination protocol for drilling equipment is presented in Subsection 3.7.

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- A high-torque drill rig using 4.25-inch inside diameter or larger, hollow stem augers will advance the borings to the desired depths. Other drill rigs may be substituted if necessary based on-site conditions.

- Each boring location will be sampled at regular intervals using a decontaminated 2-foot split-spoon sampler. The samplers and associated equipment that contacts the samples will be decontaminated between samples in accordance with the requirements outlined in Subsection 3.7. All sample cores will be logged by a geologist using the United Soil Classification System (USCS). All soil descriptions will be recorded on a boring log. An example of a boring log is shown in Figure 3-1. If the drill rig is visibly contaminated, the drill rig will be steam cleaned between drilling locations. All down hole drilling equipment will be decontaminated in accordance with the requirements of Subsection 3.7.

- Sample cores will be screened for volatile compounds using a photo-ionization detector (PID). Samples may be collected for laboratory analysis as described in the following subsection.

- Borings will continue until a predetermined depth is reached. This depth will be determined from the results of the CPT investigation.

- In the event that a soil boring must be abandoned, the soil boring will be abandoned by pouring 6 inches of a bentonite chip water slurry in the bottom of the hole, returning as much of the soil removed from the boring as possible to the hole, and adding a 6-inch bentonite cap to the top of the hole. Any cuttings remaining will be placed in a 55-gallon drum and managed according to the requirements specified in Section 9.

Subsurface Soil Sampling Procedure

The soil samples will be collected using a decontaminated 2-foot split-spoon sampler. The need for samples, number of samples, the types of analysis, will be determined based on the results of this investigation. The samplers and associated equipment that contacts the samples will be decontaminated between samples in accordance with the requirements outlined in Subsection 3.8. Following removal from the borehole, the continuous or split-spoon sampler will be opened on a clean surface (e.g., polyethylene sheeting).

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Sample intervals retained for laboratory analysis will be collected according to the following protocol. For VOC samples, sample collection will commence immediately upon opening the split-spoon sampler in order to minimize the loss of VOCs, if present. The VOC samples will be collected first as a grab sample. Sample material from the undisturbed sample interval will be collected in three 5-gram Encore samplers (additional volume is required for MS/MSD analysis). Samples will be collected using the Encore Stainless steel sampler designed for this application. No mixing or compositing will be performed on the sample material, thereby limiting the loss of VOCs from the sample. Duplicate samples will be collected as close to the original sample in the interval as soon as possible. The remainder of the sample material will be homogenized. Additional sample volume will be collected and submitted for percent dry weight analysis.

The homogenizing procedure is designed to increase the probability that a relatively small sample aliquot is representative of a relatively large soil volume removed from a sample location, thereby enhancing the representativeness and reproducibility of the sample. Sample material will be placed in a decontaminated stainless steel bowl or tray, and a decontaminated stainless steel spoon or spatula will be used to break up the material into pieces approximately 1/2 inch or less in diameter. The sample material will then be mixed using decontaminated spoons or spatulas. This action will be repeated at least three times. The homogenizing process will be considered complete when the texture and color of the sample appear uniform throughout. Upon completion of the homogenization process, samples can be collected for laboratory analysis. These samples will be considered grab samples. Sample containers will be filled in a sequence such that more volatile suites of parameters are collected first followed by inorganics, etc.

The homogenization procedure will be followed for soil samples, regardless of appearance, in order to ensure consistency, unless stated elsewhere in this document. All reusable sampling equipment, including the stainless steel spatulas, split spoons, spoons, bowls, will be decontaminated between each sample in accordance with procedures outlined in Subsection 3.7.

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3.2.3 Sediment Sampling

The proposed sediment sampling locations, the number of samples, and the type of analysis for sediment samples is presented in Subsection 2.2.3.

To obtain the sediment samples from the Rock River and Dry Creek, a spring-loaded Eckman dredge, Ponar grab sampler, bucket auger, or other suitable device will be used. The samplers will be attached to a rope, as applicable. The rope will be used to lower the device into the water until it contacts the bottom of the river. Upon contact, the sampler will automatically close, and sediment is trapped within the sampler. The sample will then be raised via the rope. Sediment sampling will be performed from the shore (i.e., no boats). Sediment sampling will always be executed in sequence starting with the first sample collected at the farthest downstream location with subsequent sampling progressing upstream. This will minimize cross contamination of locations due to disturbed sediment. For sediment samples near suspected outfalls, sediment samples will be collected using a decontaminated stainless steel spatula, spoon, or scoop. Depending on the site conditions, (i.e., site outfalls) the Field Team Leader (FTL) will determine the most appropriate sediment sampling approach to implement at the site.

After each sample is retrieved, the sediment sample will be opened onto a clean surface (e.g., polyethylene sheeting or a stainless steel bowl) The VOC samples will be collected in three 5-gram Encore samplers (additional volume is required for MS/MSD analysis). Samples will be collected using the Encore stainless steel sampler designed for this application. No mixing or compositing will be performed on the sample material, thereby limiting the loss of VOCs from the sample. Duplicate samples will be collected as soon as possible. Additional sample volume will be collected and submitted for percent dry weight analysis.

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All reusable sampling equipment, including the stainless steel spatulas, split spoons, spoons, bowls, will be decontaminated between each sample in accordance with procedures outlined in Subsection 3.7.

3.3 HYDROGEOLOGICAL INVESTIGATIONS

Hydrogeologic investigation activities include collection of residential well samples, CPT groundwater samples, newly installed and existing monitoring well samples, and surface water samples. Methods and procedures for these activities are discussed in the following subsections.

3.3.1 Residential Well Sampling

The 10 residences to be sampled, and the type of analysis for residential well samples is presented in Subsection 2.3.1. The IDPH is expected to garner access to the residential well sampling locations. WESTON will coordinate with the U.S. EPA and/or the IDPH in these activities. Permission will be obtained from the occupants before sampling. To begin the residential well sampling operation, the water system must first be purged using the following procedures:

- An outside cold water tap/spigot will be located and turned on to start the well pump.
- The pump will be allowed to operate for at least 20 minutes.
- Field measurements of pH, specific conductance, temperature, dissolved oxygen (DO), oxidation/reduction potential (ORP), ferrous iron, and turbidity will be taken and recorded in the field notebook. Purging will continue until the measurements for each of parameters have stabilized (±0.25 units for pH, ±10 percent for specific conductance, ± 1 °C, and ±10 percent for turbidity) for two consecutive rounds of readings. DO and ORP data are to be used primarily for evaluation of oxidation-reduction zones within the aquifer, and as such will not be used as criteria for stabilization. Ferrous iron will be taken after stabilization. WESTON SOPs for measuring pH, conductivity, temperature, DO, and ORP are presented in Appendix E.

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Water samples will be obtained from outside taps/spigots as close as possible to the pump. Samples will not be collected from leaky or faulty spigots. Samples will be collected prior to passing through aerators, strainers, hoses, filters, water softeners, or holding tanks. A steady flowing water stream at moderate pressure will be used to prevent splashing and the dislodging of particles in the faucet or water line.

The sample container caps will not be placed on the ground or in a pocket. The bottle will be held in one hand, the cap in the other, the bottle cap will be kept right side up (threads down), and care will be taken not to touch the inside of the cap. The sample bottle will be handled so neither fingers nor the faucet touch the inside of the bottle. Under no circumstances will sample containers be rinsed before use.

Sample bottles will be filled at an angle in order to limit splashing and bubbling. The sample container, volume and preservation requirements are presented in Table 7-1. VOC sample vials will be preserved with hydrochloric acid (HCl) prior to the addition of the sample. The VOC sample vials will be filled such that no air space is present in the bottle after it is capped. A new, preserved VOC container will be used to collect each sample. If bubbles persist, an unpreserved VOC sample will be collected (the Field Sample Manager will note the absence of the preservative on the sample paperwork and in the field logbook). Flow will not be adjusted during sampling in order to avoid dislodging any particles in the faucet. Sample containers will be filled directly from the tap or spigot.

The time of sample collection will be recorded. Any deviation from standard sampling practice will also be recorded. The name(s) of the occupant, the occupant's exact mailing address, and the occupant's telephone number will be entered into the field sampling logbook.

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3.3.2 CPT Groundwater Sampling

Following acquisition of stratigraphic data from instrumented CPT tests, data will be reduced and evaluated in the field and appropriate groundwater sample intervals selected as discussed in Subsection 2.3.2. Once sample intervals are selected, groundwater samples will be collected by advancing a stainless steel shielded well point and sample barrel to the desired depth at the end of the penetrometer string. This will be accomplished at a new probing location within 10 feet of the CPT test location. The wellpoint can then be sampled by opening the shield and allowing groundwater to enter and then retrieving the sampler. This method is sufficient where only VOCs will be collected and limited volume is required.

Groundwater samples will be collected as grab samples during this activity. Limited or no purging of groundwater will take place. This method will produce representative groundwater samples from the selected depth interval. Samples will not be filtered.

A field measurement of pH, specific conductance, temperature, DO, ORP, ferrous iron, and turbidity will be recorded in the field notebook. Sample bottles will be filled at an angle in order to limit splashing and bubbling. The sample container, volume and preservation requirements are presented in Table 7-1.

3.3.3 Monitoring Well Installation

The proposed number and location of monitoring wells and the rationale for monitoring well installation was discussed in Subsection 2.3.3. Each well will be screened entirely in the sand and gravel aquifer. Figure 3-2 shows the typical construction details of the monitoring wells that will be installed during the field investigation. After completion of each well boring, a monitoring well will be constructed using the following materials and methods:

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- The monitoring well will be constructed of 2-inch diameter stainless steel piping (type 304) with flush-threaded couplings. Well screens will be 10 feet long, constructed of stainless steel with continuously slotted 0.010-inch openings. The well riser will be covered with a slip cap.

- The annular space around the screen will be filled with a silica sand pack that is allowed to collapse as the augers are pulled out of the ground. The sand pack will extend 2 feet above the top of the screened interval.

- After the sand pack is in place, the screen will be sealed with 2 feet of bentonite pellets. The bentonite pellets will be hydrated with potable water and allowed to stand for 15 minutes before construction continues. Alternately, a high-solids pure bentonite grout will be used as a seal if a significant column of standing water is present. This material will be tremmied into place from the bottom up to the surface ensure sealing materials are placed properly.

- A minimum 2-foot-diameter by 4-inch-thick concrete pad will be installed at the ground surface around each of the monitoring wells. The pads will be made of ready-mix concrete and will be sloped away from the well to provide a water runoff diversion.

- To provide well protection, each well will be furnished with a flush mount cover or stick-up steel protective casing with locking cap. The flush mount cover or steel casing will be set in a concrete apron at all monitoring well locations. Locks will be provided for each flush mount cover or casing.

After installation of all monitoring wells, each well will be developed using the following procedures:

- All equipment to be introduced into the well will be decontaminated in accordance with the procedures outlined in Subsection 3.7.

- The depth to the water level in the well and the total depth of the well will be measured with an electrical sounding device (accuracy ±0.01 feet). The depth to water and the time of measurement will be recorded. The reference point for these depths will be the top of the well casing. The volume of standing water in the well will be calculated. Volume of water in a 2-inch-diameter well (gallons) = height of the standing water column in the well (feet) x 0.163 (gallons/foot). The well volume will be recorded.

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- Each well will be developed by alternately surging and purging for a minimum of 30 minutes. The surge and purge cycle will consist of several minutes of surging followed by several minutes of purging to remove the material collecting in the bottom of the well. The surging will be accomplished by rapidly moving a weighted bailer or surge block in the screened interval. This will displace and suspend any silt or fine sand buildup in the filter pack. This will be followed by purging the well using a bailer or pump to remove the suspended sediment.

- A positive displacement pump, bladder pump or disposable bailer will be used for development, and will be decontaminated in accordance with Subsection 3.8 before being used in a well. A minimum of three well volumes will be removed from the well. After removing the third well volume, readings for pH, temperature, specific conductance, and turbidity will be recorded. Well development will continue until these readings stabilize for two consecutive volumes (0.25 units for pH, ±10 percent for specific conductance, ± 1 °C for temperature , and ±10 percent for turbidity). A maximum of five well volumes will be purged during development.

- If the well can be purged dry, the well will be developed in a manner which limits agitation by slowly purging the well dry. Wells which can be purged dry may not be surged and no water will be added to the well.

Well development water will be managed in accordance with the requirements outlined in Section 9.

3.3.4 Monitoring Well Sampling

The sampling locations, the number of samples and the type of analysis for monitoring well samples is presented in Subsection 2.3.4. Purging and sampling will occur no sooner than 24 hours after well development. Monitoring wells will be sampled using a Grunfos Redi-flo pump, or equivalent submersible pump. As a backup, disposable bailers will be available if sampling with the pump is not feasible due to depth or other problems. All downhole equipment will be decontaminated pursuant to the protocols outlined in Subsection 3.7. Each sample will be collected using the following methodology:

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- The depth to the water level in the well and the total depth of the well will be measured with an electrical sounding device (accuracy ±0.01 feet). The depth to water and the time of measurement will be recorded. The reference point for these depths will be the top of the well casing.

- The volume of standing water in the well will be calculated and recorded. Volume of water in a 2-inch-diameter well (gallons) = height of standing water column in the well (feet) x 0.163 (gallons/foot).

- A positive displacement pump will be used for purging, and will be decontaminated before being used in a well.

- A minimum of three purge volumes will be removed from the well. After removing the third well volume, a field measurement of pH, specific conductance, temperature, and turbidity will be recorded in the field notebook. Purging will continue until the measurements for these parameters have stabilized (±0.25 units for pH, ±10 percent for specific conductance, ±1 °C, and ±10 percent for turbidity) for two consecutive rounds of readings. Measurements for DO and ORP will also be collected to evaluate natural attention characteristics but will not be used for stabilization. A maximum of five well volumes will be purged during sampling. WESTON SOPs for measuring pH, conductivity, temperature, turbidity, DO, ferrous iron, and ORP are presented in Appendix E.

- If the monitoring well runs dry before three volumes have been removed, the well will be allowed to recharge for 15 minutes and then be pumped dry again and allowed to recharge before sampling.

- Samples will be collected directly from the pump after the well purging has been completed. The VOCs sample will be collected first followed by other less volatile sample fractions. All samples will be unfiltered.

- Sample bottles will be filled at an angle in order to limit splashing and bubbling. The sample container, volume and preservation requirements are presented in Table 7-1. VOC sample bottles will be preserved with hydrochloric acid (HCl) prior to the addition of the sample. The VOC sample bottles will be filled such that no air space is present in the bottle after it is capped. If bubbles appear after the bottle is capped, a new sample will be collected. A new, preserved VOC container will be used to collect the sample. If bubbles persist, an unpreserved VOC sample will be collected (the Field Sample Manager will note the absence of the preservative on the sample paperwork and in the field logbook).

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3.3.5 Groundwater Elevation Measurements

Water level measurements at the monitoring wells will be conducted using the following protocols:

- The water level probe and cable will be decontaminated before each use with a distilled water rinse.
- Depth to water will be measured with an electrical sounding device (accuracy ±0.01 ft). The reference point for this measurement will be the top of the well casing.
- The depth to water and the time will be recorded in a field logbook.

The depth of the well will be measured with a electronic water level indicator. This activity will take place at the same time as the monitoring well survey.

All newly installed monitoring wells will be surveyed vertical reference using ground control equipment (transit, rod, etc.) at the following locations: ground surface, top of inner stainless steel riser, and top of outer protective casing. Horizontal control of wells will be accomplished by measuring the location of each monitoring well in relation to easily identifiable structures, roadways, property lines, etc. Vertical control points will be surveyed with respect to fixed datum points or temporary bench marks if fixed datums are not available. The accuracy of vertical measurements will be ±0.01 feet. Water levels will be referenced by elevation. This activity will take place during the field investigation after the monitoring wells have been installed.

3.3.6 Hydraulic Testing

Existing data from nearby area testing will be used to the extent it is available and valid, as determined by the U.S. EPA. Hydraulic tests (slug tests) will be performed in the newly installed monitoring wells after development and sampling. The slug tests will be conducted using a pressure transducer connected to an In-Situ Hermit 1000B data logger. A sealed PVC cylinder (slug) will be

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used to induce changes in water level. Two tests per well will be conducted. Both a rising head and a falling head test will be performed at each well, as described below:

- All equipment introduced into the well is decontaminated in accordance with procedures described in Subsection 3.7.
- The static water level is recorded.
- The pressure transducer is placed inside the well, approximately 3 to 5 feet from the bottom of the well (approximately halfway through the screened interval).
- The data logger is activated and the slug is introduced into the water column, displacing the water above the static level. The falling head test continues until the water level stabilizes at the static level.
- The slug is removed from the water column, and the water in the monitoring well falls below the static level. The rising head test continues until the water level stabilizes at the static level.

The water level versus time data collected from the slug tests will be used to calculate hydraulic conductivity values using the Bouwer and Rice (1976, 1989) method of analysis.

3.4 HYDROLOGICAL INVESTIGATIONS

3.4.1 Surface Water Sampling

The sampling locations, the number of samples and the type of analysis for surface water samples is presented in Subsection 2.4.1. Surface water samples will be collected from approximately the same location as the sediment samples. To the extent practicable, surface water samples will be collected before sediment samples at a specific location. However, if sediment sampling is performed before surface water sampling at a specific location, in order to minimize the interference of disturbed sediment in the surface water sample, a period of 30 minutes to one hour will elapse between when sediment sample collection is completed and surface water sampling begins at a

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sample location. Additional settlement time will be allowed if the sediment remains suspended in the water following the 1-hour elapsed period.

The following surface water sampling procedures will be used:

- A disposable bailer or long-handled sampler with a sample container clamped to the end will be lowered into the water. The container will be inverted, lowered to the depth of interest and held at approximately 45 degrees with the mouth of the bottle facing downstream. The sampling device will be allowed to fill with water.
- The water will then be poured into the appropriate sample containers.
- When sampling running water, surface water sampling will progress from downstream to upstream locations to eliminate the impact of sediment disturbance and/or cross-contamination of locations.
- All field sampling equipment used for surface water sampling will be decontaminated before collecting a sample at a new location. Decontamination protocols will be in accordance with Subsection 3.7.

3.4.2 Surface Water Elevation Measurements

The elevation of the Rock River and Dry Creek will be surveyed using ground control equipment (transit, rod, etc.) and referenced to a nearby datum used for surveying the monitoring wells.

3.5 ECOLOGICAL INVESTIGATIONS

Ecological investigations will involve literature search and potential site reconnaissance activities by a qualified ecological assessment specialist. The investigation will include wetland and habitat delineation on or adjacent to the site, wildlife observations at the site, a qualitative characterization of aquatic and terrestrial communities present or that could be present at the site, and identification

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of endangered species known to exist on the site or in close proximity. No biota sampling and populations studies are planned at this time.

3.6 WASTE DISPOSAL

Soil cuttings, to the extent possible, will be returned to the borehole upon completion of drilling and sampling activities. Purge water generated during groundwater sampling will be discharged to the ground surface adjacent to the sampling point. Any investigative derived waste which cannot be handled as described above will be stored on site in 55-gallon D.O.T. approved drums. At the completion of the RI/FS investigations, the drummed waste will be characterized for disposal acceptance.

3.7 DECONTAMINATION PROCEDURES

All sampling equipment including stainless steel spoons, spatulas, scoops, bowls, split-spoon and continuous samplers and bailers will be decontaminated before being used to collect a sample. The decontamination protocol for sampling equipment is presented in Table 3-1.

The working end of the drill rig and all downhole and associated drilling equipment, tools and materials will be decontaminated prior to drilling each new soil boring and monitoring well. Only decontaminated equipment will be placed in a boring or well. All drilling-related equipment (except the split-spoon or continuous sampler) will be decontaminated in accordance with the protocols presented in Table 3-2.

The management of water generated during decontamination will be in accordance with the requirements outlined in Section 9.

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A specific decontamination area will not be constructed at the site since the site is primarily private residential and commercial properties. Decontamination activities will be carried at the sampling locations.

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WESTON

GEOLOGIC DRILL LOG

PROJECT NAME AND LOCATION	PAGE NO.	HOLE NO.

START	FINISH	DRILLER	DRILL METHOD	BOREHOLE DIAMETER	WELL DIAMETER	TOTAL DEPTH

LOGGER	TOP of CASING ELEV.	GROUND ELEVATION	DEPTH/ELEVATION GROUNDWATER	DATE MEASURED

SAMPLE NO.	SAMPLE TYPE	RECOVERY "	SAMPLE BLOWS*	ELEV	DEPTH	GRAPHIC LOG	WELL CONSTRUCTION	CLASS-IFICATION	SAMPLE INTERVAL	DESCRIPTION	NOTES

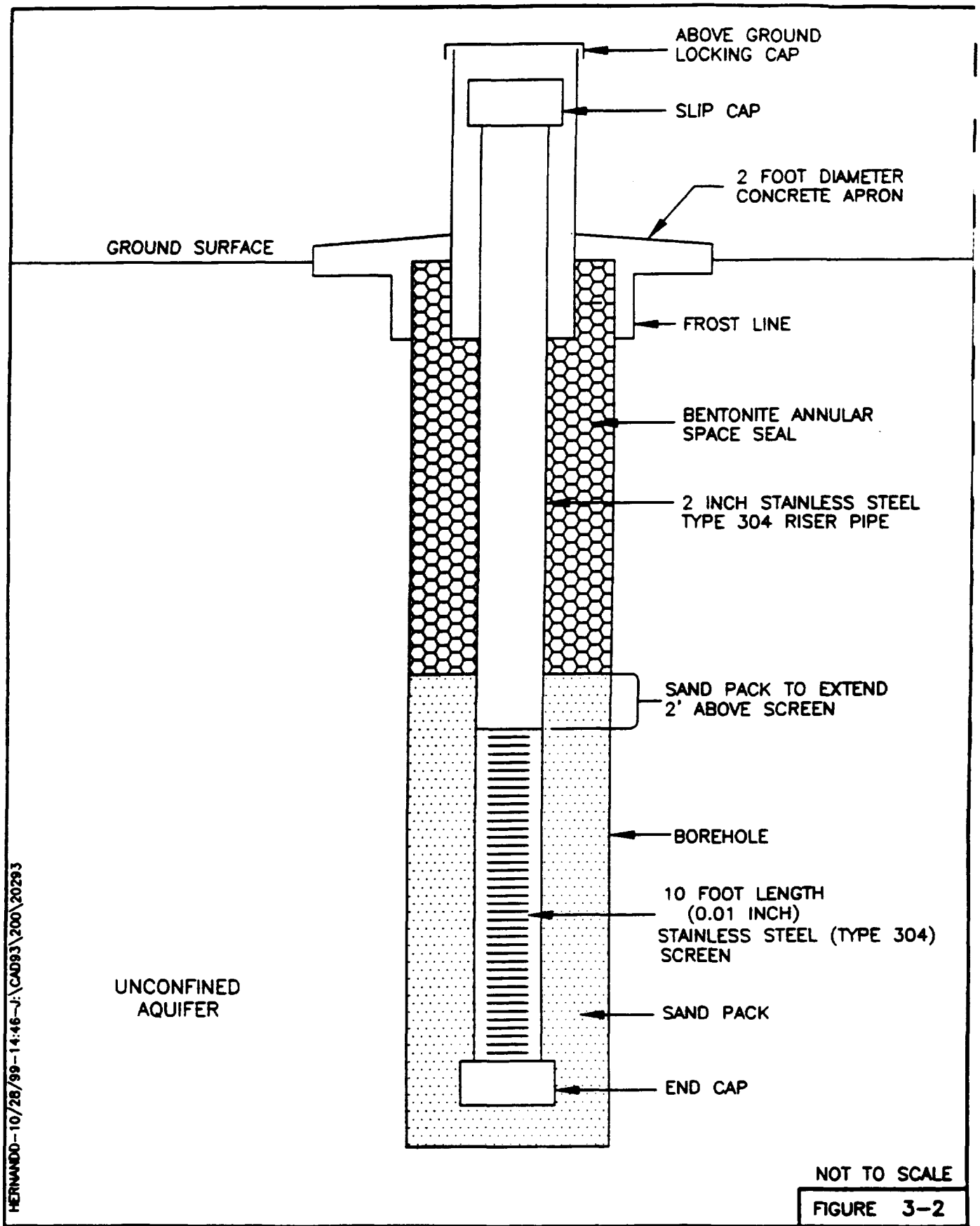
*ASTM D1586
SS = SPLIT SPOON
D = DENNISON
ST = SHELBY TUBE
C = CORE
CT = CUTTINGS
CS = CONTINUOUS SAMPLER
OT = OTHER

PAGE NO. HOLE NO.

FIGURE 3-1

RESPONSE ACTION CONTRACT
U.S. EPA CONTRACT No. 68-W7-0026
WORK ASSIGNMENT No. 036-RICO-05MZ
DOCUMENT CONTROL No. RFW036-2E-ADKA

EXAMPLE BORING LOG FORM
EVERGREEN MANOR
Roscoe, Illinois



NOT TO SCALE

FIGURE 3-2

RESPONSE ACTION CONTRACT U.S. EPA CONTRACT No. 68-W7-0026 WORK ASSIGNMENT No. 036-RICO-05MZ DOCUMENT CONTROL No. RFW036-2E-ADKA	TYPICAL MONITORING WELL CONSTRUCTION EVERGREEN MANOR Roscoe, Illinois

Table 3-1

Standard Decontamination Protocol for Sampling Equipment
Evergreen ManorSite
Roscoe, Illinois

Step	Procedure
1	Scrub equipment thoroughly with soft-bristle brushes in a phosphate-free, low-sudsing detergent solution.
2	Rinse equipment with tap water by submerging and/or spraying. (See note below.)
3	Rinse equipment with reagent-grade distilled/deionized water until dripping and allow to air dry for 1 to 2 minutes.
4	Rinse equipment a second time with deionized water by spraying until dripping.
5	Place equipment on polypropylene or aluminum foil and allow to air-dry for 5 to 10 minutes.
6	Wrap equipment in polypropylene or aluminum foil for handling and/or storage until next use.

Note: The decontamination liquids will be managed as described in Section 9. If sampling equipment was used to collect oily or adhesive types of contaminated media, or the presence of organic compound residue is suspected, a rinse via spraying with isopropanol will be included after Step 2.

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Table 3-2

Standard Decontamination Protocol for Drilling Equipment
Evergreen Manor Site
Roscoe, Illinois

Step	Procedure
1	Move the drilling rig or other equipment/materials to the designated decontamination area at the site.
2	Support all augers and related downhole drilling equipment aboveground and individually steam-clean.
3	Steam-clean the control panel and working area of the drill rig.
4	Place all decontaminated well materials (e.g., well casing, well screen) on clean polypropylene sheeting until use.

Notes:

All steam cleaning will be performed using pressurized steam. Steam cleaning will continue until all solid material and/or visible contamination is removed.

The decontamination liquid will be managed as described in Section 9.

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SECTION 4
FIELD QUALITY CONTROL SAMPLES

The sampling effort at the Evergreen Manor site will include the following types of field QC samples:

- Field duplicates.
- Matrix spikes/matrix spike duplicates.
- Field blanks.
- Trip blanks.

Section 4.1 of the Evergreen Manor site QAPP explains the purpose behind each type of QC sample. Sample containers and handling and shipment procedures that will be used are identical to those used for the investigative samples. Each field QC sample will be documented on a chain-of-custody form. Table 2-1 shows the specific level of QC effort for field activities. The following subsections detail the collection procedures for each QC sample type.

4.1 FIELD DUPLICATE SAMPLES

Field duplicate samples will be collected at selected locations during soil, sediment, surface water, and groundwater (residential wells, monitoring wells, and CPT samples) at a 1-per-10 sample frequency, using procedures identical to those used for the investigative samples. A verification sample for groundwater and soil/sediment VOC analysis will also be collected for laboratory confirmation of mobile laboratory results. These duplicate samples will also be collected at a 1-per-10 sample frequency, using procedures identical to those used for the investigative samples. Duplicate samples will be analyzed for the same parameters as the investigative sample unless otherwise stated in the FSP or QAPP. Duplicate samples will be collected by alternatively filling two sets of sample bottles from the same sample unit (e.g., bailer, split spoon, scoop, etc.). For samples requiring chemical analyses, the VOC fraction for each duplicate sample will be collected

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immediately after the VOC fraction for the investigative sample to minimize the loss of VOCs during sample collection.

4.2 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES

Matrix spike/matrix spike duplicate samples (MS/MSDs) will be collected on a one per 20 sample (or less) basis for soil, sediment, surface water, and groundwater (residential well, monitoring well, and CPT samples). MS/MSD samples are investigative samples on which MS/MSD analyses are performed. MS/MSD analyses only apply to organic samples; for inorganic samples, duplicate/spike analyses are performed. No MS/MSD or duplicate/spike analyses are performed on geotechnical samples (i.e., grain-size, permeability, etc.). Investigative soil and sediment samples assigned for MS/MSD and spike/duplicate analyses do not require the collection of extra sample volume. Extra sample volume is required for aqueous MS/MSD and spike/duplicate analyses. For groundwater MS/MSD analyses, triple the normal volume is required for volatile organics, and double the normal volume is required for semivolatile, pesticides/PCBs, and total metals aliquots.

Field blank, trip blanks and field duplicate samples will not be used as MS/MSD and spike/duplicate samples. All MS/MSD and spike/duplicate samples will be identified as such on all sample paperwork.

4.3 FIELD BLANKS

Field blank samples will be collected during surface water, monitoring well water, and groundwater sampling events (monitoring well and CPT samples). Field blanks will not be collected during residential well water sampling events. One field blank will be collected for every 10 or fewer investigative aqueous samples collected during the field sampling activities. For groundwater samples, field blanks will be obtained by pouring ultra-pure water (laboratory-grade water) over and/or through a decontaminated or disposable sampling device such as a bailer, and collecting the water in the required sample containers. All disposable sampler parts such as tubing will be replaced

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with unused or decontaminated equipment prior to collecting the field blank. If a sample device is used for surface water sampling, ultra-pure water will be poured over or through the device (in a fashion which mimics actual sampling) into a sample container. The sample will be transferred from the collection containers to the field blank sample container in a manner similar to the investigative sample. Each field blank will be analyzed for the same parameters as the investigative samples in accordance with the same analytical methodologies. The U.S. EPA Region V CRL discourages the use of water blanks for soil and sediment samples; therefore, no field blanks will be collected for soil and sediment samples. All field blanks will be identified as such on all sample documentation.

4.4 TRIP BLANKS

One trip blank sample will be enclosed in each sample shipment container in which aqueous VOC samples are included. All sample handling, volume, packaging and preservation requirements for the trip blank will be identical to the investigative VOC samples. The trip blanks will be obtained by pouring ultra-pure water (laboratory-grade water) directly in the pre-preserved sample containers under laboratory-type conditions as soon as it is practicable (given the sample holding time) prior to the beginning of a field sampling event. The trip blanks will then be subsequently placed in a sample shipment container and accompany field personnel to the site. The trip blank will be documented and identified as such on all sample documentation.

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SECTION 5

SAMPLE NUMBERING SYSTEM

All samples for analysis, including QC samples, will be given a unique sample number. The sample numbers will be recorded in the field logbook, the chain-of-custody, and the shipment documents.

WESTON will assign each sample two identification numbers: a project sample number and a SAS laboratory label number. The project sample number highlights the sample matrix and location, and will be used for documentation purposes in field logbooks, as well as for presentation of the analytical data in WESTON memoranda and reports. The SAS laboratory label number is used by the U.S. EPA to track samples.

5.1 PROJECT SAMPLE NUMBERING SYSTEM

The WESTON project sample numbering system will be composed of the following components:

- Project Identifier: The first part of the project sample number will consist of a three character designation. This three character code will be used to identify the Evergreen Manor site and the phase of the field investigation. **EM1** corresponds to Evergreen Manor site, phase 1 of the field investigation.

- Sample Type and Sample Location: This shall consist of the following:

  - A two-character sample type code. For the proposed field sampling, SB for subsurface soil samples, SD for sediment samples, MW for monitoring well samples, CPT for CPT groundwater samples, RW for residential well samples, and SW for surface water samples.

  - A sample location code. The above sample type code will be combined with sample location identification (e.g, SB02 for soil boring "SB02", MW02 for monitoring well "MW02"). For field blanks and trip blanks, the two-character sample type code will be combined with FB for field blanks and TB for trip blanks (e.g, MWTB for a monitoring well trip blank). The sample type codes and corresponding location numbers are presented in Table 5-1.

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- Sequence Identifier. This shall consist of the following:

  - A two-digit sequence number that tracks the number of samples collected from a specific location. Sequence 01 refers to the first sample interval, and sequence 02 refers to the second sample interval. Sample depths will not be a part of the sample code; rather, depth information will be recorded in the site field logbook and presented with the analytical results.

  - If the sample is a field duplicate sample, the above will be combined with DP. If the sample is a matrix spike/matrix spike duplicate sample, the above will be combined with MSD.

  - For field and trip blanks, the two-digit sequence number will denote the sequential number of field or trip blank sample collected for that sample type.

Some examples of the WESTON project sample numbering system are as follows:

- EM1-SB02-01DP: Evergreen Manor site, phase 1; soil boring location 2; duplicate of first sample collected at this location.

- EM1-MW03-01: Evergreen Manor site, phase 1; monitoring well location 3; first groundwater sample at this location.

- EM1-MWTB-01: Evergreen Manor site, phase 1; ground water sampling, first trip blank sample.

- EM1-MWFB-03: Evergreen Manor site, phase 1; monitoring well water sampling, third field blank sample.

- EM1-MW01-01MSD: Evergreen Manor site, phase 1; monitoring well location 1; first sample collected at this location; sample is a matrix spike/matrix spike duplicate sample.

- EM1-SD03-01: Evergreen Manor site, phase 1; sediment sample location 3; first sample collected at this location.

- EM1-CPT03-01: Evergreen Manor site, phase 1; CPT groundwater sample location 3; first groundwater sample at this location.

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5.2 SAS LABORATORY SAMPLE NUMBERING SYSTEM

Sample numbering for SAS samples will follow the U.S. EPA Region V Central Regional Laboratory (CRL) sample numbering system. The CRL sample numbering system contains the following elements:

- 2000—Indicates fiscal year (October 1, 1999 through September 30, 2000).
- ZG—Indicates Contractor Code (WESTON).
- Two-digit laboratory identification number.
- S—Indicates sample type (S = Sample, D = Duplicate, R = Blank).
- Two-digit sequential sample number (01-99).

An example SAS laboratory sample number is as follows: 2000ZG02S01—The first sample collected by WESTON in fiscal year 2000 for the Evergreen Manor site for analysis by Chemtec.

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Table 5-1

Sample Codes and Location Numbers
Evergreen Manor Site
Roscoe, Illinois

Sample Type	Sample Type Code	Sample Location Code
Soil Boring	SB	01 through *
Sediment	SD	01 through *
Monitoring Well Water	MW	01 through *
CPT Water	CPT	01 through *
Surface Water	SW	01 through *

* Final count depends on the number of samples collected.

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SECTION 6

SAMPLE DOCUMENTATION AND TRACKING

6.1 FIELD RECORDS

Field observations and other information pertinent to the collection of samples will be recorded in the field. All entries will be made in a bound logbook in ink. Logbooks will be identified by unique sequential numbers. The data to be recorded for each sample will include date, time (24-hour military time reference), sample number, sample location, sample appearance, and name of the persons collecting the sample. In addition, general information will be recorded in the logbook daily, including personnel present at the site, level of protection being worn, and weather. Photographs will also be taken and logged to document sampling activities.

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

Field chain-of-custody (COC) procedures are presented in Subsection 6.2 of the QAPP, with a detailed summary in Subsection 6.2.4 of the QAPP. Details on the completion of field sample COC documentation are discussed in Subsection 6.3 of the FSP.

6.3 SAMPLE DOCUMENTATION FORMS

Sample documentation forms required by the U.S. EPA are numbered. Copies of the multiple copy forms must accompany samples to the laboratory. Upon return from the field, the FSM must give the other copies to the SMC, who will forward them to the RSCC or SMO, ensuring that the appropriate paperwork is attached.

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Required paperwork for SAS chemical laboratory samples includes SAS packing lists, sample tags and COC seals. All sample documentation forms will be completed by WESTON personnel (mainly the assigned FSM) in accordance with the requirements outlined in the *U.S. EPA Region V Sample Handling Manual* (U.S. EPA Region V, March 1989) or the most recent revision. The U.S. EPA is currently using a combined COC/SAS packing list (COC/SAS). The U.S. EPA Region V Region Sample Control Coordinator (RSCC) is responsible for providing WESTON with updates on changes in the sample documentation forms and requirements. The WESTON Sample Management Coordinator (SMC) will train all field personnel on any new documentation requirements before field activities begin.

The critical aspects of the documentation protocol for shipping samples to the SAS laboratories are summarized below.

Chain-of-Custody/SAS Packing List Form

Combination COC/SAS packing lists are used instead of COC/TR forms for samples sent for SAS analysis. To maintain sample custody in accordance with the U.S. EPA requirements, the following sample documentation protocol must be implemented:

- Each sample shipment container must have at least one COC/SAS Packing List form enclosed if samples are being sent to the SAS laboratories.
- Each sample in a shipment container must be identified and documented on the accompanying COC/SAS Packing List form.
- All information associated with the samples must be included on the COC/SAS Packing List form as shown in the completed example presented in Appendix E.

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- The COC seal numbers on seals assigned to a particular cooler must be documented on the COC/SAS Packing List forms in that cooler in the section provided.

- The carrier service person does not have to sign the COC form if the custody seals remain intact. The airbill number must be written on the COC/SAS Packing List form in the section provided.

- The site name will be printed on only certain copies of the COC/SAS Packing List form since this document does not allow this information to show up on the copies sent to the SAS laboratories.

- Field personnel must carefully transcribe SAS sample numbers onto the COC/SAS Packing List form in the space provided.

Figures 6-1 and 6-2 present examples of a COC/SAS Packing List combination form and COC/Organic Traffic Report form. Appendix E contains completed examples of these forms.

Chain-of-Custody Seals

- Two seals per shipping container are used to secure the lid and provide evidence that samples have not been tampered with.

- The COC seals must be covered with clean tape to avoid accidental damage during shipment.

- The COC seal numbers must be documented on the COC/SAS Packing List or stand-alone COC forms as herein described.

- All sample shipment containers require COC seals.

Sample Tags

- Each sample container must have a Sample Tag affixed to it with string or wire.

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- COC/SAS Packing List or stand-alone COC form numbers are recorded in the "Remarks" section of the tag.
- Sample Tag numbers are recorded on the stand-alone COC form or on the COC/SAS Packing List form.

Figure 6-3 presents an example of a sample tag and a chain of custody seal.

All paperwork accompanying the samples being shipped to the SAS laboratories will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies will be made of all sample documentation and retained for in-house files.

All sample handling procedures and paperwork will be performed and completed according to guidance set forth in the *U.S. EPA. Region V CRL Sample Handling Manual*, (U.S. EPA, 1989) (or most recent version) and the *U.S. EPA User's Guide to the Contract Laboratory Program*, (U.S. EPA, December 1988).

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EPA United States Environmental Protection Agency
Contract Laboratory Program

Special Analytical Services Packing List/Chain of Custody

SAS No.

Case No.

1. Project Code | Account Code | 2. Region No. | Sampling Co. | 4. Date Shipped | Carrier

Regional Information | Sampler *(Name)* | Airbill Number

Non-Superfund Program | Sampler Signature | 5. Ship To

Site Name | 3. Purpose* Lead: SF, PRP, ST, FED; Early Action: CLEM, PA, REM; SI, ESI, RI, OIL, UST; Long Term Action: FS, RD, RA, O&M, NPLD

City, State | Site Spill ID | ATTN:

6. Matrix *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil
7. Waste
8. Other *(Specify in Column A)*

7. Preservative *(Enter in Column D)*
1. HCl
2. HNO3
3. NAHSO4
4. H2SO4
5. NAOH
6. Ice Only
7. Other *(Specify in Column D)*
N. Not Preserved

Sample Numbers (From Labels)	A Matrix (from Box 6) / Other	B Conc.: Low Med High	C Sample Type Comp./Grab	D Preservative (from Box 7) / Other	E Analysis	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Field QC Qualifier B Blank S Spike D Duplicate R Rinsate PE Perform. eval Not a QC Sample

Shipment for SAS Complete? (Y / N) | Page ___ of ___ | Sample(s) to be Used for Laboratory QC | Additional Sampler Signatures | Chain of Custody Seal Number(s)

CHAIN OF CUSTODY RECORD

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 by *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received for Laboratory by: *(Signature)*	Date/Time	Remarks Is custody seal intact? Y / N / none	

A21-012-7 REV. 3/94

DISTRIBUTION: White - Region Copy; Gold - Lab Copy for Return to Region; Yellow - Data User**; Pink - Lab Copy for Return to Data User**

EPA Form 9110-3

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

**Data User means the organization which contracted the laboratory services

51538

FIGURE 6-1

RESPONSE ACTION CONTRACT
U.S. EPA CONTRACT No. 68-W7-0026
WORK ASSIGNMENT No. 036-RICO-05MZ
DOCUMENT CONTROL No. RFW036-2E-ADKA

SPECIAL ANALYTICAL SERVICES PACKING LIST AND CHAIN-OF-CUSTODY FORM
EVERGREEN MANOR
Roscoe, Illinois

EPA United States Environmental Protection Agency
Contract Laboratory Program

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

Case No.

1 Project Code	2 Region No	Sampling Co	4. Date Shipped / Carrier
Account Code	Sampler (Name)		Airbill Number
Site Name	Sampler Signature		5. Ship To:
City, State / Site Spill ID / Op Unit	3 Purpose** Lead: SF, PRP, ST, FED, BZ; Early Action: IA, PA, REM, RI, SI, ESI; Long Term Action: RIFS, RD, RA, O&M		ATTN:

6. Matrix (Enter in Column A)
1 Surface Water
2 Ground Water
3 Leachate
4 Field QC
5 Soil/Sediment
6 PE-water
7 PE-soil
8 Other (specify in Column A)

7 Preservative (Enter in Column D)
1 HCl
2 HNO3
3 NaHSO4
4 H2SO4
5 Ice only
6 CH3OH
7 Other (specify in Column D)
N Not Preserved

CLP Sample Numbers (from labels)	A Matrix (from Box 6) Other	B Conc Low Med	C Sample Type Comp / Grab	D Preservative (from Box 7) Other	E RAS Analysis TA (circle one) PR* 7 14 21 VOA	TA (circle one) PR* 7 14 21 BNA	TA (circle one) PR* 7 14 21 Pest/PCB	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Corresponding CLP Inorganic Sample No.	J Sampler Initials	K Field QC Qualifier B = Blank S = Field Spike D = Field Duplicate R = Rinsate PE = Perform Eval

Shipment for Case Complete? (Y/N)	Page ___ of ___	VOA MS/MSD Required? Y/N Sample #:	Additional Sampler Signatures
		BNA MS/MSD Required? Y/N Sample #:	Chain of Custody Seal Number(s)
		Pest/PCB MS/MSD Required? Y/N Sample #:	

*PR provides 7-day data turnaround in addition to preliminary results. Requests for preliminary results will increase analytical costs.

Chain of Custody Record

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 by (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	

Distribution: Blue - Region Copy; White - Lab Copy for Return to SMO; Pink - SMO Copy; Yellow - Lab Copy for Return to Region

See Reverse for Additional Standard Instructions
**See Reverse for Purpose Code Definitions

EPA Form 9110-2 (2/99)

CLASS 99 001

389653

FIGURE 6-2

RESPONSE ACTION CONTRACT
U.S. EPA CONTRACT No. 68-W7-0026
WORK ASSIGNMENT No. 036-RICO-05MZ
DOCUMENT CONTROL No. RFW036-2E-ADKA

ORGANIC TRAFFIC REPORT AND CHAIN-OF-CUSTODY FORM
EVERGREEN MANOR
Roscoe, Illinois

DESIGNATE		Samplers (signatures)	PRESERVATIVE: H_2SO_4 ☐ ICE ☐ HCL ☐ HNO_3 ☐ NaOH ☐ Other ☐			
Grab			**ANALYSES**			
Comp.			VOA		METALS	
			ABN		CYANIDE	
			PEST/PCB			
					Mercury	
Time			Pesticides		Fluoride	
			Herbicides		Nitrate/Nitrite	
			PCB		TOC	
Month/Day/Year			PCDD/PCDF		BOD	
			2.3,7,8-TCDD		COD	
			Ames Mutagen		TDS	
			Asbestos		TSS	
			Phosphorus		O&G	
Sample Number		Station Number and Location	Phenols		Sulfate	
			PAH		Chloride	
			TCLP		Sulfide	
			TOX		Ammonia	
			CBOD		Alkalinity	
			Bio-Acute		Acidity	
Case # or Project Code			Bio-Chronic		TKN	
			Matrix:			
			Remarks:			
			USE FOR MS/MSD ☐			
			Tag Number 5- 054142		Lab Sample Number	

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION V
OFFICIAL SEAL
№ 42040

FIGURE 6-3

RESPONSE ACTION CONTRACT	SAMPLE TAG/ CHAIN OF CUSTODY SEAL
U.S. EPA CONTRACT No. 68-W7-0026 WORK ASSIGNMENT No. 036-RICO-05MZ DOCUMENT CONTROL No. RFW036-2E-ADKA	EVERGREEN MANOR Roscoe, Illinois

SECTION 7
SAMPLE HANDLING

7.1 SAMPLE CONTAINERS AND SAMPLE PRESERVATION

All samples collected for analysis will be containerized, preserved, packaged and shipped in accordance with *U.S. EPA Region V CRL Sample Handling Manual*, (U.S. EPA, 1989), *U.S. EPA User's Guide to the Contract Laboratory Program* (U.S. EPA, 1988), The U.S. Department of Transportation's regulations (49 CFR 173 to 177), the Nuclear Regulatory Commission (NRC) standards for protection against radiation (10 CFR 20) and *Dangerous Goods Regulations*, (International Air Transport Association (IATA), (1998). Table 7-1 lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters and media applicable to the Evergreen Manor site field investigation. WESTON will obtain sample containers according to U.S. EPA specifications as described in Section 10.

7.2 SAMPLE PACKAGING AND SHIPMENT

All samples shipped from the Evergreen Manor site must be shipped in accordance with U.S. Department of Transportation regulations and must comply with *Dangerous Goods Regulations* (IATA, 1998) if shipped by air transportation.

Following sampling, the exterior of all sample bottles will be initially decontaminated near the sampling location by wiping with a moist cloth. The filled sample containers will not be sprayed with water during decontamination because this water could contact the sample if the container was not tightly sealed. In preparation for shipment to the SAS laboratories, all samples will be packaged in accordance with the following general procedures:

- Check to make sure container cap is securely tightened. Seal with tape. Mark liquid levels of water samples if bottles are partially full.

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- Make sure the SAS sample names and sample tags are securely attached to the sample containers for SAS organic, inorganic and radiological analytes. Place each container in a zip-lock baggie, ensuring that the sample tags can be read.

- Low concentration samples will be placed in a shipment container lined with a large polyethylene bag. Enough vermiculite or equivalent absorbent material will be packed around the samples to minimize the possibility of sample container breakage. The temperature will be maintained at 4° C with cold packs or ice sealed in plastic bags as appropriate to the sample. The remaining space in the container will be filled with additional packing material and the large bag sealed. Radiological samples do not require cooling with coldpacks or ice.

- If a sample is deemed medium concentration or a dangerous good, it will be packaged in accordance with the IATA *Dangerous Goods Regulations*, based on the classification of the samples.

- Place COC/SAS Packing List forms in a zip-lock bag and tape to inside of shipment container lid.

- Close shipment container and seal it shut with strapping tape. If shipment container has a drain port, seal it shut with tape. Place custody seals across seam between the container lid and base so that custody seal would be broken if shipment container was opened. Cover custody seals with waterproof tape.

- Affix airbill with shipper's and recipient's names and addresses to top of shipment container. Affix a second mailing label with the same information to the top of container in case airbill becomes detached from container during shipment. Place "This End Up" labels on container as specified by IATA.

The WESTON FTL must contact the WESTON SMC to confirm sample shipment dates for all analyses. The FTL will notify the SMC of any last-minute changes in sampling schedule that will affect the sample shipment schedule.

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Table 7-1

Sample Container, Volume, and Preservation Requirements
Evergreen Manor Site
Roscoe, Illinois

Matrix Type	Analysis	Sample Concentration Level	No. of Bottles	Type of Bottles	Preservatives	Technical Holding Time*
Groundwater (CPT, Monitoring Well, Residential Well, and Surface Water)	Volatiles	Low	2	40-mL glass vials	Cool, 4°C 4 drops 1:1 HCl to pH <2	14 days
Groundwater (Monitoring Well and Residential Wells)	Orthophosphate	Any	1	4-L polyethylene bottle	Cool, 4°C H_2SO_4 to pH <2	28 days
	Ammonia	Any		1-L polyethylene bottle	Cool, 4°C H_2SO_4 to pH <2	28 days
	Nitrate	Any		1-L polyethylene bottle	Cool, 4°C H_2SO_4 to pH <2	7 days (24 hours if unpreserved)
	Nitrite	Any		1-L polyethylene bottle	Cool, 4°C	7 days
	Chemical Oxygen Demand	Any		1-L polyethylene bottle	Cool, 4°C H_2SO_4 to pH <2	28 days
	Sulfide	Any		1-L polyethylene bottle	Cool, 4°C Zinc acetate + NaOH to pH between 9 and 11	7 days
	Sulfate	Any		1-L polyethylene bottle	Cool, 4°C	28 days
Soil/Sediment	SAS-Volatiles	Low		4 oz. glass jar	Cool, 4°C Laboratory to preserve upon receipt	48 hours for preservation, 14 days for analysis
	Total Organic Carbon	Any	1	8 oz glass jar	Cool, 4°C	6 months
	Grain Size	Any		16 oz glass jar	NA	NA

* All holding times are from the date of sample collection.

Note: Aqueous MS/MSD samples will require triple the normal volume for volatile organics. One trip blank will accompany each aqueous VOA shipment container. Trip blanks accompanying water samples will be collected in two 40-mL glass vials. No trip blank will be sent for soil samples.

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SECTION 8

SAMPLING TEAM ORGANIZATION

The sampling team organization is discussed in Subsection 3.3 of the QAPP.

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SECTION 9

MANAGEMENT OF INVESTIGATION-DERIVED WASTES

For purposes of this FSP, investigative-derived wastes (IDW) are defined as any by-product of the field activities that is suspected or known to be contaminated with other hazardous substances. The performance of field activities will produce waste products such as development and purge groundwater, decontamination wastewater, drill cuttings, and expendable personnel protective equipment. As much as possible, the drill cuttings from the soil borings will be put back in the soil borings. The remaining drill cuttings will be containerized in 55-gallon drums.

A temporary decontamination area will be set up on site adjacent to each sampling locaiton. Wastewater which does not exhibit overt indications of contamination determined visually and with real-time monitoring equipment, will be allowed to drain to the ground surface. Where this method is not possible or appropriate, wastewater will be containerized in DOT-approved drums. Depending on the volume of waste generated during the field activities and on the duration of storage time, the water in the drums may be pumped into a portable on-site storage tank. Storing the water in a tank would reduce the amount of drums needed.

If containerization is necessary, each type of waste will be segregated during the field activity and containerized separately. All storage containers will be labeled appropriately. Wastes will be stored at the site in a secured staging area until the analytical results of the site investigation are interpreted. At that time, each segregated waste will be evaluated based on the field data and disposal arrangements executed in accordance with appropriate local, state, or federal regulations. If deemed appropriate, the management of the wastes will be incorporated into the remedial action for the site. WESTON will refer to the U.S. EPA's *Management of Investigation-Derived Wastes During Site Inspections* (U.S. EPA, 1991) for guidance on off-site disposal policy, if this action is deemed necessary.

This document was prepared by Roy F. Weston, Inc., expressly for U.S. EPA. It shall not be released or disclosed in whole or in part without the express, written permission of U.S. EPA.

SECTION 10
SAMPLE CONTAINER PROCUREMENT

All sample containers being used for chemical analysis during the Evergreen Manor site sampling program will be purchased by WESTON from a reputable supplier capable of providing the bottle quantity and type that meet or exceed the strict quality control requirements set forth by the U.S. EPA in OSWER Directive No. 9240.0-05A. Sample bottles being used for chemical analysis (SAS) will be provided by the WESTON procured laboratory.

All sample containers (bottles) will be prepared according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers*, (U.S. EPA, 1992) or the most current revision. It will be ensured that the bottles used for the sampling activity do not contain target organic and inorganic contaminants exceeding the level specified in the above-mentioned document. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. The field team leader or the leader's designee will record the bottle lot numbers associated with each sample collected during the Evergreen Manor sampling effort. This data will be maintained in the project evidence file and will be available, if requested, for U.S. EPA review.

In addition, the data for field blanks and trip blanks will be monitored for contamination, and corrective actions will be taken as soon as a problem is identified. This will be accomplished either by discontinuing the use of a specific bottle lot, contacting the bottle suppliers for retesting the representative bottle from a suspect lot, resampling the suspected samples, validating the data (taking into account that the contaminants could be introduced by the laboratory (e.g., by common laboratory solvents, sample handling artifacts) or could be bottle QC problems) so as to make an educated determination if the bottles and hence, the data are still usable, whichever is appropriate.

This document was prepared by Roy F. Weston, Inc., expressly for U.S. EPA. It shall not be released or disclosed in whole or in part without the express, written permission of U.S. EPA.

For the Evergreen Manor site, the corrective actions will be conducted comprehensively to avoid the use of identified contaminated lots from other projects, and to ensure that if the bottle suppliers are deemed unresponsive or unable to provide cleaned bottles as specified, then other U.S. EPA-related projects are not negatively affected by the use of the noncompliant bottles.

If re-sampling is deemed necessary, WESTON will require authorization for additional effort. Any schedule delays will be brought to the attention of the U.S. EPA RPM/WAM.

This document was prepared by Roy F. Weston, Inc., expressly for U.S. EPA. It shall not be released or disclosed in whole or in part without the express, written permission of U.S. EPA.

APPENDIX B

SAS REQUESTS

U.S. Environmental Protection Agency Region V
77 West Jackson, SM- 5J
Chicago, IL 60604
PHONE: (312) 353-2310 FAX: (312) 886-0186

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling :May 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734 Site/Spill ID# MZ	

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 delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**
   Analysis for volatile organics in soil/sediment and groundwater. The volatile organics require analysis by FASP VOA -05 method for ESAT's mobile laboratory.

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

   Approximately 250 groundwater samples and 8 soil samples for VOC analysis.

3. **Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.)**
   Superfund remedial.

4. **Estimated date(s) of collection:**
   May 2000

5. **Estimated date(s) and method of shipment:**
   Daily, overnight courier (e.g., Federal Express)

6. **Number of days analysis and data required after laboratory receipt of samples:**
   Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**
   FASP-VOAMS-01

   Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

8. **Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**
   None

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.

CLP-type data package required.

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

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 span identified in section 6, above

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
As per FASP-VOAMS-01		

II. QC REQUIREMENTS

Audit	Frequency of Audits	Limits [illegible] or Co[illegible]
As per FASP-VOAMS-01		

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

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

5/012-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling : May 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734	Site/Spill ID# MZ

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 delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of sulfide in water (groundwater, surface water, etc.). Samples will be unfiltered. Results shall be reported as mg/L S^{2-}. No deviation is allowed for any portion of this SAS without the permission of the TPO.

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

Monitoring and residential well groundwaters - Twenty-five sulfide samples (includes field duplicates and field blanks).

3. Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.):
Superfund remedial.

4. Estimated date(s) of collection:
May 2000

5. Estimated date(s) and method of shipment:
Daily, overnight courier (e.g., Federal Express)

6. Number of days analysis and data required after laboratory receipt of samples:
Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

Method 376.1 Sulfide (Titrimetric, Iodine)

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Samples are perserved with 2N zinc acetate at the rate of 1.5 ml/l plus 6N NaOH to pH between 9 and 11. Samples must be analyzed within 7 days of 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 shall be clearly identified. Bench records tabulating the order of analysis of calibration standards, lab control standards, blanks, spikes, samples, etc. shall be submitted. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, [illegible] sample and/or extract chain of custody forms and copies of pages from preparation 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.

All records of analysis and calculations shall be legible and be 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. Corresponding true or target values and associated 95% confidence limits for analysis results shall be submitted for all reference samples used.

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

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. Photocopies of chain of custody forms and airbills may be submitted along with the exact location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:
Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
Sulfide	1.0 mg/l	Report results to the nearest 0.1 mg/l (below 10 mg/l) or to 2 significant figures above 10 mg/l.

Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. Method 376.1

Audit	Frequency of Audits	Limits (% or Conc.)
Duplicate samples	at least one per group of 20 or fewer samples	±10 % RPD or ± 0.3 mg/l
Analytical Spike	at least one per group of 20 or fewer samples	80-120%
Reference Sample	at least one per group of 20 or fewer samples.	

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact Region V.

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

5/011-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V		
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore	
C. Telephone Number:	(312) 353-2310	(312) 886-1488	
D. Date of Request:	October 1999	Date of Sampling : February 2000	
E. Site Name:	Evergreen Manor, Roscoe, Illinois		
Cerclis ID#	ILD984836734	Site/Spill ID#	MZ

1. General description of analytical service requested:

Analysis of sulfate in water samples (surface water, groundwater, drinking water, etc.). Results are reported as mg/L SO_4. No deviation is allowed for any portion of the SAS without the permission of the TPO.

2. Definition and number of work units involved (specify whether whole samples or fractions; and whether low, medium, or high concentration):

Monitoring well and residential wells - Twenty-five sulfate samples (includes field duplicates and field blanks).

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

Superfund remedial.

4. Estimated date(s) of collection:

February 2000

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

Daily, overnight courier (e.g., Federal Express)

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

Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

1. METHOD 375.4 Sulfate (Turbidimetric)

Note: One sulfate standard curve is used for concentrations between about 1 and 40 mg/L.

Samples will be kept at 4°C until analysis and validation of results. The holding time for sulfate samples is not to exceed 28 days from sample collection.

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Sulfate standards will be prepared daily from stock solution. Samples with absorbances or turbidities exceeding the calibration range shall be diluted and rerun. Sulfate concentrations for reanalysis dilutions for the turbidimetric method shall be in the 20 to 40 mg/L range.

For the turbidimetric method, sulfate concentrations shall be corrected (to the extent allowable) against background turbidity and color per the method and shall be clearly indicated in the raw data worksheets. If "blank" correction problems are encountered where other methods shall be used to analyze the sulfate concentration of the sample, see

Section III, "Action Required...," below.

Ion chromatography can be used as an alternate analysis method for the determination of sulfate in waters; refer to SAS Protocol 5/011A for appropriate EPA protocols and QA audits and procedures. However, prior approval is required.

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

The laboratory shall report sample all analysis results and quality control information. The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, calibration verification standards, samples, duplicates, spike samples, dilutions, laboratory control samples, etc. as specified in this SAS.

All procedures shall be clearly identified. All raw data, forms, calculations, worksheets, instrument readouts, preparation forms, internal sample and/or extract chain of custody forms, strip charts, and copies pages from preparation, weighing, and analysis notebooks shall be submitted. Submit records of weighings of all samples, blanks, duplicates and reference samples, including initial, intermediate and final weighings. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals **NOTED on the photocopies**.

All records of analysis and calculations shall be legible and be 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. For the MTB colorimetric method, records of the determination (verification) of the reagent purity shall be submitted.

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

All original sample tags, chain of custody forms, SAS packing lists, airbills, original analysis data 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. Photocopies of chain of custody forms and airbills may be submitted with a record of the location of the originals, to the Region within the time frame listed in section 6 above.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limits	Precision Desired
Sulfate	5 mg/L	Undiluted samples (5-40 mg/L) duplic. differences= ±5 mg/L
Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.		Diluted samples: ±10% RPD

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. Method 375.4

Audit	Frequency of Audits	Limits (% or Conc.)
Matrix spike[A]	at least one per group of 20 or fewer samples	60 - 140% recovery
Duplicate sample	at least one per group of 20 or fewer samples	RPD 10%
Preparation blank	at least one per group of 20or fewer samples	< 5 mg/L SO_4
Initial and continuing calibration verification standard	at least one per group of 20 or fewer samples	80 - 120% recovery
Laboratory control sample	one per 20 0or daily sample set	80 - 120% recovery [B]

[A] For the turbidimetric method: Suggested matrix spike protocols are:

Undiluted samples:

mg/L SO_4 < or = 20: Add 10 mg/L SO_4 spike

mg/L SO_4 > 20 but < 40: Dilute sample 1:1, then add 10 mg/L SO_4 spike

mg/L SO_4 > = 40: Dilute sample; see "Diluted samples" below

Diluted samples: Dilute sample so that resulting SO_4 concentration is 20 ± 2 mg/L; then add 10 mg/L SO_4 spike; Be sure to report the sample (non spiked) SO_4 concentration using a dilution that was the same magnitude (factor) as that used for the matrix spiked sample.

[A] For colorimetric method: Matrix spike concentration shall be greater than 30% of the sample concentration, but the spike sample response shall not exceed the working range of the calibration standard curve.

[B] Note that this solution is different from those used for calibration verification.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED OR CONTRACT REQUIREMENTS CANNOT BE MET:

Contact the Region.

Attachment I

DETERMINATION OF SULFATE

MODIFIED Version of METHOD 375.4 - CLP-M - Turbidimetric

1. Scope

1.1 Samples in the range of about 1 to about 40 mg SO4/L can be analyzed. Higher concentrations require the use of diluted samples.

2. Summary of Method

2.2 In the turbidimetric method, sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, spectrophotometer, or filter photometer and compared to a curve prepared from standard sulfate solutions.

3. Sample Handling and Preservation

3.1 Refrigerate at 4±2°C and submit to laboratory for analysis. Laboratory filtration is recommended for samples containing suspended or settled solid matter.

4. Interferences

4.1 Color.

4.2 Suspended matter can be removed by filtration.

4.3 Large amounts of organic material or silica concentrations greater than 500 mg/L.

Small amounts, relative to the sulfate concentration of color and/or turbidity can be corrected using the protocol described below.

5. Apparatus

5.1 Magnetic stirrer capable of adjusting stirring speed so that a constant stirring speed can be maintained without splashing should be used. Identical shape and size magnetic stirring bars should be used.

5.2 Photometer: <u>One</u> of the following should be used in order of preference.

5.2.1 Nephelometer.

5.2.2 Spectrophotometer: Capable of being used at 420 nm.

5.2.3 Filter photometer: Having a violet filter that has a maximum transmittance near 420 nm.

5.2.4 Operational Requirements: Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of that particular instrument. Sensitivity, instrument detection limit, precision, linear dynamic range, and interference effects must be investigated and determined for each instrument used.

5.3 Stopwatch or suitable electric timer.

Attachment I - continued

5.4 Measuring spoon with a 0.2 to 0.3 ml volume capacity.

5.5 Analytical balance capable of weighing to 0.1 mg.

5.6 Filtration apparatus utilizing either vacuum or pressure, having a filter support and having a large catch reservoir.

5.7 Sulfate free filter paper or membrane filter (effective 0.2 to 0.45 micron pore size).

5.8 Erlenmeyer flasks, 250 ml volume.

5.9 Suitable volumetric labware for preparation of reagent solutions, standards' solutions and sample dilutions, typically 100 ml and larger.

5.10 Graduated cylinders, 100 ml volume (to deliver).

6. Reagents

6.1 Reagent water: ASTM Type I water or equivalent

6.2 Barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) crystals, 20 to 30 mesh.

6.3 Conditioning reagent: In a 500 ml volumetric flask with magnetic stirring, dissolve 75 g of sodium chloride in 275 ml of reagent water. With stirring add 30 ml of concentrated HCl, 100 ml of 95% ethanol or isopropanol and 50 ml of glycerol. Dilute to volume with reagent water and mix well.

6.4 Standard sulfate stock solution: Dissolve 0.1479 g of dried (105°C) sodium sulfate (Na_2SO_4) in reagent water and dilute to 1 L with reagent water

6.5 Working standards: Prepare working standards by diluting the following volumes of standard solution (6.4) to 100 ml:

ml dilute SO_4 soln (6.4)	to make	mg/L SO_4 standard
0		0.0
2		2.0
5		5.0
10		10.0
20		20.0
30		30.0
40		40.0

7. Procedure

7.1 Formation of barium sulfate turbidity.

7.1.1 Transfer 100 ml of sample or a suitable portion diluted to 100 ml into a 250-ml Erlenmeyer flask.

7.1.2 Add exactly 5.0 ml of conditioning reagent (6.3).

7.1.3 Carefully introduce a clean magnetic stirring bar into the sample flask. Place flask on magnetic stirrer preset at constant speed (one that is fairly rapid but does cause splashing); do not reset speed once the run has begun.

7.1.4 While stirring add all at once, 0.2 g barium chloride crystals with the measuring spoon..

7.1.5 Stir for exactly one minute then remove the flask from the stirrer.

7.2 Calibration Curve

Ideally the standard calibration curve should be established daily and when either new batches of conditioning reagent (6.3) are prepared or if different batches or lots of barium chloride dihydrate crystals (6.2) are used. Also the curve should be constructed prior to running samples to facilitate calculating the sulfate concentration of any sample. Typical instrument (5.2) response units would be either NTU's, %T's, absorbances, or mm or cm peak heights corresponding to these response units if chart recordings are generated.

7.2.1 Prepare the working standard solutions in section 6.5.

7.2.2 For each working standard, develop the barium sulfate turbidity and measure it using steps found in sections 7.3 and 7.4 below.

7.3 Measurement of barium sulfate turbidity.

7.3.1 Immediately following the one minute stirring period, pour the mixed solution into a clean absorbance cell and measure the turbidity at 30 second intervals for four minutes using the appropriate photometer (5.2).

7.3.2 Record the maximum response reading obtained during the four minute period and use it to calculate the sulfate concentration of the sample. If any sample result is greater than the high standard, dilute the sample with reagent water and rerun.

7.4 Correction for sample color and turbidity. (**NOTE** - Correction is made using response values **not** concentration.)

7.4.1 Run a sample "blank" using a separate aliquot using procedure steps 7.1.1, 7.1.2, and 7.1.3 **without** any addition of barium chloride.

7.4.2 After stirring for a minute, pour the solution into a separate clean absorbance cell and measure the photometer (5.2) response. Record and clearly indicate this sample "blank" response value on the worksheet.

7.4.3 Preferably run the sample "blank" prior to running the sample with the barium chloride added, this would facilitate using the same absorbance cell to measure both "blank" and "sample" responses.

7.4.4 Use the "blank" response value to make the appropriate correction (see below, under section 8.2.1).

8. Calculations

8.1 Prepare a standard curve by plotting the instrument responses against the corresponding sulfate concentrations. Plot a **smooth** curve rather than a "segmented plateaus" or "point-to-point flat stepped" plot. A hardcopy plot of the resulting calibration curve should be provided.

8.2 Determine the sulfate concentration of the sample by comparing the maximum instrument value obtained (7.3.2) with the corresponding sulfate concentration indicated on the standard curve plot for that response.

8.2.1 To apply a "blank" correction, first subtract the "blank" sample response value from the "sample" response value then use the resulting "net" response value in locating a sulfate concentration from the standard curve.

8.2.2 Rules of thumb concerning application of "blank" correction:

8.2.2.0 Always be sure that the "blank" **RESPONSE** value is **CLEARLY** indicated as such in raw data records. As noted in 7.4 above, the correction is made using response values **not**

concentration.

8.2.2.1 If the "blank" response correction corresponds to a sulfate concentration of 8 or less mg/L SO_4 then use the blank correction.

8.2.2.2 If the "blank" response correction corresponds to a sulfate concentration of more than 8 mg/L SO_4 but less than 20 mg/L, then use the correction BUT be sure to report that the sample sulfate concentration as possibly being biased due to high background interference. Preferably use another analysis method for determining sulfate.

8.2.2.3 If the "blank" response correction corresponds to a sulfate concentration of 20 or more mg/L SO_4, DO NOT use the correction AND DO NOT REPORT any sulfate result for the sample, unless dilution can be used, and be sure to indicate that background interference was too high to accurately determine sulfate using the turbidimetric method. Other analysis methods should be used for determining sulfate; also contact the Region and notify them of the occurrence of high background interference.

8.2.2.4 Dilution may tried to reduce the "blank" background interference but, in addition to the above rules, DO NOT REPORT any sulfate value determined using dilution if the final sulfate calculated using the "blank" is less than 40 divided by the dilution factor. If such instances are encountered, it should be indicated that the background interference was too high to accurately determine sulfate using the turbidimetric method; other analysis methods should be used for determining sulfate; also contact the Region and notify them of the occurrence of high background interference.

8.3 Calculation Example showing use of "blank" correction:

Sample response = 20 mm peak height

"Blank" sample response = 3 mm peak height

"Net" sample response = 20 - 3 = 17 mm peak height

SO_4 concentration from calibration curve = 9 mg/L SO_4

8.4 Use the value from 8.2 and calculate:

SO_4, mg/L = (mg/L SO_4 in 8.2) (Dilution Factor)

8.5 Report sulfate result obtained in 8.4 for the sample.

9. Quality Control

The following quality control analyses (at a minimum) shall be performed for this method, instrument calibration, initial calibration blank, continuing calibration blank, and preparation blank, initial calibration verification, continuing calibration verification, duplicate sample, and laboratory control sample.

10. Bibliography

10.1 USEPA Contract Laboratory Program, Statement of Work for Water Quality Parameters in Multi-Concentration Water, WQP01.0, page D-60 (10/95)

10.2 Standard Methods for the Examination of Water and Wastewater, 18th Edition, p 4-134, Method 4500-SO_4^{2-}E, (1992).

10.3 USEPA, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 375.4, (1983).

5/014-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V		
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore	
C. Telephone Number:	(312) 353-2310	(312) 886-1488	
D. Date of Request:	October 1999	Date of Sampling : February 2000	
E. Site Name:	Evergreen Manor, Roscoe, Illinois		
Cerclis ID#	ILD984836734	Site/Spill ID#	MZ

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 delays 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 nitrite(as mg/L NO_2 - N) in waters. This method is applicable to surface waters, ground waters, drinking waters, etc. Samples will be unfiltered.

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

Twenty-five water samples will be analyzed (includes field duplicates and field blanks).

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

Superfund Remedial

4. Estimated date(s) of collection:

February 2000

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

Daily, overnight courier (e.g.,Federal Express)

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

The laboratory shall report analysis results within 35 days of receipt of last sample in shipment. Samples shall be analyzed within 7 days of collection.

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

Method 354.1 Nitrogen, nitrite (Spectrophotometric)

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

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Holding times for the water samples are 48 hours from time of collection. It is recommended that the laboratory check the samples for residual chlorine and/or sulfide using test strips such as starch iodide or lead acetate. The laboratory shall remove these interferences prior to analysis if they exist and note such presence in the case narrative. Dilute and re-analyze the neutralized samples if the sample response is greater than that of the highest standard. The laboratory may dilute the samples up to ten-fold prior to analysis so long as the final analytical working range does not fall outside 0.1 to 10.0 mg/L N. More sensitive measurements may be made at the discretion of the laboratory.

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

The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, samples, duplicates, spike samples, laboratory control samples, dilutions, re-analyses, etc.

All procedures used shall 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. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals. Any sample treatment to remove interferences shall be reported.

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):

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. Photocopies may be submitted along with a record of the location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
NO_2 N	0.1 mg/L NO_2 N	Differences in duplicate sample results are to be < ± 20% RPD

Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. Required quality control analyses for Methods 354.1

Audit	Frequency of Audits	Limits (% or Conc.)
Matrix spike[A]	at least one per group of 20 or fewer samples.	75 - 125 % recovery
Duplicate sample	at least one per group of 20 or fewer samples.	< ± 20 % RPD
Preparation blank (filtrate)	One for every 20 or fewer samples.	< 0.1 mg/l N
Laboratory control sample	one per daily sample set	85 - 115 % recovery

[A] Matrix spike concentrations shall be greater than 30% of the sample concentration but the spike sample response shall not exceed the working range of the calibration curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

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

5/016-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal

A. EPA Region/Client: Region V
B. RSCC Representative: H. Pham Technical Project Officer (TPO): C. Moore
C. Telephone Number: (312) 353-2310 (312) 886-1488
D. Date of Request: October 1999 Date of Sampling :February 2000
E. Site Name: Evergreen Manor, Roscoe, Illinois
Cerclis ID# ILD984836734 Site/Spill ID# MZ

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 delays 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 orthophosphate in waters. This method is applicable to surface waters, drinking water, groundwater, leachate, etc. Samples will be unfiltered. Results shall be reported as mg/l P. No deviation is allowed for any portion of this SAS without the permission of the TPO.

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

   Monitoring and residential wells - Twenty-five orthophosphate samples (includes field duplicates and field blanks).

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

   Superfund Remedial

4. **Estimated date(s) of collection:**
   February 2000

5. **Estimated date(s) and method of shipment:**
   Daily, overnight courier (e.g., Federal Express)

6. **Number of days analysis and data required after laboratory receipt of samples:**
   Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

   Method 365.2 (Colorimetric)
   Samples will be preserved in the field with 1 ml/l H_2SO_4 to pH $\leq$ 2, and stored at 4°C until analysis and validation of results.

   Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Dilute and redigest samples with absorbence or peak height higher than the highest standard. All standards, blanks, audits, etc. shall be digested. The holding time shall not exceed 28 days from sample collection. The calibration curve shall have five different levels of standards including a zero concentration 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.**

The laboratory shall perform data reduction and shall report sample analysis data and quality control information. The sample analysis data package shall include all documentation, data reporting forms and raw data.

All procedures used shall 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. 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 be 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 will be reported as mg/l P.

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

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. Photocopies may be submitted with a record of the location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:
Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. **Name of sampling/shipping contact:** Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
Orthophosphate	0.05 mg/L	Differences in duplicate sample results are to be < 0.05mg/L for concentrations < 0.5 mg/L and < 20% RPD for concentrations >= 0.5 mg/L.

Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. Refer Method 365.2

Audit	Frequency of Audits	Limits (% or Conc.)
Analytical Sample [A]	at least one per group of 20 or fewer samples.	80 - 120 % recovery
Duplicate Sample	at least one per group of 20 or fewer samples.	±10% RPD for conc. > 0.5 mg/l; ±0.05 for conc. < 0.5mg/l
Preparation blank (also serves as a calibration blank).	at least one per group of 20 or fewer samples.	< 0.05 mg/l
Initial calibration verification solution	at least one per group of 20 or fewer samples.	85-115% recovery
continuing calibration verification solution	at least one per group of 20 samples	85-115% recovery
Laboratory control sample	One per daily sample set	80-120% recovery
Instrument Detection Limit Determination	Per SDG	IDL ≤ CRQL

[A] Analytical spike concentrations shall be greater than 30% of the sample concentration but the spike sample response shall not exceed the working range of the calibration curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

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

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling : February 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734	Site/Spill ID# MZ

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 delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of nitrate (as mg/L NO_3 - N) in waters. This method is applicable to surface waters, ground waters, drinking waters, etc. Samples will be unfiltered.

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

Twenty-five water samples will be analyzed (includes field duplicates and field blanks).

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

Superfund Remedial

4. Estimated date(s) of collection:

February 2000

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

Daily, overnight courier (e.g.,Federal Express)

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

The laboratory shall report analysis results within 35 days of receipt of last sample in shipment. Samples shall be analyzed within 7 days of receipt if preserved, or within 24 hours if unpreserved.

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

1. Method 352.1 (Colorimetric, Brucine)

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

Samples shall be preserved in the field with 1ml/L H_2SO_4 to a pH of $<= 2$.

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Analysis should be made as soon as possible. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid and refrigerated.

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

The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, calibration verification standards, samples, duplicates, spike samples, laboratory control samples, dilutions, re-analyses, etc.

All procedures used shall 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. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals. Any sample treatment to remove interferences shall be reported.

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):

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. Photocopies may be submitted along with a record of the location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
NO_3 N	0.1 mg/L NO_3 N	Differences in duplicate sample results are to be < ±20% RPD

Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. See Methods 352.1 for required quality control.

Audit	Frequency of Audits	Limits (% or Conc.)
Analytical spike sample	at least one per group of 10 or fewer samples	75-125% recovery
Duplicate sample	at least one per group of 10 or fewer samples.	< ±20 % RPD
Preparation blank	One for every 20 or fewer samples.	< 0.1 mg/l N
Initial and continuing calibration verification standard	at least one per group of 10 or fewer samples.	90 - 110 % recovery
Continuing calibration blank	At least one per group of 10 or fewer samples.	less than the MDL
Laboratory control sample	one per daily sample set	85 - 115 % recovery

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

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

5/027-6/96

`U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client: Region V
B. RSCC Rep: H. Pham Technical Project Officer (TPO): C. Moore
C. Telephone Number: (312) 353-2310 (312) 886-1488
D. Date of Request: October 1999 Date of Sampling : February 2000
E. Site Name: Evergreen Manor, Roscoe, Illinois
Cerclis ID# ILD984836734 Site/Spill ID# MZ

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 delays 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 percent (%) organic carbon in soil (air-dried, passed through a 100 or a 140 mesh screen). Applicable concentration range is 0.1% to 10.0% or more. Results will be reported as % organic carbon, dry weight. Percent solids data for each soil sample will be included. Total carbon shall be measured using sub-sample aliquots 50 mg or larger. No deviation is allowed for any portion of this SAS without the permission of the TPO.

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

Soil - Eighteen total organic carbon (includes field duplicates)

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

Superfund remedial.

4. Estimated date(s) of collection:

February 2000

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

Daily, overnight courier (e.g., Federal Express)

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

Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

Use method 9060 as modified in the attachments.

Note: Test for the presence of inorganic carbon (Attachment II, Section 2) before the determination of organic carbon.

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

See attachment III. Note that although attachment III recommends certain QC audits, the QC audits listed on page 3 of this SAS are **required**. Also, total carbon measurements shall be made using air dried sample aliquots 50 mg or larger.

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 crossouts and/or notations made on the raw data shall be initialed and dated. Submit all original percent solids data including; initial, intermediate and final weights of all samples and final percent solids results. 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 complete, 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.

Note: Analysis is performed on an air-dried aliquot; results are therefore reported as % organic carbon, dry weight.

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

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. Photocopies may be submitted with a record of the location of the originals.

Payment of laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
% organic carbon in soil	0.10%	Differences in duplicate sample results shall be +/- 20%.

Note: These are minimum requirements. Report actual detection limit(s) based on allowable methodology options.

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

Audit	Frequency of Audits	Limits (% or Conc.)
Preparation blank*	One per group of 10 or at least twice.	< = 0.1%
Matrix Duplicate*	One per group of 10 or fewer samples.	< = 20% RPD in duplicate sample results or <0.2% organic carbon difference at concentrations <0.5%
Matrix spike*	One per group of 10 or fewer samples.	75 - 125% recovery
Positive control (to be determined by the laboratory, i.e. LCS)*	One per group of 10 or fewer samples.	85 - 115% recovery
Calibration Verification and Calibration blanks (if applicable)*	One per group of 10 or fewer samples.	90 - 110% recovery for calibration checks and <0.1% organic carbon for blanks.

*Please see Attachment V.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

ATTACHMENT I

Determination of % organic carbon in soil uses sub-aliquots of air-dried soil passed through a 100 mesh or a 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 2% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil shall be the 1) Dry combustion (resistance furnace); 2) Dry combustion (induction furnace); 3) Dry combustion (automated methods); or 4) Wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses", Part 2 - Chemical and Microbiological properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because no laboratory doing organic carbon analysis of soil should be without it.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd, ed., as appropriate. Soils can be calcareous or non-calcareous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory will provide information within the case narrative concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items #8 and #9 of this SAS.

ATTACHMENT II

Analytical Methods - Organic Carbon in Soil

1. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.

2. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (5 min). If organic carbon is absent, proceed with Total Carbon in items #7c, or 7d of MSA, Part II, Section 29-3.3.1. If inorganic carbon is present, or the test is not definitive, proceed with items #7e, or #7f of MSA, Part II, Section 29-3.3.1 prior to Total Carbon measurements in Item #7c or #7d of MSA, Part II, Section 29-3.3.1.

3. Total Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:

   (a) Total Carbon (Dry Combustion - Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.

   (b) Total Carbon (Dry Combustion - High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.

   (c) Total Carbon (Dry Combustion - Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3 and 29-2.2.4.

   Note: Total Carbon instruments designed for TOC in Water (Method 415.1) may be too sensitive. Routinely observed TOC concentrations in the range of 1% to 5% require sample aliquots less than 2 mg. These small aliquot weights will severely effect accuracy of Total Carbon data. Instruments using an air-dried sample aliquot of 50 mg or larger should be used.

4. Total Carbon (Wet Digestion), MSA, part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing $K_2C_5O_7$). CO_2 evolved is absorbed and weighed, or absorbed in standard base and titrated.

   1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.

5. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H_2SO_3). After several hours, remove the excess H_2SO_3 by leaving overnight in an evacuated desiccator. Read citation for further details.

6. Pretreatment prior to Wet Digestion, MSA, Part II, Section 29-3.3.2. Inorganic carbon is removed by sulfuric acid - ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

7. Use only the methods specified above or obtain approval of the Region prior to use of other method. Test procedure description, and description of specific measurement principles including equivalency to each of the 10 items of Figure 29-1 of MSA, part II and sample pretreatments of Section 29-3, MSA, Part II.

ATTACHMENT II (continued)

Analytical Methods - Organic Carbon in Soil

8. Laboratory performing Total Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in samples.

ATTACHMENT III

A variety of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide a calibration curve (even if it is external calibration), including a zero concentration standard. Provide a positive control (a test sample prepared independently from calibration standards) that provides a measure of the accuracy of the system. This should be done for all systems including gravimetric read-outs.

When using an automated analytical system, the following calibration procedure must be used and documented in the raw data and on summary sheets:

1. Calibrate the instrument using a zero standard and four non-zero standards over the appropriate calibration range.

2. Analyze the zero standard and the four calibration standards.

3. If the calibration standard concentrations are ± 5% of the 'true value' for the mid-range and high level standards and ± 10% for the low to mid-range standards, the curve can be used.

4. If the standards are outside of the windows in #3 above, the laboratory must again calibrate the instrument, and repeat steps 2-3. If the laboratory cannot meet the criteria specified above, contact the Region immediately.

If the EPA QC check sample results (or the independently prepared positive control sample results) are out of control, analyses must be halted, corrective action must be taken, and sample analysis must be repeated.

ATTACHMENT IV

Analytical Results Required

As part of the Case Narrative, attach a description of the test procedure and instrumentation used for the measurement of Total Carbon and removal of any Inorganic Carbon. Test procedure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In the Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and steps taken to resolve any problems. A sample preparation log must be provided for all samples whether or not pretreatment for the presence of inorganic carbon was necessary.

Bench records tabulating any order of any sample weights and tare weights of absorbed CO_2, instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be complete and legible. Report results as % organic carbon, dry weight. Also report the percent solid for each sample.

Note: Do not correct the sample result for percent solid; the analysis uses a dried sample aliquot.

ATTACHMENT V

DEFINITION OF QC AUDIT TERMS

1.	Matrix Spike:	The sample is spiked before pretreatment (ie. digestion, distillation etc.) and subsequent analysis.
2.	Lab Spike:	The sample is spiked after pretreatment at the time of analysis. If pretreatment is not necessary the sample is simply spiked and analyzed.
3.	Matrix Duplicate:	The sample is split into duplicate aliquots and pretreated (ie. digestion, distillation etc.) for subsequent analysis.
4.	Lab Duplicate:	The sample is analyzed in duplicate after pretreatment at the time of analysis. If pretreatment is not necessary the sample is simply analyzed in duplicate.
5.	Preparation Blank:	Laboratory reagent water pretreated as a sample.
6.	Lab Blank:	Laboratory reagent water (without pretreatment) analyzed as a sample.
7.	Calibration Blank:	Analysis of calibration zero standard to verify baseline.
8.	Calibration Verification:	A control standard prepared at a known concentration on the calibration curve (generally, a mid-range concentration).
9.	Laboratory Control Standard (LCS):	A control standard, with a certified concentration, treated as a sample (including pretreatment, if necessary).

5/019-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling : February 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734	Site/Spill ID# MZ

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 delays 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:** No deviation is allowed for any portion of the SAS without the permission of the TPO.

   Analysis of chemical oxygen demand (COD); (50-800 mg/L) analyses of water samples. This method is applicable to surface water, groundwater, wastewater, drinking water, leachate, etc. Samples will be unfiltered. The analysis uses Method 410.1

   If field specific conductance values are greater than or equal to 5000 μmhos/cm, field personnel shall indicate the values on the SAS Packing Lists with the instruction "Check for Possible Chloride Interference in COD Test".

   Results shall be reported as mg/L COD.

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

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

   Monitoring and residential wells - Twenty-five COD (includes field duplicates and field blanks).

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

   Superfund remedial.

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

   February 2000

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

   Daily, overnight courier (e.g., Federal Express)

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

Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

1. Method 410.1-CLP-M (Titrimetric) or Method 5220B (HACH),.

Samples shall be preserved with 1 mL/L of H_2SO_4 to pH < 2 and kept at 4°C until sample analysis and validation of results are completed. Holding time is not to exceed 28 days from date of sample collection.

Note: Laboratory data rejection and non-payment shall be recommended if methods other than those specified in this document are used.

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

1. Check sample pH (wide range pH paper can be used). If pH > 2, contact the Region for further instructions.
2. Use 50 mL sample aliquots. Use 0.25 N ferrous ammonium sulfate (FAS) titrant for mid level analysis.
3. Dilute and re-analyze any samples with COD values > 800 mg/L or titrant volumes < 5.0 mL.
4. Any sample aliquots < 50 mL shall be diluted to 50 mL with reagent water.
5. Titration blanks shall be determined at least in duplicate on each day of analysis and shall not differ by more than ± 0.1 mL FAS.
6. Use potassium hydrogen phthalate as the matrix spike compound.
7. Samples shall be refluxed for at least two hours.
8. Homogenize sample aliquots, as necessary, to obtain representative sample aliquots.

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 shall be left to program discretion.

The laboratory shall perform data reduction and shall report sample analysis data and quality control information. The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, calibration verification standards, samples, duplicates, spikes, dilutions, re-analyses, and laboratory control samples, etc.

All procedures used shall 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. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

Bench records of titrant standardization, titration volumes for sample blanks and samples, and QA audits, shall be provided for each method used. All records for chloride analysis shall be provided for any samples so specified on the SAS Traffic Report or SAS packing List. Separate bench records shall be provided for any COD determinations of high chloride samples (> 2000 mg/l) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type.

All records of analysis and calculations shall be legible and be 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):

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. Photocopies may be submitted along with a record of the location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
COD (mid level) **Note:** These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.	50 mg/L	Differences in duplicate sample results are to be < = 50 mg/L for concentrations < 250 mg/L and < ±20% RPD for concentrations > = 250 mg/L.

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

Audit	Frequency of Audits	Limits (% or Conc.)
Matrix Spike (KHP)[A]	One per group of 20 or fewer samples.	80 - 120% recovery
Duplicate sample	One per group of 20 or fewer samples.	Difference < 50 mg/L or 20% RPD (410.1)
Preparation blank	One per group of 20 or fewer samples	< 5 mg/L (low level); < 50 mg/L (high level)
Titration Blank	Two per sample set for each method used.	Difference in FAS titrant volumes not to exceed 0.1 mL.
Laboratory control sample	One per sample set for each method used.	85 - 115% recovery
Reflux (Reagent) Blanks	Two per sample set for each method used.	<5 mg/L (low level); <50 mg/L (mid level)

[A] Matrix spike concentrations shall be greater than 30% of the sample concentration but the spike level shall not exceed 800 mg/L for mid level COD analyses.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

5/013 - 6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SM-5J
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling :February 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734	Site/Spill ID# MZ

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 delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of ammonia in waters. This method is applicable to surface waters, ground waters, drinking waters, etc. All samples will be unfiltered. Results will be reported as mg/l NH_3. No deviation is allowed for any portion of this SAS without the permission of the TPO.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether aqueous or soil and sediments; and whether low, medium, or high concentration):
Monitoring and residential wells - Twenty-five ammonia samples (includes field duplicates and field blanks).

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

4. Estimated date(s) of collection:
February 2000

5. Estimated date(s) and method of shipment:
Daily, overnight courier (e.g., Federal Express)

6. Number of days analysis and data required after laboratory receipt of samples:
Data packages are due within 35 days after the receipt of the last sample in a sample delivery group.

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

1. Method 350.2 (Colorimetric, Titrimetric Potentiometric Distillation)

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

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

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

Check sample pH with wide range pH paper. If pH > 2, contact Region for instructions. Dilute and re-analyze

samples with concentrations higher than the highest calibration standard. The holding time is not to exceed 28 days from sample collection. All solutions should be made with ammonia-free water.

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.

The laboratory shall perform data reduction and shall report sample analysis results and quality control information. The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, duplicates, spike samples, laboratory control samples, dilutions, and re-analyses, etc.

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 - NH_3-N.

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

Payment of laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:
Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired
Ammonia	0.1 mg/l -NH_3	Differences in duplicate sample results are to be < 0.1mg/l for concentrations < 1mg/l and < ±20% RPD for concentrations > = 1mg/l. Report results to the nearest 0.05mg/l for conc.'s < 1mg/l and 2 sig. figures for conc.'s > = 1 mg/l.
Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. Required quality control analyses are listed in section 9 of each analysis method.

Audit	Frequency of Audits	Limits (% or Conc.)
Analytical sample[A]	at least one per group of 20 or fewer samples.	75 - 125 % recovery
Duplicate sample	at least one per group of 20 or fewer samples.	±20 % RPD or 0.1 mg/l
Preparation blank	at least one per group of 20 or fewer samples.	< = 0.1 mg/l-N
Laboratory control sample	one per daily sample set	85 - 115 % recovery

[A] Spike sample concentrations shall be greater than 30% of the sample concentration but the spike sample response shall not exceed the working range of the calibration curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact Region V.

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

U.S. Environmental Protection Agency Region V
77 West Jackson, SM- 5J
Chicago, IL 60604
PHONE: (312) 353-2310 FAX: (312) 886-0186

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V	
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore
C. Telephone Number:	(312) 353-2310	(312) 886-1488
D. Date of Request:	October 1999	Date of Sampling : February 2000
E. Site Name:	Evergreen Manor, Roscoe, Illinois	
Cerclis ID#	ILD984836734	Site/Spill ID# MZ

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 delays 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 particle size in soil samples. See Attachment 1 for the format of the results. No deviation is allowed for any portion of the SAS without the permission of the TPO.

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

Eighteen samples will be analyzed.

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

Superfund Remedial

4. Estimated date(s) of collection:

February 2000

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

Daily, Overnight courier (e.g., Federal Express)

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

Laboratory should report results within 35 days of receipt of last sample.

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

ASTM D-422, Particle Size Analysis of Soils
ASTM D-421, Sample Preparation for Particle Size Analysis

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

1.) Air dry entire sample and record the air-dried weight. If the sample contains large pieces of material other than soil, such as wood, leaves, leather, etc., this material shall be removed by the analyst after the original air-dried weight has been recorded. A short written description of the material removed shall be recorded. The weight of the material removed shall be recorded. Record net weight of air-dried soil submitted.

NOTE: Sample is air-dried by spreading out sample in an aluminum pan or a crucible to dry. Length of time to air dry sample (24 hours to possibly several days) will depend on type of soil received and its moisture content. Final air dried soil shall have < 5% moisture.

2.) The air-dried soil is prepared for mechanical analysis as described in ASTM D-421.

3.) ASTM D-422 gives minimum sample sizes for samples with nominal diameters of the largest particles ranging from 3/8" to 3". Furthermore, these samples as well as samples with the largest particles less than 3/8" require a minimum sample size of 115 grams for sandy soils and 65 grams for silt/clay to yield portions passing a No. 10 sieve. These are minimum amounts, more can be used if possible. If there is insufficient sample size, it is acceptable to use the entire sample for analysis to meet these minimum requirements.

4.) If these minimum requirements are not met due to insufficient sample size or if the total sample weight after drying is less than 200 grams, contact the Region immediately. Include the following information: sample number, sample weight and required sample weight. The Region will determine whether to proceed with or terminate analyses.

5.) COARSE FRACTION: Perform a sieve analysis of the portion retained on the No. 10 sieve according to the ASTM test method using the following sieves - 3" (75 mm), 2" (50 mm), 1 1/2" (37.5 mm), 1" (25 mm), 3/4" (19.0 mm), 3/8" (9.5 mm), No. 4 (4.75 mm) and No. 10 (2.0 mm).

6.) FINE FRACTION: Perform a sieve analysis of the portion that passes a No. 10 sieve according to ASTM D-422 using the following sieves - No. 20, No. 40, No. 60, No. 80, No. 100 and No. 200.

7.) HYDROMETER TESTING: Perform hydrometer, hygroscopic moisture, and specific gravity analysis on the fraction that passes a No. 10 sieve. Give a written description of the portion passing the No. 10 sieve. If sandy, use 100 grams for hydrometer analyses, and if clay/silt, use 50 grams for hydrometer analysis.

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

1.) Report results as percent finer than the specified particle size. Present data as tabulated AND in the form of a grain-sized distribution curve on a semi-logarithmic chart with percent finer by weight plotted on the arithmetic scale and grain size plotted on the logarithmic scale.

2.) All hand-written raw data shall be submitted on Attachment 1. This is mandatory. The laboratory shall also supply any and all supporting raw data report forms from which data is reported on the Attachment 1 form.

3.) If the laboratory uses a computer program or spreadsheet to calculate particle size and/or hydrometer values, several factors will have to be taken into account by the laboratory. The formulas used in the computer calculations may not be the same as in the ASTM methods, but both may be

mathematically equivalent. The constants (K, L and specific gravity correction factors) might be calculated from the complex formulas rather then extrapolated from the tables in ASTM D422. The laboratory is required, if a computer program or spread sheet is used, to show that all formulas in the program are mathematically equivalent to the ASTM formulas and also to show by sample calculation where all the values used in the formula came from. This can be done in the case narrative.

4.) All calculations may be performed using a computer generated speadsheet but both handwritten raw data AND computer spreadsheets shall be submitted as case deliverables.

5.) If the laboratory uses a computer generated report form in place of Attachment 3, it shall contain the following:

A. Total air-dried weight of sample submitted.
B. Total air-dried weight of extraneous material removed.
C. Total air-dried weight of soil submitted (A - B).
D. Total weight of soil retained on a No. 10 sieve.
E. Total weight of soil passing a No. 10 sieve.
F. Complete coarse fraction sieve analysis.
G. Complete fine fraction sieve analysis.
H. Complete hydrometer analysis including date and time, temperature, hydrometer readings (actual and corrected, hydrometer composite correction, and type of hydrometer used.
I. Complete hygroscopic moisture analysis.
J. Complete specific gravity analysis.
K. Written description of extraneous material removed (if any), and of material passing the No. 10 sieve (sand or clay/silt)

6.) A copy of the graph the laboratory used to determine the hydrometer composite correction , based on temperature change, must be supplied in the data package, along with the date determined.

7.) All procedures used shall 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. Submit records of the weighings of all samples and duplicate samples including initial and final weighings. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

8.) All records of analysis and calculations shall be legible and be 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):

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.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Kurt Fischer
Roy F. Weston, Inc
3 Hawthorn Parkway Suite 400
Vernon Hills, Illinois 60060
(847) 918-4055 (fax)

11. Name of sampling/shipping contact: Tonya Balla
Phone: (847) 918-4094

I. DATA REQUIREMENTS

Parameter:	Detection Limit:	Precision Desired:
% finer (or passing)	1 %	Differences in duplicate sample results for sieve analyses are to be < 35 % RPD or a difference of ± 1.0% for values less then 2.0 %. There are no QC limits for Hydrometer testing but a duplicate is to be assayed for comparison only.
Note: These are minimum requirements. Report actual detection limit(s) used, based on allowable methodology options.		

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

Audit	Frequency of Audits	Limits (% or Conc.)
Duplicates	1 per 10 or fewer samples	SIEVE ANALYSES:
NOTE: Duplicate RPD data is calculated using the % soil retained in each sieve.		Each fraction: <35% RPD, or a difference of ± 1.0 % for values less then 2.0 %. HYDROMETER ANALYSES:
		NO QC LIMITS but a duplicate is to be assayed for comparison only.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact the Region.

ASTM D-422 Particle Size Analysis of Soils

ATTACHMENT 1

Laboratory:_____

EPA Sample No.:_____

Analyst:_____

Lab Sample No.:_____

Sample Preparation:

Total weight air-dried sample:_____Grams
Total weight air-dried extraneous material:_____Grams
Total weight air dried soil:_____Grams
Total weight soil retained No. 10 sieve:_____Grams
Total weight soil passing No. 10 sieve:_____Grams

% Retained No. 10 sieve:_____%

% Passing No. 10 sieve:_____%

% Passing No. 10 sieve description (sandy or clay/silt):_____

Extraneous material description:_____

Sieve Analysis, Date:	Sieve Size	Weight retained, grams
Coarse:	3"	
	2"	
	1 1/2"	
	1"	
	3/4"	
	3/8"	
	No. 4	
	No. 10	
Fine:	No. 20	
	No. 40	
	No. 60	
	No. 80	
	No. 100	
	No. 200	

HYDROMETER ANALYSIS: DATE:_____

TYPE OF HYDROMETER:_____

TIME (min)	ACTUAL TIME	HYDROMETER ACTUAL	HYDROMETER COMP. CORR.	CORRECTED HYDROMETER	TEMP. C
2					
5					
15					
30					
60					
250					
1440					

HYGROSCOPIC MOISTURE ANALYSES:

Tare:_____g
Sample Weight:_____g
Tare + Dry Weight:_____g
Dry Weight:_____g
Per Cent Moisture:_____%

SPECIFIC GRAVITY ANALYSES:

Pycnometer wt.:_____g
Pycnometer + water:_____g
Pycnometer + water + soil:_____g
Temperature, °C:_____
Specific Gravity:_____

APPENDIX C

CHEMTECH LABORATORY SOPS

NITRITE NITROGEN

REFERENCE

Methods for Chemical Analysis of Water and Wastes, method 354.1.

SAFETY

All samples submitted to an environmental laboratory should be treated as potential health hazards.

DEFINITIONS

Field Sample: All samples submitted by the client, including field quality control samples such as field blanks and trip blanks.

MS: Matrix spike.

Quality Control Sample: Samples prepared at the laboratory for quality control purposes, including method blanks, matrix spikes, replicates, blank spikes, *etc.* Calibration standards are not included.

RPD: Relative percent difference.

SCOPE AND APPLICATION

Analytes:	Nitrite
Matrices:	Water
Regulations:	NPDES

PROCEDURE

Initial Calibration

1. Prepare the following standards in a series of Nessler tubes.

Nitrate Standard Solution (mL)	Reagent Water (mL)	Concentration (mg NO_2-N/L)
0.0	50.0	Blank
0.5	49.5	0.01
1.0	49.0	0.02
1.5	48.5	0.03
2.0	48.0	0.04
3.0	47.0	0.06
4.0	46.0	0.08
5.0	45.0	0.10
10.0	40.0	0.20

2. Analyze the standards as for samples below.

3. Prepare a standard curve by plotting the absorbance of each standard against the concentration (NO_2-N/L) for each standard.

Sample Analysis

1. If the sample has a pH greater than 10 or a total alkalinity greater than 600 mg/L, adjust the sample pH to 6 with Diluted Hydrochloric Acid (1:3).

2. If necessary, filter the sample through a 0.45 μm filter using the first portion of filtrate to rinse the filter flask.

3. Place 50 mL of sample, or an aliquot diluted to 50 mL, in a 50-mL Nessler tube.

4. Add 2 mL of Buffer-Color Reagent to each standard and sample, mix, and allow color to develop for at least 15 minutes. The color reaction medium should be between pH 1.5 and 2.0

5. Read the absorbance at 540 nm against the blank.

6. Read the sample concentration directly from the curve. If the sample was diluted, correct for dilution:

$$\textit{Corrected concentration} = \frac{50 \times \textit{concentration from curve}}{\textit{mL of sample used}}$$

Quality Control Samples

1. Method blank

   a. Prepare one method blank each time samples are analyzed by this procedure, or one per 20 samples analyzed by this procedure, whichever is more frequent.

   b. The blank must be free of NO_2-N at or above the MDL.

2. MS

   a. Prepare one MS per 20 samples analyzed by this method, or one each 30 days, whichever is more frequent.

   b. Preparation and nominal concentration

      Add 0.25 mL of Nitrite Stock Solution to about 25 mL of pH corrected sample in a 50-mL volumetric flask. Dilute to volume with sample and transfer to a 50-mL Nessler tube. Analyze as for samples. The spike added concentration is 0.05 mg NO_2-N/L.

      If the client has designated a sample to be spiked, the analyst must use the designated sample to prepare the MS. Do not spike any field quality control samples.

   c. Calculate the percent recovery for the MS.

$$\%Recovery = \frac{\text{spiked sample result} - \text{sample result}}{\text{spike added}} \times 100$$

3. Duplicate

   a. Prepare one duplicate per 20 samples analyzed by this method, or one each 30 days, whichever is more frequent.

   b. If the client has designated a sample to be duplicated, the analyst must use the designated sample to prepare the duplicate. Do not prepare the duplicate from any field quality control samples.

   c. Calculate the RPD.

$$RPD = \frac{|\text{original sample result} - \text{duplicate sample result}|}{\frac{1}{2} \times (\text{original sample result} + \text{duplicate sample result})} \times 100$$

REAGENTS

1. Reagent Water: deionized water in which nitrite nitrogen is not detected at or above the MDL.

2. Chloroform.

3. Hydrochloric Acid: reagent grade.

4. *N*-(1-Naphthyl)ethylenediamine Dihydrochloride.

5. Sodium Acetate.

6. Sodium Nitrite: anhydrous, reagent grade. Dry in desiccator 24 hours before use.

7. Sulfanilamide.

8. Buffer-Color Reagent: Transfer 105 mL of Hydrochloric Acid, 5.0 g o f Sulfanilamide, and 0.5 g of *N*-(1-Naphthyl)ethylenediamine Dihydrochloride to a 500-mL volumetric flask containing about 250 mL of Reagent Water. Stir until dissolved. Add 136 g of Sodium Acetate and stir again until dissolved. Dilute to volume with Reagent Water. Shelf life is one month when stored in dark.

9. Diluted Hydrochloric Acid (1:3): Slowly add 100 mL of Hydrochloric Acid to 300 mL of Reagent Water in a 500-mL beaker with constant stirring.

10. Nitrite Stock Solution: 0.10 mg N/mL. Transfer 0.1493 g of Sodium Nitrite to a 1000-mL volumetric flask containing about 900 mL of Reagent Water. Dissolve the Sodium Nitrite, add 2 mL of Chloroform, and dilute to volume with Reagent Water.

11. Nitrite Standard Solution: 0.001 mg N/mL. Transfer 10.0 mL of Nitrite Stock Solution to a 100-mL volumetric flask containing about 50 mL of Reagent Water. Dilute to volume with Reagent Water.

CHEMTECH

EPA Method 352.1

NITROGEN, NITRATE

SCOPE AND APPLICATION

1. This method is applicable to the analysis of ground water, drinking, surface, and saline waters, and domestic and industrial wastes Modification can be made to remove or correct for turbidity, color, salinity, or dissolved organic compounds in the sample
2. The applicable range of concentration is 0.1 to 2 mg NO_3-N per liter of sample.

SUMMARY OF METHOD

This method is based upon the reaction of the nitrate ion with brucine sulfate in a 13 N H_2SO_4 solution at a temperature of 100°C. The color of the resulting complex is measured at 410 nm. Temperature control of the color reaction is extremely critical

INTERFERENCES

1. Dissolved organic matter will cause an off color in 13 N H_2SO_4 and must be compensated for by additions of all reagents except the brucinesulfanilic acid reagent. This also applies to natural color, not due to dissolved organics, that is present.
2. Strong oxidizing or reducing agents cause interference.
3. Ferrous and ferric iron and quadrivalent manganese give slight positive interferences for concentrations more than 1 mg/L.
4. Uneven Heating of the samples and standards during the reaction time will result in erratic values. The necessity for absolute control of temperature during the critical color development period cannot be too strongly emphasized.

APPARATUS AND MATERIALS

1. Spectrophotometer or filter photometer suitable for measuring absorbance at 410 nm
2. Sufficient number of 40- to 50-mL glass sample tubes for reagent blanks, standards, and samples.
3. Neoprene-coated wire racks to hold sample tubes.
4. Water bath suitable for use at 100°C. This bath should contain a stirring mechanism so that all tubes are at the same temperature and should be of sufficient capacity to accept the required number of tubes without a significant drop in temperature when the tubes are immersed.
5. Water bath suitable for use at 10-15°C.

REAGENTS

1. Distilled water free of nitrite and nitrate.
2. Sodium chloride solution (30%). Dissolve 300 g NaCl in distilled water and dilute to 1 liter.
3. Sulfuric acid solution: Carefully add 500 mL concentrated H_2SO_4 to 125 mL distilled water. Cool and keep tightly stoppered to prevent absorption of atmospheric moisture.
4. Brucine-sulfanilic acid reagent: Dissolve 1 g brucine sulfate -- $(C_{23}H_{26}N_2O_4)_2*H_2SO_4*7H_2O$ -- and 0.1 g sulfanilic acid ($NH_2C_6H_4SO_3H*H_2O$) in 70 mL hot distilled water. Add 3 mL concentrated HCl, cool, mix, and dilute to 100 mL with distilled water. Store in a dark bottle at 5°C. This solution is stable for several months; the pink color that develops slowly does not affect its usefulness. Mark bottle with warning,

   "CAUTION: Brucine Sulfate is toxic; do not ingest."
5. Potassium nitrate stock solution (1.0 mL = 0.1 mg NO_3-N): Dissolve 0.7218 g anhydrous potassium nitrate (KNO_3) in distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 mL chloroform per liter. This solution is stable for at least 6 months.
6. Potassium nitrate standard solution (1.0 mL = 0.001 mg NO_3-N). Dilute 10.0 mL of the stock solution (5.5) to 1 liter in a volumetric flask. This standard solution should be prepared fresh weekly.
7. Acetic acid (1+3): Dilute 1 volume glacial acetic acid (CH_3COOH) with 3 volumes of distilled water.
8. Sodium hydroxide (1 N): Dissolve 40 g of NaOH in distilled water. Cool and dilute to 1 liter.

PROCEDURE

1. Check the sample log sheet(s) and the sample(s) and report any inconsistencies to the supervisor.
2. Adjust the pH of the samples to approximately 7 with acetic acid or sodium hydroxide. If necessary, filter to remove turbidity. Sulfuric acid can be used in place of acetic acid, if preferred.
3. Set up the required number of sample tubes in the rack to handle reagent blank, standards, and samples. Space tubes evenly throughout the rack to allow for even flow of bath water between the tubes. This should assist in achieving uniform heating of all tubes.
4. If it is necessary to correct for color or dissolved organic matter which will cause color on heating, run a set of duplicate samples to which all reagents, except the brucine-sulfanilic acid, have been added.
5. Pipet 10.0 mL of standards and samples or an aliquot of the samples diluted to 10.0 mL into the sample tubes.
6. If the samples are saline, add 2 mL of the 30% sodium chloride solution to the reagent blank, standards, and samples. For freshwater samples, sodium chloride solution may be omitted. Mix contents of tubes by swirling and place rack in cold-water bath (0-10°C).
7. Pipet 10.0 mL of sulfuric acid solution into each tube and mix by swirling. Allow tubes to come to thermal equilibrium in the cold bath. Be sure that temperatures have equilibrated in all tubes before continuing.

8. Remove rack of tubes from the hot-water bath, immerse in the cold water bath, and allow to reach thermal equilibrium (20-25°C).
9. Add 0.5 ml of brucine-sulfanilic acid reagent to each tubeand carefully mix by swirling, then place the rack of tubes in the 100°C water bath for exactly 25 minutes
   Caution: Make sure that the water bath temperature is not less than 98°C
10. Read absorbance against the reagent blank at 410 nm using a 1-cm or longer cell.
11. Run the prepared calibration standards.
12. Run the statistical check for the instrument calibration. Make sure that the calculated regression constant is equal to or greater than 0.995.
13. Verify the instrument calibration with an independently prepared check standard after instrument calibration.
14. Run the sample analysis.
15. Correct the absorbance of the sample without the brucine sulfanilic reagent.
16. Run the sample dilution for the OVER CALIBRATION range analytical results.
17. Run the required quality checks and report the inconsistencies of the quality checks to a supervisor.

QUALITY CONTROL

Unless otherwise specified, the following Chemtech Quality Control criteria must be followed.

1. All quality control data should be maintained and available for easy reference or inspection
2. Employ a minimum of one blank sample for a batch of 20 samples or less.
3. Verify calibration (midrange prepared CCV) for every ten or less sample analysis. The analytical result should be within 90% to 110% of the actual value.
4. Verify blank CCB standard for every ten or less sample analysis. The analytical result should be less than the MDL or the CRDL.
5. Run the CCV & CCB at the end of each analytical batch samples.
6. Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process as required. The RPD for the duplicate sample should be within the 85% to 115% range.
7. Run one spike sample for every 10 samples. The spike recovery should be within 75% to 125% of the actual spike value.

Approved by: _____ Date: 7/13/98
Technical Director

TEST NAME: AMMONIA (DISTILLATION)

REFERENCE

EPA Methods for Chemical Analysis of Water and Wastes, March 1983, Method 350.2 (Colorimetric; Titrimetric; Potentiometric - Distillation Procedure).

MDL - 0.10 mg/l

SCOPE AND APPLICATION

-This distillation method covers the determination of ammonia-nitrogen in drinking, surface and saline waters, domestic and industrial wastes.

-The method covers the range from about 0.10 mg/l to 1.00 mg NH_3-N/L for the colorimetric procedure, from 1.0 to 25 mg/l for the titrimetric procedure.

SUMMARY OF METHOD

The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization, or titrimetrically with standard sulfuric acid with the use of a mixed indicator. The choice between the first two depends on the concentration of ammonia.

PRESERVATION AND HANDLING

Preserve by addition of 2ml conc. H_2SO_4 per liter and refrigerate to 4^oC.

HOLDING TIME

28 days from time of sampling.

INTERFERENCES

-A number of aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (without distillation), has been discarded as an official method.

-Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at pH 9.5 at which distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesslerization in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerization.

-Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

APPARATUS

-distillation apparatus with 800ml flasks
-Spectrophotometer for use at 425nm and providing a light path of 1cm.
-Volumetric flasks, 50ml
-Erlenmeyer flasks, 500ml

REAGENTS

-Stock ammonium solution - 1.0ml = 1.0mg NH_3-N. Dissolve 3.819g NH_4Cl in deionized water and bring to volume in a 1l volumetric flask.

-Standard ammonium solution - 1.0ml = 0.01mg NH_3-N. (10ppm) Dilute 10.0ml of stock solution to 1 liter in a volumetric flask.

-Boric acid solution - Dissolve 20g H_3BO_3 in deionized water and dilute to 1 liter.

-Mixed indicator - Mix 2 volumes of 0.2% methyl red in 95% ethyl alcohol with 1 volume of 0.2% methylene blue in 95% ethyl alcohol. Prepare fresh every 30 days.

-Nessler reagent - Dissolve 100g mercuric iodine and 70g potassium iodide in a small amount of deionized water. Add this mixture with stirring to a cooled solution of 160g of NaOH in 500ml of deionized water. Dilute the mixture to 1 liter. Stable, if stored in pyrex bottle out of direct sunlight, for one year.

-Borate buffer - Add 88ml 0.1 N NaOH solution to 500ml of 0.025M sodium tetraborate solution (5.0 anhydrous $Na_2B_4O_7$ or 9.5g $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute to 1 liter.

-Sulfuric acid, standard solution - 0.02N

-Sulfuric acid, standard solution 0.10N. Prepare a stock solution of approximately 0.1N acid by diluting 3ml of conc. H_2SO_4 to 1 liter with deionized water. Standardized against 0.05N Na_2CO_3 solution, as in alkalinity SOP. By proper dilution, the standard 0.02N acid can be prepared.

-Sodium Hydroxide, 6N - dissolve 240g in liter deionized water.

-Sodium thiosulfate - dissolve 3.5g $Na_2S_2O_3 \cdot 5H_2O$ in deionized water and dilute to 1 liter. One ml of this solution will remove 1 mg/l of residual chlorine in 500ml of sample.

PROCEDURE

1. Preparation of equipment: Add 500ml of deionized water to an 800ml Kjeldahl flask. The addition of boiling chips which have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.

2. Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 400ml of sample add 6N NaOH until the pH is 9.5, using a pH meter.

3. Distillation: Transfer the sample to an 800ml Kjeldahl flask and add 25ml of borate buffer. Distill 300ml at the rate of 6-10 ml/min. into 50ml of boric acid solution contained in a 500ml erlenmeyer flask. The extension of condenser tip must be below the level of the boric acid solution. Dilute the distillate to 500ml and nesslerize an aliquot to obtain an approximate value of the NH_3-N concentration. For concentrations above 1 mg/l the ammonia should be determined titrimetrically. For concentrations below this value, it is determined colorimetrically. (ICM will usually use the colorimetric method for all ammonia distillations unless otherwise requested).

4. Determination of ammonia in distillate:
a. Titrimetric: Add 3 drops of the mixed indicator to the distillate and titrate the ammonia with 0.02N H_2SO_4, matching the end point against a blank containing the same volume of deionized water and H_3BO_3 solution.
b. Colorimetric determination: Prepare a series of standards in 50ml volumetric flasks as follows:

mls of std. 10 mg/l sol.	mg/l NH_3-N
0.0	0.00
0.5	0.10
2.0	0.40
5.0	1.00
8.0	1.60
10.0	2.00

Dilute each flask to 50ml with deionized water, add 2.0ml Nessler reagent and mix. After 20 minutes, read absorbance at 425nm against the blank. From the values obtained plot absorbance vs. NH_3-N conc. for the standard curve. Determine the ammonia in the distillate by nesslerization 50 ml or an aliquot diluted to 50ml and reading the absorbance at 425nm as described for the standards. NH_3-N is read from the curve in mg/l and calculated for each sample.

CALCULATIONS

Titrimetric-

$$\underset{(mg/l)}{NH_3\text{-}N} = \frac{A \times 0.28 \times 1000}{ml\ sample}$$

Where:

A = ml 0.02N H_2SO_4 used.

Spectrophotometric-

$$\frac{(mg/l\ from\ std.\ curve)(mls\ distillate(500))}{mls\ sample} \times dil.factor = mg/L\ NH_{3\text{-}N}$$

It is recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable.

QC REQUIREMENTS:

Preparation Blank:

-One preparation blank, consisting of deionized water, must be carried through procedure, for each batch.
-If value of blank is less than or equal to MDL, no correction of results is performed.
-If value of blank is above MDL, the lowest concentration of analyte in any sample must be 10x the blank concentration. Otherwise, all samples associated with the blank with analyte less than 10x the blank concentration and above the MDL, must be redigested and reanalyzed for that analyte. No correction of results is performed.

Duplicate Samples:

A duplicate sample must be run once every 20 samples. The control limits are +/- 20 RPD, or +/- 1 MDL (when less than 5x MDL)
If duplicate sample is outside control limits:
1) Check technique (esp. homogeneity of sample)
2) Rerun duplicate
If duplicate still fails - contact supervisor, technical director for assistance. Contact client.

Spike Samples:

A spiked sample must be run once every 20 samples. The control limits are 75-125% recovery. If spike sample is outside control limits:
1) Try a dilution (eliminate interference)
2) Check calculation
3) Check technique (pipetting, homogeneity)
4) Use standard addition methodology
If spike still fails - contact supervisor, technical director for assistance. Contact client.

External Control Sample:

An external control sample must be run once every 20 samples. The control limits are the 95% C.I. Analyze the external control sample first. If external control sample is outside of limits for colorimetric tests stop analysis.
1) Review methodology and SOP to make sure samples were prepared properly.
2) Prepare new stock solution, re-standardize if necessary and prepare a new standard curve.
3) Re-analyze external control sample.

For Titrimetric tests, stop analysis.
1) Review methodology and SOP to make sure samples were prepared properly.
2) Re-prepare primary standard and re-standardize titrant.
3) Re-analyze external control sample.

For Gravimetric tests, stop analysis.
1) Review methodology and SOP to make sure samples were prepared properly.
2) Recalibrate and check balance.
3) Re-analyze external control sample.

DOCUMENTATION REQUIREMENTS:

All standard and titrant preparations are recorded in a reagent log, along with the date and preparer's signature.

Standardizations are recorded in a standardization log.

All data produced is written on standard laboratory log sheets particular to that parameter being analyzed, along with the date and analyst's signature. All data produced is checked by the supervisor and subsequently signed, and then reviewed by our QC department.

SULFATE: TURBIDIMETRIC

EPA 375.4

REFERENCE:

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, method 375.4.

APPLICABILITY:

Analysis of water samples for sulfate. Approved for SDWA and CWA.

IMPORTANT NOTES:

Suspended matter and color will interfere with the measurement of turbidity. Sample blanks must be analyzed to eliminate these interferences.

PROCEDURE:

1. Perform this analysis on the UNPRESERVED sample.

2. Initial Calibration: Prepare a series of standards as outlined below. Prepare each standard in a 250-mL Erlenmeyer flask containing a TFE-coated magnetic stir bar.

Standard Sodium Sulfate (mL)	Water (mL)	Concentration (mg/L SO_4^{-2})
0	100	0
5	95	5
10	90	10
20	80	20
30	70	30
40	60	40

3. Add exactly 5.0 mL of Conditioning Reagent, place the flask on a magnetic stirrer, and stir at a speed just below the point where the solution splashes.

4. While the solution is being stirred, add a measuring spoonful (0.2 - 0.3 cc) of Barium Chloride Crystals and begin timing immediately.

5. Stir exactly 1.0 minutes, then immediately pour solution into an absorbance cell. Continue stirring the solution in the Erlenmeyer flask. Measure the turbidity at 420 nm.

6. At 30-second intervals, pour solution from the Erlenmeyer flask into an absorbance cell and measure the turbidity at 420 nm. Record the maximum reading obtained in the 4 minute period.

7. Continuing Calibration Check: Analyze a 20 mg/L SO_4^{-2} standard after each batch of sample analyses.

8. Sample Analysis: Treat a 100-mL aliquot of sample according to steps 3 through 6 above. If the sample contains more than 40 mg/L SO_4^{-2}, dilute an appropriate aliquot to 100 mL for analysis.

9. Prepare and analyze a sample blank by treating a second aliquot of sample as in step 8, omitting the Barium Chloride Crystals.

10. Prepare a matrix spike by adding 1.0 mL of Sulfate Spiking Solution to 100-mL aliquot of the sample to be spiked. Analyze the spike according to steps 3 through 6 above.

QUALITY ASSURANCE:

1. This method must be validated by the successful analysis of a blind quality control sample before samples can be analyzed.

2. A method detection limit (MDL) study (procedure AP0001) must be completed before samples can be analyzed. The MDL study must be repeated whenever a significant change in the procedure is made.

3. The required quantitation limit is 5.0 mg/L SO_4^{-2}.

4. The minimum allowable coefficient of correlation for the initial calibration is 0.99.

5. The maximum allowable percent difference (%D) for the continuing calibration check is 20. If the percent difference exceeds this value, a new initial calibration must be performed and all samples analyzed since the last acceptable continuing calibration check must be reanalyzed.

6. A reagent blank consisting of reagent water must be analyzed at a minimum frequency of one per batch or one per 20 samples, whichever is more frequent. The level of SO_4^{-2} detected in the reagent blank must be less than the required quantitation limit.

7. A sample duplicate must be analyzed at a minimum frequency of one per 20 samples or one per month, whichever is more frequent. The control limit for relative percent difference (RPD) is 10.

8. A matrix spike must be analyzed at a minimum frequency of one per 20 samples or one per month, whichever is more frequent. The control limits for recovery are 60% - 140%.

CALCULATIONS:

1. Initial calibration: Use a calculator or computer program to calculate the linear regression for the initial calibration. If the coefficient of correlation is less than 0.99, the sample concentrations must be read from a plot of absorbance vs.

concentration. (Analysts capable of calculating quadratic fit may try this alternative if the linear fit is less than 0.99; the quadratic fit also must be at least 0.99.)

2. Sample concentration:

$$C_S = (C_C - C_B) \times D$$

where C_S = sample concentration, mg/L
C_C = sample concentration from curve, mg/L
C_B = sample blank concentration from curve, mg/L
D = dilution factor

Report results to two significant figures.

3. Quality control results:

$$RPD = \frac{200 \times |C_S - C_D|}{C_S + C_D}$$

$$Rec = \frac{100 \times (C_M - C_S)}{C_T}$$

where RPD = relative percent difference, %
Rec = matrix spike recovery, %
C_S = unspiked sample concentration, mg/L
C_D = duplicate sample concentration, mg/mL
C_M = matrix spike concentration, mg/L
C_T = theoretical spike concentration, mg/L

Report recovery and RPD to the nearest 1%.

REAGENTS:

1. Hydrochloric Acid: Reagent grade.

2. Sodium Chloride: Reagent grade.

3. 95% Isopropanol.

4. Glycerol.

5. Sodium sulfate: Reagent grade, anhydrous.

6. Barium Sulfate Crystals: Reagent grade, 20 - 30 mesh.

7. Conditioning Reagent: Transfer 30 mL of Hydrochloric Acid to 1000-mL beaker containing 300 mL of reagent water. Add 75 g of Sodium Chloride and stir until dissolved. Add 100 mL of 95% Isopropanol and 50 mL of glycerol and mix. Store in a tightly sealed container and discard after 6 months.

8. Standard Sodium Sulfate: 100 mg/L. Transfer 0.1479 g of Sodium Sulfate to a 1000-mL volumetric flask. Dilute to volume with reagent water and mix until all crystals are dissolved. Store in a tightly sealed container and discard after 6 months.

9. Sulfate Spiking Solution: 2.0 mg/mL. Transfer 0.2958 g of Sodium Sulfate to a 100-mL volumetric flask. Dilute to volume with reagent water and mix until all crystals are dissolved. Store in a tightly sealed container and discard after 6 months.

ORTHOPHOSPHATE: MANUAL

EPA 365.2

REFERENCE:

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, method 365.2.

APPLICABILITY:

Analysis of water samples for orthophosphate. Approved for CWA and NPDES.

IMPORTANT NOTES:

None.

PROCEDURE:

1. Perform this analysis on the FILTERED sample.

2. All glassware used for this procedure must be rinsed with Hydrochloric Acid Solution prior to use.

3. Prepare a series of standards by transferring the following volumes of Phosphate Working Standard to a series of 50-mL volumetric flasks.

Phosphate Working Standard mL	Concentration mg/L
0	0.00
5.0	0.05
10.0	0.10
20.0	0.20
30.0	0.30

40.0	0.40
50.0	0.50

4. Adjust the pH of a 50-mL aliquot of sample to 7.0 ± 0.2 with Sodium Hydroxide Solution or Sulfuric Acid Solution.

5. Prepare a reagent using 50 mL of reagent water as the sample.

6. Prepare a matrix spike by adding 1.0 mL of Phosphate Spiking Solution to a 50-mL aliquot of the sample selected for spiking. The theoretical concentration of this spike is 0.25 µg P/L.

7. Add 8.0 ml of Combined Reagent to the sample and mix thoroughly. After a minimum of ten minutes, but no longer than thirty minutes, measure the color absorbance of each sample at 880 nm with a spectrophotometer, using the reagent blank as the reference solution.

   NOTE 1: If the same volume of sodium hydroxide solution is not used to adjust the pH of the standards and samples, a volume correction has to be employed.

QUALITY ASSURANCE:

1. This method must be validated by the successful analysis of a blind quality control sample before samples can be analyzed.

2. A method detection limit (MDL) study (procedure AP0001) must be completed before samples can be analyzed. The MDL study must be repeated whenever a significant change in the procedure is made.

3. The required quantitation limit is 0.05 mg/L.

4. A new calibration curve must be run when the continuing calibration check fails quality control criteria or once each 30 days, whichever occurs first.

5. The minimum allowable coefficient of correlation for the calibration is 0.995.

6. The maximum allowable percent difference (%D) for the continuing calibration is 10.

7. Analyze a reagent blank with each batch of samples analyzed or one per 20 samples, whichever is more frequent. The level of orthophosphate detected in the reagent blank must be less than the required quantitation limit.

8. Analyze a duplicate sample at a minimum frequency of one per 20 samples or one per month, whichever is more frequent. The control limit for relative percent difference (RPD) is 10.

9. Analyze a matrix spike at a minimum frequency of one per 20 samples or one per month, whichever is more frequent. The control limits for recovery are 80% - 120%.

CALCULATIONS:

1. Sample concentration:

Use a calculator or computer software to calculate the linear regression. Alternatively, the values can be plotted on graph paper. Read the concentrations directly from the calculator's linear regression function or from the manual plot.

$$C_s = C_C \times D$$

where C_s = sample concentration, mg P/L
C_C = concentration from curve, mg P/L
D = dilution factor

Report results to two significant figures.

2. Quality control results:

$$RPD = |C_s - C_D| \ / \ ((C_s + C_D)/2)$$

$$Rec = 100 \times (C_M - C_s) \ / \ C_T$$

where RPD = relative percent difference, %
Rec = matrix spike recovery, %
C_s = unspiked sample concentration, mg/L
C_D = duplicate sample concentration, mg/mL
C_M = matrix spike concentration, mg/L
C_T = theoretical spike concentration, mg/L

Report recovery and RPD to the nearest 1%.

REAGENTS AND MATERIALS:

1. Ammonium Molybdate: Tetrahydrate, reagent grade.

2. Antimony Potassium Tartrate: Reagent grade.

3. Ascorbic Acid: Reagent grade.

4. Hydrochloric Acid: Reagent grade.

5. Potassium Dihydrogen Phosphate: Reagent grade.

6. Sodium Hydroxide: Reagent grade.

7. Sulfuric Acid: Reagent grade.

8. Ammonium Molybdate Solution: Transfer 20 g of Ammonium Molybdate to a 500-mL volumetric flask and dilute to volume with reagent water. Store in a refrigerator in a plastic bottle and discard after 6 months.

9. Antimony Potassium Tartrate Solution: Transfer 1.3715 g of Antimony Potassium Tartrate to a 500-mL volumetric flask containing about 400 mL of reagent water. Dilute to volume with reagent water. Store in a refrigerator in a dark bottle and discard after 6 months.

10. Ascorbic Acid Solution: 0.1*M*. Dissolve 1.76 g of Ascorbic Acid in 100 mL of reagent water in a beaker. Store in a tightly closed bottle in a refrigerator and discard after 1 week.

11. Combined Reagent: Mix the following solutions in the order given to prepare 100 ml of the mixed reagent.

Sulfuric Acid Solution	50 mL
Antimony Potassium Tartrate Solution	5 mL
Ammonium Molybdate Solution	15 mL
Ascorbic Acid Solution	30 mL

Mix after addition of each solution. All reagents must reach room temperature before they are mixed. If turbidity forms, shake and let stand for a few minutes until the turbidity disappears before proceeding. Prepare fresh daily.

12. Hydrochloric Acid Solution: 1:1. Slowly and carefully add 500 mL of Hydrochloric Acid to 500 mL of reagent water in a 2000-mL beaker. Store in a tightly closed container and discard after 6 months.

13. Phosphate Spiking Solution: 12.5 µg P/mL. Transfer 25 mL of Phosphate Stock Solution to a 100-mL volumetric flask and dilute to volume with reagent water. Store in a refrigerator in a tightly closed bottle and discard after 6 months.

14. Phosphate Stock Solution: ~~0.05~~ 50 mg P/mL. Transfer 0.2197 g of Potassium Dihydrogen Phosphate to a 1000-mL volumetric flask and dilute to volume with reagent water. Store in a refrigerator in a tightly closed bottle and discard after 6 months.

15. Phosphate Working Standard: 0.50 µg P/mL. Dilute 10 mL of Phosphate Stock Solution to 1000 mL with reagent water. Prepare fresh daily.

16. Sodium Hydroxide Solution: 1*N*. Transfer 40 g of Sodium Hydroxide to a 1000-mL volumetric flask and dilute to volume with reagent water. Store in a plastic bottle and discard after 6 months.

17. Sulfuric Acid Solution: 5*N*. Slowly and carefully add 70 mL of Sulfuric Acid to a 500-mL volumetric flask containing about 300 mL of reagent water. Dilute to volume with reagent water. Store in a tightly closed container and discard after 6 months.

SOP # DATE: 12/13/95 DEPT. MGR.
QA MGR.

TEST NAME: TOTAL ORGANIC CARBON (TOC)-SOIL AND SEDIMENT

REFERENCE

US EPA Test Methods for Evaluating Solid Waste-SW846, Method 9060 Modified, Revision C, September 1986
O.I. Corporation Operating Procedures Manual, Model 524C.

MDL - 500 mg/kg

SCOPE AND APPLICATION

This method includes the measurement of organic carbon in soils and sediments.

SUMMARY OF METHOD

Organic carbon is a sample converted to carbon dioxide (CO_2) by wet chemical oxidation. The CO_2 formed can be measured directly by an infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

Carbonate and bicarbonate are inorganic forms of carbon which must be separated from the total organic carbon component. This is accomplished by acidifying the samples, converting the carbonates to CO_2 and degassing prior to analysis.

PRESERVATION AND HANDLING

Refrigerate to 4°C

HOLDING TIME

28 days from time of sampling.

INTERFERENCES

Carbonate and bicarbonate carbon represent an interference in this test and must be removed. To purge carbonates and bicarbonates from samples, oxygen is bubbled through samples for 6 minutes prior to sealing.

APPARATUS

-O.I. Corporation TOC analyzer
-O.I. Corporation Purge and Seal Unit
-Ampules
-Volumetric pipets
-Volumetric flasks
-Crucible
-Metal spatula

REAGENTS

-Saturated potassium persulfate - dissolve 70 g of reagent grade potassium persulfate in 1l deionized water. Mix.

-10% phosphoric acid - Add 100 ml of conc. phosphoric acid, reagent grade, to 500 ml deionized water. Bring to 1l with deionized water. Mix.

-Stock carbon solution (KHP) - dissolve 2.126 g of KHP (potassium biphthalate), reagent grade, in deionized water and bring to 1l in a volumetric flask. (1000 g mg/l C) (1.0 ml = 1.0 mg C).

-Magnesium perchlorate - granular, reagent grade

CALIBRATION PROCEDURE

-From the stock carbon solution(1000 mg/l) take 10.0 ml and dilute to 100 ml in volumetric flask. (100 mg/l C) Prepare standard solutions as follows:

ug TOC	mls 100 mg/l solution in 100 ml volumetric flask
0	0
10	1.0
30	3.0
50	5.0
70	7.0
100	10.0

-Follow procedure for purging, sealing and reading standards.

-Calibration curve must have a 0.995 correlation coefficient.

SAMPLE PREPARATION PROCEDURE

1. Using a metal spatula mix the soil until homogeneous.

2. Weigh out 20 mg of soil. Transfer to ampule. Repeat this four times for each sample.

3. Add 1.0 ml of 10% H_3PO_4 to each ampule.

4. Add 2.0 ml of deionized water(Type I). Rinse sides of ampule when adding the water.

5. Cover ampules with aluminum foil and let stand 30 minutes.

6. Add 2.0 ml of saturated $K_2S_2O_3$ solution.

7. Add an additional 2.0 ml of deionized water(Type I).

8. Purge contents of ampules for 6 minutes.

9. Rinse ampules with 3.0 ml of deionized water(Type I). Rinse sides of ampule when adding the water.

10. Seal ampules using Ampule Seal Procedure.

12. Analyze ampules using TOC analyzer procedure.

AMPULE SEAL PROCEDURE

1. Turn on oxygen supply, tank pressure must be at least 500 psi and flow pressure set at 20 lbs. Flip switch to heat catalyst to 500°C.

   a. Turn open the propane tank valve on the back of unit and flip the propane switch on the front.

   b. Turn the propane adjust control all the way clockwise then turn back 1/2 turn (on). Light the burner and adjust flame to above 1/2 inch length.

   c. Flip on oxygen switch on the front and turn oxygen adjust control so that the flame is almost jet like.

2. Place top of ampule in holding clamp and position supporter about 1/4 inch from the bottom of the ampule. Swing flame so that the jets surround the ampule. In about 5 seconds the flame should melt the glass and the ampule will drop into the holder, lower the holder another 1/4 inch and let the flame burn through. As soon as it is through, swing the burner away. Carefully remove the ampule and waste glass.

3. When all the ampules are sealed use a metal test tube rack and digest the samples in a 130°C oven for 30 minutes.

TOC ANALYZER PROCEDURE

1. The main power switch is always on. Replace the drying reagent (magnesium perchlorate) each time the analyzer is used. It is the white powder that is in the 2 tubes on the front of the analyzer and the 3 tubes on the side. Push up on the tube to remove it, empty the tube, and flush with deionized water to clean, then dry with blow-hard. Put some $Mg(ClO_4)_2$ between two pieces of weighing paper and slightly crush it. Do not make a dust out of it. Place a loose ball of cotton in one end of the clean, dry tube and fill with $Mg(ClO_4)_2$. Cover with another ball of cotton. Replace tube. Turn on oxygen (500 tank, 20 flow) for 30 minutes before beginning analysis. Slide a rubber grommet over the sealed tip until its snug and secure it onto the apparatus. Adjust the flow rate to 13 with the zero gas control.

2. Turn on Model 526 integrator. Press the auto zero button to stop display from counting up and to set a temporary zero.

3. To set zero: Below the gauge are three controls (RANGE SW, ZERO, SPAN). Turn the zero control until the needle on the gauge is just below the 0. Push the auto zero button and flip the clear switch, the display will now show all 0's. Slowly turn the zero control clockwise display is counting then turn zero control counter-clockwise until until the display stops counting up. Flip the clear switch, the display should show all 0's and the needle should be pointing on 0. The analyzer is now set to run samples. Occasionally, it may be necessary to reset the zero.

4. To analyze an ampule place the grommet on, secure in place, and adjust flow to 13. If flow will not go to 13, there is an oxygen leak in the system. Check the seals and adjust. Each time an ampule is set up there will be air in the system-let the display count up until it stops, then flip the clear switch to reset display to zero. This must be done with each ampule, otherwise you will have false readings. Push and turn the breaking apparatus until the ampule is open. Quickly push sample probe into ampule close to the bottom. Watch the gauge, if it gets pinned past 100 then the ampule is too concentrated, use a dilution. Record the final number from the integrator display and repeat process on all samples.

CALCULATIONS

$$\frac{\text{ug (from standard curve)}}{\text{sample wt. (g)} \times \text{decimal fraction of solids}} = \text{mg/kg DWB}$$

Weigh out 5 of 10 g of sample into a tared crucible. Dry overnight at 105° C. Allow to cool in a desiccator before weighing.

$$\% \text{ Solids} = \frac{(\text{g of dried sample}) - (\text{weight of crucible})}{(\text{weight of wet sample} + \text{crucible}) - (\text{weight of crucible})}$$

QC REQUIREMENTS:

Analyze standards each time the analyzer is used. Use 10 ug, 50 ug, 100 ug standards. Compare with previous standards.

Preparation Blank:
-One preparation blank, consisting of deionized water, must be carried through the procedure, for each batch(ie-not greater than 20 samples).
-If value of blank is less than or equal to MDL, no correction of results is performed.
-If value of blank is above MDL, the lowest concentration of analyte in any sample must be 10x the blank concentration. Otherwise, all samples associated with the blank with analyte less than 10x the blank concentration and above the MDL, must be redigested and reanalyzed for that analyte. No correction of results is performed.

Replicate analysis:
-Run all samples in quadruplicate, report both the average and the range.

Spike Duplicate Samples:
-Analyze a spike and spike duplicate sample once every 10 samples. The control limits are 75-125% recovery and the control limits for the spike/spike duplicate control limits for the spike/spike duplicate RPD's are +/-20% or +/-1 MDL(when less than 5x the MDL). If spike recoveries are outside control limits:
1) Try a dilution (eliminate interference)
2) Check calculation
3) Check technique (pipetting, homogeneity)
4) Use standard addition methodology
If spike still fails - contact supervisor, technicial director for assistance. Contact client.

-If spike/spike duplicate sample RPD is outside control limits for precision:
1) Check technique(sample homogeneity)
2) Rerun MS/MSD set
If duplicate still fails - contact supervisor, technical director for assistance. Contact client.

External Control Sample:
-Analyze an external control sample every 15 samples. The control limits are the 95% C.I. Analyze the external control sample first. If external control sample is outside of criteria:
1) Review methodology and SOP to make sure samples were prepared properly.
2) Prepare new stock solution, re-standardize if necessary and prepare a new standard curve.
3) Re-analyze external control sample.

DOCUMENTATION REQUIREMENTS:

All standard and titrant preparations are recorded in a reagent log, along with the date and preparer's signature.

Standardizations are recorded in a standardization log.

All data produced is written on standard laboratory log sheets particular to that parameter being analyzed, along with the date and analyst's signature. All data produced is checked by the supervisor and subsequently signed, and then reviewed by our QC department.

STANDARD TEST METHOD FOR PARTICLE-SIZE ANALYSIS OF SOILS

(ASTM D 422)

DOCUMENT CONTROL PAGE

QA/QC OFFICER

LABORATORY DIRECTOR

1.0 Scope

This test method covers the quantitative determination of particle sizes in soils for both the particle-size test to the #200 sieve (.074 mm) and the particle-size with hydrometer (<.074 mm). The hydrometer portion of this test uses the sedimentation process over a twenty-four (24) hour time period.

2.0 Reference

2.1 ASTM Method D422

3.0 Apparatus and Reagents

3.1 Balance - sensitive to 0.01 gram.

3.2 Sieve Shaker Model RX 86.

3.3 Hydrometer bulb - conforming to the requirements for hydrometers 151H or 152H in Specification E100 for ASTM Hydrometers.

3.4 Sedimentation cylinder - marked for volume of 1000 ml and conforming to a height, from the bottom of the cylinder, of 36 +/-2 cm to the 1000 ml mark.

3.5 Sampler - a riffle sampler or sample splitter for quartering the samples

3.6 Sieves - conform to requirements of Specification E11 (ASTM), they include: 1 in., #10-2mm, #20-841um, #40-425um, #60-259um, #100-149um, #200-75um, pan and cover.

3.7 Thermometer - accurate to 1° F.

3.8 Timer - a device capable of reading seconds and minutes.

3.9 Sodium Hexametaphosphate Solution - dissolve 40 g of sodium hexametaphosphate in one liter of distilled or demineralized water.

3.10 Stirring Apparatus- a mechanically operated stirring device with a speed of not less than 10,000 rpm. The paddles will be operated not less than ¾ in. not 1-1/2 in. above the bottom.

3.11 Dispersion cup- A special dispersion cup conforming to ASTM D422

3.12 Constant Temperature room- maintained at or near 20°C (68°F)

4.0 Holding Time

4.1 The maximum holding time for sieve analysis shall be seven days from collection date.

4.2 The Hexavalent Chromium holding time of 48 hrs will begin as soon as the sieve analysis is complete on that sample.

5.0 Sample Preparation

5.1 Air dry the sample for 24 hours as per Chemtech SOP CCG-GRAIN-SZ2.

5.2 The purpose of sieve analysis is to obtain the grain size of the classic particles as they were developed. This is often difficult because:

5.2.1 Classic sand grains may acquire overgrowth, or be cemented into tough aggregates to form hard sandstones.

5.2.2 Clay minerals because of their flaky character and surface electrical charges tend to cluster in lumps. The idea of disaggregation is to separate all of the individual grains without smashing any of them, and to remove all chemically precipitated substances. To avoid smashing grains as much as possible, one should start with gentle treatments and gradually work up to the more severe treatments only if the gentle ones fail.

SIEVE ANALYSIS OF PORTION RETAINED ON #200 SIEVE

6.0 Procedure

6.1 Prior to sample analysis , the sieves are washed, dried and brushed to insure that no material is retained on the sieve between sample analyses.

6.2 The mass of air-dried soil selected for testing shall be sufficient to yield quantities for mechanical sieving (portion retained on the #10 mesh sieve) as follows:

Nominal Diameter of Largest Particles		Approximate Minimum Mass of Portion
inches	mm	grams
3/8	9.5	500
¾	19.0	1000
1	25.4	2000
1 ½	38.1	3000
2	50.8	4000
3	76.2	5000

6.3 Weigh out the appropriate amount of material to the nearest 0.01 gram.

6.4 Assemble the sieve sizes to be used:

¼ in.
#10 - 2mm
#20 - 841um
#40 - 425um
#60 - 250um
#100 - 149um
#200 - 75um

6.4.1 Prior to sample analysis, record the weight of each sieve, and pan. Record the weight to the nearest 0.01 gram on the laboratory worksheet.

6.5 Nest the screens in order, coarsest at the top, pan on the bottom. Pour the sample in to the top sieve. Place a cover on the stack.

6.6 Place the sacked screens in the Seive Shaker Model RX 86, fasten very tightly, and sieve for 15 minutes. Sieving time should be constant for all samples (15 minutes is the accepted time).

6.7 After sieving is complete weigh to the nearest 0.01 grams, each sieve with the amount of material retained. Complete this step for each sieve, as well as the lower pan. Record the weight on the weighs on the worksheet.

6.7.1 Calculate the percent retained on each sieve and in the pan.

6.8 The material retained on the ¼ in. sieve is stored in a Ziplock Bag. Label the bag with the sample identification number and size fraction (+1/4-in.). Store at 4° C. This portion will be analyzed for Total and Hexavalent Chromium.

6.9 Combine the material collected from sieves #10, #20, #40, #60, #100 and #200. Store the material in a Ziplock Bag. Label the bag with the sample identification number and size fraction (1/4-in. x 200m). Store at 4° C. This portion will be analyzed for Total and Hexavalent Chromium.

6.10 Divide the minus #200 material in the lower pan into two portions. The first portion will be analyzed for Hexavalent Chromium and Total Chromium. A minimum of 20 grams is required. The analysis has the highest priority. Store the material in a Ziplock Bag. Label the bag with the sample identification number and size fraction (minus 200m). Store at 4° C. The second portion will be used for the hydrometer analysis if sufficient material remains. Store the material in a Ziplock Bag.

HYDROMETER ANALYSIS OF PORTION PASSING THE #200 SIEVE

7.0 Determination of Composite Correction for Hydrometer Reading

7.1 Prepare 1000 ml of liquid composed of distilled water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test (control sample).

7.2 Place the thermometer and hydrometer bulb in the control sample.

8.0 Sample preparation and set-up.

8.1 Hygroscopic Moisture

8.1.1 Determine the percent moisture of the minus 200 mesh material using Chemtech SOP NO. CCG-WET-GRAIN-SZ1. Correct the remaining test material mass to determine an accurate dry mass for the sample being tested.

8.2 When the soil is mostly of the clay and silt sizes, weigh a sample of the air dry soil of approximately 50 grams. When the soil is mostly sand the sample should be approximately 100 grams. Using a riffle sampler reduce the sample to the required weight.

8.3 Place the sample in a 250 mL beaker and cover with 125 mL of sodium hexametaphosphate solution. Stir until the soil is thoroughly wetted. Allow to soak for at least 16 hours.

8.4 At the end of the soaking period, disperse the sample further, using dispersion cup in accordance with apparatus listed above. Place soil in dispersion cup and add distilled water, if necessary, so that the cup is more than half full. Stir for a period of 1 minute.

8.5 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled water until the total volume is 1000 mL.

8.6 Using the palm of the hand over the open end of the cylinder, turn the cylinder upside down and back for a period of 1 minute to complete the agitation of the slurry. At the end of 1 minute set the cylinder in a convenient location and take and record hydrometer readings at the following intervals of time (measured fr the beginning of the sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30 ,60, 250, and 1440 minutes. At these times you shall carefully insert hydrometer into slurry about 20 to 25 seconds before the reading is due. After the reading is taken remove hydrometer and insert (using a spinning motion) into a graduate containing distilled water.

8.7 Take temperature of the slurry at each reading.

CALCULATIONS AND REPORT

9.0 Percentages of Soil in Suspension

9.1 Calculate the mass of the total sample represented by the mass of the soil used the hydrometer test, by dividing the oven-dry mass used by the percentage passing the #200-sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

9.2 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows:

$$P = (Ra/W) \times 100$$

where:

a = correction factor (for density) to be applied to the hydrometer reading (see Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension

R = hydrometer reading with composite correction applied (control sample reading),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed.

10.0 Diameter of Soil Particles

10.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated as follows:

$$D = K x (L/T)^{0.5}$$

where:

D = diameter of particle, mm,

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles (Table 2),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm (Table 3),

T = interval of time from the beginning of sedimentaiton to the taking of the reading, min.

11.0 Particle Size Analysis Results

11.1 Calculate the percent of the sample retained on and percent passing each sieve.

11.2 Calculate the percent collected in the pan.

11.3 Correct the percentage from the hydrometer analysis by multiplying the percentage retained in the pan times the percentage from the hydrometer reading, divided by 100.

12.0 Graph

12.1 Plot the diameters of the particles, on a logarithmic scale, as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate.

TABLE 1
Values of Correction, a, for Different Specific Gravities of Soil Particles

Specific Gravity	Correction Factor
2.95	0.94
2.90	0.95
2.85	0.96
2.80	097
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

TABLE 2
Values of K or Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature	Specific Gravity of Soil Particles								
(C)	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.01323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

TABLE 3
Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder

Hydrometer 152 H

Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
0	16.3	31	11.2
1	16.1	32	11.1
2	16.0	33	10.9
3	15.8	34	10.7
4	15.6	35	10.6
5	15.5	36	10.4
6	15.3	37	10.2
7	15.2	38	10.1
8	15.0	39	9.9
9	14.8	40	9.7
10	14.7	41	9.6
11	14.5	42	9.4
12	14.3	43	9.2
13	14.2	44	9.1
14	14.0	45	8.9
15	13.8	46	8.8
16	13.7	47	8.6
17	13.5	48	8.4
18	13.3	49	8.3
19	13.3	50	8.1
20	13.0	51	7.9
21	12.9	52	7.8
22	12.7	53	7.6
23	12.5	54	7.4
24	12.4	55	7.3
25	12.2	56	7.1
26	12.0	57	7.0
27	11.9	58	6.8
28	11.7	59	6.6
29	11.5	60	6.5
30	11.4		

TEST NAME: COD - CHEMICAL OXYGEN DEMAND

REFERENCE: Hach Handbook of Water Analysis, 1979, Method 8000
Standards for the Examination of Water and Wastewater, 18th Edition, 1992, Method 5220B(used for standardization of titrant)

MDL: Low Range - 5 mg/l
High Range - 50 mg/l

SCOPE AND APPLICATION:

This method covers the determination of COD in surface waters, sludge, domestic and industrial wastes.

SUMMARY OF METHOD:

Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ istitrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed or it is measured spectrophotometrically against standards.

PRESERVATION AND HANDLING:

Preserve samples with sulfuric acid to pH <2 and maintain at 4°C.

HOLDING TIME:

28 days from time of sampling

INTERFERENCES:

Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight chain aliphatic compounds are oxidized more effectively when silver sulfate ($AgSO_4$) is added as a catalyst. However, $AgSO_4$ reacts with chloride, bromide, and iodide to produce precipitates that are oxidized only partially. The difficulties caused by halides can be overcome largely, though not completely, by complexing with mercuric sulfate($HgSO_4$) before refluxing.

APPARATUS:

- COD reactor
- Hach ampules
- Volumetric flasks
- Volumetric pipets
- 10 ml micro buret and 25 ml buret
- Spectrophotometer
- Erlenmeyer flasks
- Test tube rack

REAGENTS:

- Hach ampules - Low Range - 0 -150 mg/l
  High Range - 50 - 2000 mg/l

  - Low range ampules contain - 0.025N $K_2Cr_2O_7$, $HgSO_4$, and $AgSO_4$.

  - High range ampules contain - 0.25N $K_2Cr_2O_7$, $HgSO_4$, and $AgSO_4$.

- Potassium Hydrogen Phthalate (KHP) - crush lightly, then dry primary standard potassium hydrogen phthalate to constant weight at 120o$_C$.
- Low range KHP: dissolve 425 mg KHP in deionized water and dilute to 1l (500 mg/l).

- High range KHP: dissolve 8.46 g KHP in deionzed water and dilute to 1l (10,000 mg/l).

For Titration:
- High range: 0.025N potassium dichromate standard: dissolve 1.225 g $K_2Cr_2O_7$ in a 1l volumetric flask in deionized water and bring to volume.

- 0.025N ferrous ammonium sulfate titrant: dissolve 9.8000g $Fe(NH_4)_2(SO_4)_2 .6H_2O$ in 500 ml deionized water. Add 20 ml conc. H_2SO_4 and bring to volume in a 1l volumetric flask.

- Low range: 0.005N Potassium dichromate standard-dissolve 0.2452 g $K_2Cr_2O_7$ in a 1L volumetric flask in deionized water and bring to volume.

- 0.005N Ferrous ammonium sulfate titrant-dissolve 1.96 g $Fe(NH_4)_2(SO_4)_2.6H_2O$ in 500 ml deionized water. Add 20 ml conc. H_2SO_4 and bring to volume in a 1L volumetric flask.

- Ferroin indicator: dissolve 1.48 g of 1 - 10 (ortho) - phenanthroline monohydrate, together with 0.70 g of ferrous sulfate seven hydrate ($FeSO_4 .7H_2O$) in 100 ml of deionized water.

CALIBRATION PROCEDURES:

Colorimetric - If samples are to be read colorimetrically, prepare standards for each range in which the samples are to be read.

Low Range:

mg/l COD	mls 500 mg/l KHP in 50 ml volumetric flask
0	0
25	2.5
50	5.0
100	10.0
150	15.0

High Range:

mg/l COD	mls 10,000 mg/l KHP in 50 ml volumetric flask
200	1.0
600	3.0
1000	5.0
1400	7.0
2000	10.0
0	0

-Add 2 ml of each standard to each ampule in the proper range and carry through the digestion procedure.

-When reading standards and samples on the low range, set wavelength to 420 nm, and zero spectrophotometer with the 150 mg/l standard (This is an inverted curve). Then, read all standards and prepare a standard curve. Correlation coefficient must be 0.995.

-When reading standards and samples on the high range, set wavelength to 620 nm and zero spectrophotometer with the 0 mg/l standard. Then, read all standards and prepare a standard curve. Correlation coefficient must be 0.995.

-Titrimetric - If samples are to be read titrimetrically, standardize both solutions of ferrous ammonium sulfate, or the solution to be used with the range of samples being analyzed, against the same normality of $K_2Cr_2O_7$:

0.025N FAS - High Range
0.005N FAS - Low Range

-Standardization: dilute 10.0 ml standard $K_2Cr_2O_7$ to about 100 ml. Add 30 ml conc. H_2SO_4 and cool. Titrate with FAS using 2 - 3 drops of ferroin indicator. End point is brown.

$$\text{Normality of FAS} = \frac{\text{Volume } K_2Cr_2O_7 \text{ titrated, ml}}{\text{Volume FAS used in titration}}$$

DIGESTION PROCEDURE:

1. Homogenize sample by mixing thoroughly.

2. Turn on COD reactor and preheat to 150°C.

3. Remove the cap of a COD vial of the desired range.

4. Pipet 2.00 ml of sample into the vial.

5. Replace cap tightly. Wipe vial clean with paper towel.

6. Gently, mix contents by inverting vial. Place vial in COD reactor.

7. Prepare a blank by adding 2.00 ml of deionized water. It is recommended that 2 to 4 blanks be run with every batch. For titration take average of blank values.

8. Heat vials for 2 hours.

9. Turn off reactor. Wait 20 minutes for the vials to cool. Remove vials from reactor and place in test tube rack.

10. Wait until vials have reached room temperature before reading colorimetrically or titrating.

-If a pure green color appears in a reacted sample, the reagent capacity may have been exceeded. Repeat the test with a diluted sample or run sample on the higher range.

CALCULATION:

Titrimetric-

$$\text{COD as mg } O_2/l = \frac{(A - B) \times N \times 8000}{\text{ml of sample}}$$

Where: A = ml FAS used for blank
B = ml FAS used for sample
N = Normality of FAS

Colorimetric-

For undiluted samples read mg/l directly from the standard curve. For samples that are diluted read the standard curve and multiply by the dilution factor.

QC REQUIREMENTS:

Preparation Blank:

-One preparation blank, consisting of deionized water, must be carried through procedure, for each batch or for every 20 samples.
-If value of blank is less than or equal to MDL, no correction of results is performed.
-If value of blank is above MDL, the lowest concentration of analyte in any sample must be 10x the blank concentration. Otherwise, all samples associated with the blank with analyte less than 10x the blank concentration and above the MDL, must be redigested and reanalyzed for that analyte. No correction of results is performed.

Duplicate Samples:

Analyze a duplicate sample once every 10 samples. The control limits are +/- 20 RPD, or +/- 1 MDL (when less than 5x MDL)
If duplicate sample is outside control limits:
1) Check technique (esp. homogeneity of sample)
2) Rerun duplicate
If duplicate still fails - contact supervisor, technical director for assistance. Contact client.

Spike Samples:

Analyze a spiked sample once every 20 samples. The control limits are 75-125% recovery. If spike sample is outside control limits:
1) Try a dilution (eliminate interference)
2) Check calculation
3) Check technique (pipetting, homogeneity)
4) Use standard addition methodology
If spike still fails - contact supervisor, technical director for assistance. Contact client.

External Control Sample:

Analyze an external control sample once every 10 samples. The control limits are the 95% C.I. Analyze the external control sample first. If external control sample is outside of limits for colorimetric tests stop analysis.
1) Review methodology and SOP to make sure samples were prepared properly.
2) Prepare new stock solution, re-standardize if necessary and prepare a new standard curve.
3) Re-analyze external control sample.

For Titrimetric tests, stop analysis.
1) Review methodology and SOP to make sure samples were prepared properly.
2) Re-prepare primary standard and re-standardize titrant.
3) Re-analyze external control sample.

DOCUMENTATION REQUIREMENTS:

Record all standard and titrant preparations in a reagent log, along with the date and preparer's signature.

Record standardizations in a standardization log.

Document all data produced on standard laboratory log sheets particular to that parameter being analyzed, along with the date and analyst's signature. All data produced is checked by the supervisor and subsequently signed, and then reviewed by our QC department.

CHEMTECH
CONSULTING GROUP, INC

Standard Operating Procedures

for

SULFIDE
MCAWW Method 376.1 (Titrimetric, Iodine)
(Revision I, Revised 12/94)

1. Scope and Application

1.1 This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.

1.2 Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class).

1.3 This method is suitable for the measurement of sulfide in concentrations above 1 mg/1.

2. Summary of Method

2.1 Excess iodine is added to a sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate or phenylarsine oxide.

3. Comments

3.1 Reduced sulfur compounds, such as sulfite, thiosulfate and hydrosulfite, which decompose in acid may yield erratic results. Also, volatile iodine-consuming substances will give high results.

3.2 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmearsureable form.

3.3 If the sample is not preserved with zinc acetate and NaOH, the analysis must be started immediately.

CHEMTECH
CONSULTING GROUP

Apparatus:

Ordinary laboratory glassware.

5. **Reagents**:

5.1 Hydrochloric acid, HCl, 6N

5.2 Standard iodine solution, 0.0250 N: Dissolve 20 to 25 g KI in a little water in a liter volumetric and add 3.2 g iodine. Allow to dissolve. Dilute to 1 liter and standardize against 0.0250 N sodium thiosulfate or phenylarsine oxide using a starch indicator.

5.3 Phenylarsine oxide 0.0250 N: commercially available.

5.4 Starch indicator; commercially available.

5.5 Procedure for standardization (see Residual Chlorine-iodometric titration Method 330.0, section 5.15).

6. **Procedure**:

6.1 Unprecipitated sample

6.1.1 Place a known amount of standard iodine solution (5.2) into a 500 mL flask. The amount should be estimated to be in excess of the amount of sulfide expected.

6.1.2 Add distilled water, if necessary, to bring the volume to approximately 20 ml.

6.1.3 Add 2 ml of 6 N HCl (5.1).

6.1.4 Pipet 200 ml of sample into the flask, keeping the tip of the pipet below the surface of the sample.

6.1.5 If the iodine color disappears add more iodine until the color remains. Record the total number of milliliters of standard iodine used in performing steps 6.1.1, and 6.1.5.

6.1.6 Titrate with the reducing solution (0.0250 N sodium thiosulfate or 0.250 N phenylarsine oxide solution (5.3) using a starch indicator (5.4) until the blue color disappears. Record the number of milliliters used.

6.2 Precipitated samples

6.2.1 Add the reagents to the sample in the original bottle. Perform steps 6.1.1., 6.1.3. 6.1.5, and 6.1.6.

6.3 Dewatered samples

6.3.1 Return the glass fibre filter paper which contains the sample to the original bottle. Add 200 ml distilled water. Perform steps 6.1.1, 6.1.3, 6.1.5, and 6.1.6.

6.3.2 The calculations (7) should be based on the volume of original sample put through the filter.

7. Calculations

7.1 One ml of 0.0250 N standard iodine solution (5.2) reacts with 0.4 mg of sulfide present in the titration vessel.

7.2 Use the formula

$$\text{mg/L sulfide} = \frac{400\,(A - B)}{\text{ml sample}}$$

where:

A = ml of 0.0250 N standard iodine solution (5.2)
B = ml of 0.0250 N standard reducing sodium thiosulfate or phenylarsine oxide solution (5.3)

8. Precision and Accuracy

8.1 The analysis of one duplicate sample, one spike sample and one reference sample for every twenty samples analyzed.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 505, Method 428D, (1975).

APPENDIX D

SOPS FOR FIELD INSTRUMENTS

STANDARD OPERATING PROCEDURES
FIELD MEASAUREMENT OF pH, TEMPERATURE AND SPECIFIC CONDUCTIVITY IN WATER

1.0 PURPOSE

This method is applicable to samples of stormwater, surface water, potable water supplies, and groundwater with measurement occurring at the sampling point. This procedure defines acceptable and consistent methods for measurement of pH, specific conductance, and temperature.

2.0 APPARATUS

The YSI Model 3560 Water Quality Monitoring System, or equivalent instrument, will be used. The instrument is a portable, microprocessor based pH, specific conductivity, and temperature meter.

3.0 REAGENTS

A) pH reference buffer solutions:

1) pH = 4.00
2) pH = 7.00
3) pH = 10.00

B) Specific conductivity buffer solutions:

1) Conductivity standards A or B = 1413 uS or12.88 mS

C) Distilled water

4.0 CALIBRATION PROCEDURES

A) Select sensor (i.e. pH, conductivity)

B) One point calibration

1) Place the sensor in the calibrating medium:

Measurement	Solution	Reading
pH	pH = 7 buffer	7.00 pH (25°C)
Cond	Hold in free air	0.00 μS
TDS	Hold in free air	0.00 mg/L

2) Press CAL - cal 1 is displayed. After endpointing, the display automatically updates to the calibrated value shown, or the temperature compensated value.

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3) If READ is pressed after cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

C) Two point calibration

1) Follow one point calibration. Place sensor in second calibrating medium: Calibration for pH measurements will utilize the two point calibration option.

Measurement	Solution	Reading
pH	pH 4 or pH 10 buffer	4.00 or 10.01 pH (at 25°C)
Cond	Cond std A or B	1413 uS or 12.88 mS
TDS	Cond std A or B	706 mg/L or 6.44 g/L

2) Press CAL - cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

5.0 SAMPLE HANDLING AND PREPARATION

Samples collected for pH, specific conductivity, and temperature should be obtained directly from the sampling point. Groundwater samples being tested during well purging can be obtained directly from the bailer.

6.0 PROCEDURES

Select sensor for required measurement (i.e. pH sensor or conductivity sensor). Attach sensor to the M90 meter. Calibrate meter to the solution corresponding to type of sensor. Meter is now ready to make a measurement following these steps:

A) Prepare sensor

1) pH - remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

2) Specific conductivity/TDS - immerse probe to halfway point in solution.

3) Temperature - pH and conductivity sensors automatically measure temperature.

B) Press MODE, READ, CAL, or M to turn meter and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press READ. Press READ again to start new measurement.

C) After use, close the fill hole and replace the wetting cap (pH).

7.0 TROUBLE-SHOOTING AND MAINTENANCE

A) Use distilled water when transferring from one solution to another.

B) Response time is function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength - pH only), allow more time for the sensor to respond.

C) Avoid handling the sensor tip.

D) Make sure no large air bubbles are trapped under the sensor when making measurements.

E) Do not use calibration standards after the expiration date.

F) Wetting caps should contain:

pH - pH 7 buffer

G) For greatest accuracy, calibrants and samples should be at the same temperature.

H) pH - keep the electrode filled with the appropriate fill solution to prevent reading drift.

I) Conductivity - the sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.

8.0 ACCURACY AND PRECISION

Accuracy for pH and specific conductance is addressed by verifying the agreement of two standards/buffers from separate lots. pH should agree within $\pm$ 0.5 standard unit. Specific conductance should agree within 10%. Accuracy for pH can also be checked by verifying on a third standard. Calibration would not be adjusted based on the third standard. Accuracy will not be verified for temperature. Temperature will not be used to determine cleanup objectives and will not halt groundwater sampling.

Precision for pH and specific conductance will be verified by checking the measurement on a split of the original sample (i.e., split buffer solution into two jars and verify precision). Measurements should be within 1 unit for pH and 10% for specific conductance. Precision for temperature will not be determined.

Accuracy and precision will be verified during each calibration (i.e., beginning of the day, middle of the day, and as required during sampling) and recorded in the logbook. Corrective action will be taken as required.

9.0 REPORTING

A) pH - report the average value of the replicate measurements to the nearest 0.1 units.

B) Temperature - report the average value of the replicate measurements to the nearest 1°C.

C) Specific Conductivity/TDS - report the average value of the replicate measurements to three significant digits.

STANDARD OPERATING PROCEDURES
FIELD MEASAUREMENT OF TURBIDITY

1.0 PURPOSE

This method is applicable to samples of groundwater, portable water supplies, surface water, and stormwater with measurement occurring at the sampling point. This procedure defines acceptable and consistent methods for measurement of turbidity.

2.0 APPARATUS

A HFDRT-15CE Portable Turbidimeter and accessories, or equivalent instrument, will be used for measuring turbidity.

3.0 PRINCIPLE OF OPERATION

The portable Turbidimeter operates on the nephelometric principle of turbidity measurement. This instrument meets the design criteria specified by the United States Environmental protection Agency, Method 180.1.

4.0 RANGE OF MEASUREMENT

The instrument measures turbidity from 0.01 to 1000 NTU in automatic range mode with automatic decimal point placement. The manual range mode measures turbidity in three ranges: 0.01 to 9.99, 10 to 99.9 and 100 to 1000 NTU. The instrument has an accuracy within 2 percent of reading plus stray light from 0-1000 NTU.

5.0 CALIBRATION

Calibration of the Turbidimeter is based on formazin, the primary standard for turbidity. The initial calibration is done at the factory. A formazin recalibration should be done at least once every three months. The equipment should be recalibrated (as required) and its batteries checked before shipping it to the field. Recalibration should be done according to the procedure described in the instrument manual (Section 3.6). Calibration checks can be achieved using sealed check standards.

6.0 ACCURACY AND PRECISION

Accuracy for turbidity will be verified using two standards from different lots. This will check the accuracy of the instrument. Accuracy should be within 10% of standard. If accuracy objectives are not met, corrective action may be required. Corrective action may include instrument recalibration by the manufacturer, or battery replacement.

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Precision during calibration may not be possible as the standards are sealed to reduce contamination. Precision will be assessed on water samples at a frequency of 1 per 10 samples. Precision will be verified by checking the measurement on a split of the original sample. Precision should be within 10%.

7.0 SAMPLE HANDLING

To ensure a representative sample, mix every sample thoroughly before aliquots are taken. Do not allow the sample to settle. When sampling from a tap, allow the water to run for at least five minutes before sampling.

8.0 TURBIDITY MEASUREMENT PROCEDURE

Follow the instrument manual for turbidity measurement. The following general steps should be used to measure turbidity:

A. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15mL), taking care to handle the sample cell by the top. Cap the cell.

B. Wipe the cell with a soft, lint-free cloth to remove moisture and fingerprints.

C. Place the instrument on a flat, sturdy surface. Do not hold the instrument while make measurements. Press: I/O.

D. Put the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the cover.

E. Select manual or automatic range selection by pressing the "RANGE" key. Using automatic range selection is recommended. When the instrument is turned on, the instrument defaults to the range mode and measurement range.

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STANDARD TECHNICAL PROCEDURE C-3

FIELD MEASUREMENT OF DISSOLVED OXYGEN (DO)

1. Scope and Application

Dissolved oxygen (DO) levels in natural waters and wastewater depend on the physical, chemical, and biochemical activities in the waterbody. Conversely, growth of many aquatic organisms, as well as the rate of corrosivity, are dependent on the DO concentration. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements should be taken in situ, since concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and is free from interference caused by color, turbidity, colloidal material, or suspended matter.

2. Apparatus

The following equipment is needed to measure dissolved oxygen concentration:

A. YSI Model 56 dissolved oxygen meter or equivalent.
B. Dissolved/oxygen/temperature probe for above meter.
C. Sufficient cable to allow the probe to contact the sample.

3. Reagents

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Distilled water

4. Calibration Procedures/Measurement Techniques

Probes differ as to specifics in use. Follow the manufacturer's instructions to obta accurate readings. The following general steps should be used to measure t dissolved oxygen concentration:

A. The equipment should be calibrated and its batteries checked in t laboratory before going to the field.

B. The probe should be conditioned in a water sample for as long a period practical before its use in the field. Long periods of dry storage followed l short periods of use in the field may result in inaccurate readings.

C. The instrument should be calibrated in the field before each measurement group of closely spaced measurements by placing the probe in water samp of known dissolved oxygen concentration (i.e., determined by Winkl method) or in a freshly air-saturated water sample of known temperatur DO values for air-saturated water can be determined by consulting a tab listing oxygen solubilities as a function of temperature and alini (Attachment A).

D. Immerse the probe in the sample. Be sure to provide for sufficient flow p the membrane, either by stirring the sample, or placing the probe in a flowi stream. Probes without stirrers placed in wells can be moved up and dow

E. Record the dissolved oxygen content and temperature of the sample in a fie logbook.

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F. Recalibrate the probe when the membrane is replaced, or as needed. Following the manufacturer's instructions. Duplicate analyses should agree within ± 0.1 mg/L.

Note than in in-situ placement of the probe is preferable, since sample handling is not involved. This however, may not always be practical. Be sure to record whether the liquid was analyzed in situ, or if a sample was taken.

Special care should be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interference.

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ATTACHMENT A

VARIATION OF DISSOLVED OXYGEN CONCENTRATION
IN WATER AS A FUNCTION OF TEMPERATURE AND SALINITY

Temperature	Chloride Concentration in Water					Difference /100 mg Chloride
	0	5000	10000	15000	20000	
	Dissolved Oxygen mg/l					
0	14.6	13.8	13.0	12.1	11.3	0.017
1	14.2	13.4	12.6	11.8	11.0	0.016
2	13.8	13.1	12.3	11.5	10.8	0.015
3	13.5	12.7	12.0	11.2	10.5	0.015
4	13.1	12.4	11.7	11.0	10.3	0.014
5	12.8	12.1	11.4	10.7	10.0	0.014
6	12.5	11.8	11.1	10.5	9.8	0.014
7	12.2	11.5	10.9	10.2	9.6	0.013
8	11.9	11.2	10.6	10.0	9.4	0.013
9	11.6	11.0	10.4	9.8	9.2	0.012
10	11.3	10.7	10.1	9.6	9.0	0.012
11	11.1	10.5	9.9	9.4	8.8	0.011
12	10.8	10.3	9.7	9.2	8.6	0.011
13	10.6	10.1	9.5	9.0	8.5	0.011
14	10.4	9.9	9.3	8.8	8.3	0.010
15	10.2	9.7	9.1	8.6	8.1	0.010
16	10.0	9.5	9.0	8.5	8.0	0.010
17	9.7	9.3	8.8	8.3	7.8	0.010
18	9.5	9.1	8.6	8.2	7.7	0.009
19	9.4	8.9	8.5	8.0	7.6	0.009
20	9.2	8.7	8.3	7.9	7.4	0.009
21	9.0	8.6	8.1	7.7	7.3	0.009
22	8.8	8.4	8.0	7.6	7.1	0.008
23	8.7	8.3	7.9	7.4	7.0	0.008
24	8.5	8.1	7.7	7.3	6.9	0.008
25	8.4	8.0	7.6	7.2	6.7	0.008
26	8.2	7.8	7.4	7.0	6.6	0.008
27	8.1	7.7	7.3	6.9	6.5	0.008
28	7.9	7.5	7.1	6.8	6.4	0.008
29	7.8	7.4	7.0	6.6	6.3	0.008
30	7.6	7.3	6.9	6.5	6.1	0.008
31	7.5					
32	7.4					
33	7.3					
34	7.2					
35	7.1					
36	7.0					
37	6.9					
38	6.8					
39	6.7					
40	6.6					
41	6.5					
42	6.4					
43	6.3					
44	6.2					
45	6.1					
46	6.0					
47	5.9					
48	5.8					
49	5.7					
50	5.6					

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Standard Operating Procedure

ORP Measurements on Ground and Surface Water Samples

1. APPLICABILITY

This method describes procedures to obtain accurate and reliable measurements of the field chemistry of water quality samples. Specifically, this method applies to measurement of oxidation-reduction potential.

The Field Sampling Plan (FSP) or other project plan may provide specific instructions about the parameters, test methods, and equipment for required field chemistry samples. Review the FSP for specific instructions that supersede the requirements specified in this procedure.

2. PROCEDURE

2.1 Preparation

Prior to performing field measurements, the following tasks must be completed:

1. Order equipment that will be needed for the task. Table 1 includes an example of a typical checklist for equipment and supplies that may be required for groundwater measurements. Verify the proper operation of all sample collection equipment and review associated operating procedures.

2. If sampling will be performed off-site, verify that permission has been granted to access the property.

3. Obtain a containment vessel for purged water.

4. Locate monitoring wells to be sampled and the appropriate decontamination area. Locate staging area and areas for managing purged water and expendable sampling materials.

5. Assemble the appropriate sampling equipment for field measurements, including flow through bath, tubing, and water quality instruments. Note that the flow through bath cannot be used if the bailer is used for sample collection.

6. Perform field calibrations for all instrumentation.

2.2. Documentation

Obtain a field notebook. The field notebook will be used to collect information on site conditions, personnel at the sample location during sampling, sample specific measurements that may be required, equipment calibrations, and anomalies observed during sampling. Sample identity information will also be entered into the notebook. In addition to the notebook entries, the FSP or applicable field procedures may also require collecting information on a standard form.

2.3 Operation

When practical, take final measurements of groundwater samples after at least three bore volumes have been purged from the well or sampling device, and as close to the time of sampling as possible.

Record measurements, instrument readings, calibration runs, and alkalinity determinations on the appropriate forms and/or in the field logbook.

2.3.1 Oxidation-Reduction Potential Measurement

A Hach One Portable pH Meter, or equivalent instrument, may be used to measure oxidation-reduction potential with the proper electrode (Hach OPR Electrode). First the meter must be zeroed for millivolt measurements.

1. With meter turned off, insert a paper clip into the BNC connector to electrically short the circuit.
2. Press and hold the button labeled "mV", simultaneously press and release the button labeled"!", then release "mV" button. The mv indicator will light, and after 10 seconds, the meter will read 0.0 mV +/- 0.2 mV. Remove paper clip and attach OPR electrode.
3. Verify the electrode response by measuring the oxidation-reduction potential of a calibration solution, prepared as specified in the Hach OPR Electrode manual. No calibration adjustment can be done with this meter; if electrode is not reading correct value, replace it with a new one Record the value on the water quality sampling record (Appendix 3.1).
4. Record observed reading on the water quality sampling record (Appendix 3.1).
5. Verify the electrode response after sample measurements by repeating Step 3. Record the value.

2.4. Post-operation Activities

1. Follow the requirements specified in the FSP for handling investigation-derived material (IDM).
2. Decontaminate sample equipment before using the equipment to purge or sample additional wells as required by the FSP.
3. Verify the completeness and accuracy of the logbook entries, and sign/initial and date all pa[illegible] Verify that all data collection forms are complete and sign/initiate and date any applicable forms.

Table 1

Example Checklist of Typical Equipment and Supplies

- ❑ pH measurements
  - _____ pH meter and probe
  - _____ pH calibration solutions
- ❑ Temperature measurements
  - _____ Laboratory-grade Celsius thermometer
- ❑ Electrical Conductivity measurements
  - _____ Electrical conductivity meter and probe
  - _____ Standard KCl calibration solutions
- ❑ Alkalinity measurements
  - _____ Alkalinity test kit (Hach)
  - _____ Titrant solutions
  - _____ Alkalinity QC solutions
  - _____ Volumetric flasks
- ❑ Dissolved Oxygen measurements
  - _____ Dissolved oxygen meter and probe
  - _____ Calibration bottle and sponge
- ❑ Oxidation-Reduction Potential measurements
  - _____ Hach ORP electrode
  - _____ pH meter
  - _____ Calibration solutions
- ❑ Flow through bath
- ❑ Teflon or plastic beakers
- ❑ Stirrers

TABLE 1, Continued

EQUIPMENT AND SUPPLIES CHECKLIST

- ❑ Plastic sheet
- ❑ Trash bags
- ❑ Cleaning wipes
- ❑ Storage containers for investigation-derived material, including waste decontamination solutions
- ❑ Disposable laboratory gloves
- ❑ Camera and film
- ❑ Any additional supplies listed in associated procedures, as needed

STANDARD OPERATING PROCEDURE
FIELD MEASUREMENT OF FERROUS IRON (Fe^{++})

1.0 Scope and Application

Although iron is the second most abundant element in the Earth's outer crust, concentrations present in water generally are small. The chemical behavior of iron and its solubility in water depends strongly on the oxidation intensity and pH in the system in which it occurs. The availability of iron in aqueous solution is effected by environmental conditions, including the degree or intensity of oxidation or reduction. Microorganisms are commonly involved in processes of oxidation and reduction of iron and some species may use these reactions as energy sources. Based on this information, the presence and extent of ferrous iron (Fe^{++}) may indicate whether an anaerobic degradation process due to depletion of oxygen, nitrate, and/or manganese is occurring.

The monitoring method discussed herein is limited to use of field testing equipment. Possible interference with turbidity may occur, therefore filtering must be completed on turbid samples. Samples must be shielded from direct sunlight and analyzed directly after sample collection.

2.0 Apparatus

The following equipment is needed to measure ferrous iron (Fe^{++}) concentrations:

- Iron, Ferrous, Test Kit (Hach Model IR-18C)

3.0 Reagents

- Ferrous iron reagent powder (1,10-phenanthroline)
- De-ionized water

3.0 Measurement Procedure

The following general steps should be used to measure ferrous iron (Fe^{++}) in a water sample:

- Fill a viewing tube to the first (5-ml) line with sample water. This is the blank sample.
- Place this tube in the top left opening of the color comparator.
- Fill the measuring vial to the 25-ml mark with sample water.
- Add the contents of one Ferrous Iron Reagent Powder pillow to the measuring vial.
- Swirl to mix. An orange color will develop if ferrous iron (Fe^{++}) is present. Allow three minutes for full color development.
- Fill another viewing tube to the first (5-ml) line with the prepared sample from the measuring vial.
- Place the second tube in the top right opening of the color comparator.
- Hold comparator up to a light source such as the sky, a window, or a lamp. Look through the openings in the front.
- Rotate the color disk until the color matches in the two openings.
- Read the mg/L ferrous iron (Fe^{++}) in the scale window.

STANDARD OPERATING PROCEDURE
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APPENDIX E

GUIDANCE FOR COMPLETING CLP/SAS PAPERWORK

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: 21 MAY 1991

SUBJECT: COMBINATION SAS PACKING LIST/CHAIN OF CUSTODY FORM

FROM: JAN PELS, RSCC

TO: ALL SAMPLERS

The new combination SAS packing list and chain of custody (COC) form has been printed. A limited supply of the form is available for a trial run to work out the bugs. Please use this form for any work you have coming up in the near future so that we can get your feedback for consideration when preparing the final revision. When you use this new form, please note any problems you have filling it out and forward these comments to me by June 5, 1991. Any improvements in the form will be made then prior to the final printing. Since this form was modeled after the combination TR/COC form, any improvements that will be made to the TR/COC form will automatically be incorporated into this form.

Please note that there are certain fields that are optional to fill out, such as samplers initials, split sample section, account code, and regional information. The concept with the new form is to eliminate redundant work by having all of the information that is currently written on the chain of custody form and on the traffic report form combined on one form, so keep this in mind when using the form.

If you are tracking bottle lot numbers, cooler numbers, etc. on the current COC forms, you can use the blank space across the middle of the form or blank space above the top border to write this information in.

Note that since you are listing tag numbers and preservatives, that you must list the analytical parameters in groups; this means that if you are collecting water samples for analytes such as total organic carbon, sulfate, chemical oxygen demand, total dissolved solids, etc., each group of analytes which will be taken out of a specifically preserved bottle should be listed on one line. See the attached example form.

Since these forms must be filled out on a per cooler basis, like the current chain of custody form, the fact that you can only list up to 10 samples shouldn't be an issue since you are limited by the size of the cooler. A second form can be filled out for additional samples in the cooler.

Remember to still write in the chain of custody seal numbers across the blank space between the SAS information and the COC information. It doesn't really matter where you list these numbers on the form as long as it doesn't interfere with other information on the form. The revised form will have a box for the COC seal numbers.

I must remind you that if you have more than one lab performing SAS analyses under the sa SAS number, you must assign different sample numbers each lab's samples. Since SAS sam numbers are not tracked in a database, the field length for SAS sample numbers is not fixe You can have sample numbers as high as E1000, as an example.

The lower right hand corner has a section for documenting whether split samples we accepted/declined by any PRPs. If you are currently using a separate form for this purpo please let me know. The Office of Regional Counsel will need to make a determination whether you can use this field in place of your current form. This issue was not raised dur the review that was done by the ORC.

Attached is an example of the form filled out. Please have samplers review this form carefu prior to using them in the field.

One major change is that the **top copy of the form is submitted to the RSCC**, with origi signatures, to include in the evidence file. SMO receives the second copy and the lab gets t bottom two copies. Once the lab signs their copies, the lab returns them to SMO and t Region, so that we will have their original signature for the the evidence file. **PLEAS STRESS THIS TO ALL FIELD SAMLERS, SINCE THIS DOCUMENT IS USED T TRACE THE SAMPLE FROM COLLECTION TO RECEIPT AT THE LAB AND IS VERY IMPORTANT DOCUMENT. You may make a photocopy to keep in your files,** b you must send us the original.

Please use this form as soon as you can in place of the old SAS packing lists and chain custody forms. Once our small supply is used up, we'll go back to the old forms until the fin revised forms are printed.

Remember that for samples shipped to the CRL, the regular Chain of Custody form must used with the CRL request forms. This combination form, as well as the TR/COCs are or for use in the CLP.

Please call if you have any questions on the use of this form.

Thank you.

EPA United States Enviromental Protection Agency
Contract Laboratory Program

Special Analytical Services Packing List/Chain of Custody

SAS No. 98ZG01

Case No. —

1. Project Code	Account Code	2. Region No. V	Sampling Co. WESTON	4. Date Shipped 10 Oct 97	Carrier FEDEX
Regional Information TFA102		Sampler (Name) TONYA BALLA		Airbill Number 9765423810	
Non-Superfund Program		Sampler Signature Tonya Balla		5. Ship To Argonne National Lab 9700 South Cass Avenue D-205 Argonne, IL 60439 ATTN: Fred Martino	
Site Name Ottawa Site - NPL-1		3. Purpose* Lead: [X] SF [ ] PRP [ ] ST [ ] FED; Early Action: [ ] CLEM [ ] PA [ ] REM; [ ] SI [ ] ESI [X] RI [ ] OIL [ ] UST; Long-Term Action: [ ] FS [ ] RD [ ] RA [ ] O&M [ ] NPLD			
City, State Ottawa, IL	Site Spill ID CP				

6. Matrix (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil
7. Waste
8. Other (Specify in Column A)

7. Preservative (Enter in Column D)
1. HCl
2. HNO3
3. NAHSO4
4. H2SO4
5. NAOH
6. Ice Only
7. Other (Specify in Column D)
N. Not Preserved

Sample Numbers (From Labels) 98ZG01	A Matrix (from Box 6) Other	B Conc.: Low Med High	C Sample Type Comp./ Grab	D Preservative (from Box 7) Other	E Analysis	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier NPL1-	H Mo/Day/ Year/Time Sample Collection	I Sampler Initials	J Field QC Qualifier B: Blank S: Spike D=Duplicate R=Rinsate PE=Perform. eval -=Not a QC Sample
S01	2	L	G	1	VOA	5-153978,79	MW01-01	10/9/97 0850	TCB	—
S02	2	L	G	1	VOA	5-153976,77	MW02-01	10/9/97 0915	TCB	—
D02	2	L	G	1	VOA	5-153826,27	MW02-01DP	10/9/97 0915	TCB	D(S02,D02)
S03	2	L	G	1	VOA	5-153829,30	MW03-01MSD	10/9/97 1350	TCB	—
R01	2	L	G	1	VOA	5-124629,30	MWTB-01	10/9/97 0810	TCB	B/BLANK
S04	2	L	G	1	VOA	5-223796,97	MW04-01	10/9/97 1735	TCB	—
R02	2	L	G	1	VOA	5-223798,99	MWFB-01	10/9/97 1600	TCB	B/BLANK

Shipment for SAS Complete? (Y) N)	Page 1 of 1	Sample(s) to be Used for Laboratory QC 98ZG01S03	Additional Sampler Signatures	Chain of Custody Seal Number(s) 138097, 138099

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Tonya Balla	10/10/97 1300				
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 Is custody seal intact? Y / N / none	

A21-012-7 REV. 3/94

DISTRIBUTION: White - Region Copy; Gold - Lab Copy for Return to Region; Yellow - Data User**; Pink - Lab Copy for Return to Data User**

EPA Form 9110-3

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

F1 200

EPA United States Enviromental Protection Agency
Contract Laboratory Program

Special Analytical Services Packing List/Chain of Custody

SAS No.: 98ZG01

Case No.: —

1. Project Code	
Account Code	
2. Region No.	V
Sampling Co.	WESTON
4. Date Shipped	10 Oct 97
Carrier	FEDEX
Regional Information	TFA102
Sampler (Name)	TONYA BALLA
Airbill Number	9765423810
Non-Superfund Program	
Sampler Signature	Tonya Balla
Site Name	Ottawa Site - NPL-1
City, State	Ottawa, IL
Site Spill ID	CP

3. Purpose*: Lead [X] SF [ ] PRP [ ] ST [ ] FED; Early Action [ ] CLEM [ ] PA [ ] REM; [ ] SI [ ] ESI [X] RI [ ] OIL [ ] UST; Long-Term Action [ ] FS [ ] RD [ ] RA [ ] O&M [ ] NPLD

5. Ship To: Argonne National Lab, 9700 South Cass Avenue, D-205, Argonne, IL 60439, ATTN: Fred Martino

6. Matrix (Enter in Column A): 1. Surface Water 2. Ground Water 3. Leachate 4. Field QC 5. Soil/Sediment 6. Oil 7. Waste 8. Other (Specify in Column A)

7. Preservative (Enter in Column D): 1. HCl 2. HNO3 3. NAHSO4 4. H2SO4 5. NAOH 6. Ice Only 7. Other (Specify in Column D) N. Not Preserved

Sample Numbers (From Labels) 98ZG01	A Matrix (from Box 6) Other	B Conc.: Low Med High	C Sample Type Comp./Grab	D Preservative (from Box 7) Other	E Analysis	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier NPL1-	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Field QC Qualifier B: Blank S: Spike D: Duplicate R: Rinsate PE: Perform eval -: Not a QC Sample
S01	2	L	G	1	VOA	5-153978,79	MW01-01	10/9/97 0850	TCB	—
S02	2	L	G	1	VOA	5-153976,77	MW02-01	10/9/97 0915	TCB	—
D02	2	L	G	1	VOA	5-153826,27	MW02-01DP	10/9/97 0915	TCB	D(S02,D02)
S03	2	L	G	1	VOA	5-153829,30	MW03-01MSD	10/9/97 1350	TCB	—
R01	2	L	G	1	VOA	5-124629,30	MWTB-01	10/9/97 0810	TCB	B/BLANK
S04	2	L	G	1	VOA	5-223796,97	MW04-01	10/9/97 1735	TCB	—
R02	2	L	G	1	VOA	5-223798,99	MWFB-01	10/9/97 1600	TCB	B/BLANK

Shipment for SAS Complete? (Y) N)	Page 1 of 1	Sample(s) to be Used for Laboratory QC	Additional Sampler Signatures	Chain of Custody Seal Number(s)
		98ZG01S03		138097, 138099

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Tonya Balla	10/10/97 1300				
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 Is custody seal intact? Y / N / none	

DISTRIBUTION: White – Region Copy; Gold – Lab Copy for Return to Region; Yellow – Data User**; Pink – Lab Copy for Return to Data User**

EPA Form 9110-3

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

A21-012-7 REV. 3/94

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: 20 AUGUST 1991

SUBJECT: COMBINATION TRAFFIC REPORT/CHAIN OF CUSTODY FORMS

FROM: JAN PELS, RSCC
Laboratory Scientific Support Section, CRL

TO: ALL REGION V SAMPLERS USING THE CONTRACT LABORATORY PROGRAM (CLP)

The latest printing of the combination traffic report (TR) and chain of custody (COC) forms has been completed. All versions of the traffic report form prior to this printing cannot be used after August 31. Please collect all old forms you have been sent and return them to me by September 15. Note that you should keep the old chain of custody forms, which will still be used for samples shipped to the EPA Central Regional Laboratory (CRL) in Chicago.

Please note that there are certain fields that are optional to fill out, such as sampler's initials, split sample section, and account code. The new form should eliminate redundant work by having all of the information that is currently written on the chain of custody form and on the traffic report form combined on one form, so keep this in mind when using the form.

If you are tracking bottle lot numbers, cooler numbers, etc., on the current COC forms, you can use the Regional Information box. If samples are being collected for a PRP oversight project, indicate 'PRP oversight' in the Regional Information box.

Remember that we do not request dissolved metals. Also, on the organic form, we are preserving all volatiles samples, including residential well samples as of February 1991, so you need to list the preservative number (HCl) in column D.

Note that since you are listing tag numbers and preservatives, that you must put the metals (water) sample information on one line and the cyanide information on another line. This would apply for the volatiles (waters will be preserved with HCl) and the BNA and Pest/PCBs fractions (they will have no preservative). Since the metals and cyanide fractions are taken from the same soil bottle, they can be written on one line.

Since these forms must be filled out on a per cooler basis, like the current chain of custody forms, the fact that you can only list up to 10 samples shouldn't be an issue since you are limited by the size of the cooler. A second form can be filled out for additional samples in the cooler; this revision has a box to note the number of pages, i.e., forms sent in a cooler.

This revision of the forms has a box for chain of custody seal numbers (remember that we use two seals per cooler).

The inorganic form has new analytes listed (nitrate/nitrite, fluoride, pH and conductivity). Th nitrate/nitrite and fluoride is planned for a new low level water (residential well) RAS SOW, an the pH and conductivity are optional tests under the current High Concentration SOW. The lo lever water inorganic RAS SOW is not in place yet, so these fields should not be used at th time.

Please call if you have any questions on these additional analytes. For routine inorgan analyses on water and soil samples, you still will only ask for total metals and/or cyanide.

The organic form also has an additional field for the current High Concentration SOW, t ARO/TOX. Please call if you have questions about this high concentration analysis. F routine organic analyses on water and soil, you will still only ask for VOA, BNA and/ Pest/PCB.

Note that the organic traffic report says that extra volume is required for the matrix spike/matr spike duplicate. This only applies to the routine low/medium/water/soil RAS SOW Remember that organic residential well samples (currently a RAS plus SAS) have Performan Evaluation (PE) samples sent with real samples to the lab and that you do not col ext volume for this contract; you must list the PE samples on this form. You will not have t numbers for these PEs, nor time/date of sampling, etc. Note which are PE ampules in t designated field QC field.

The lower right hand corner has a section for documenting whether split samples we accepted/declined by any PRPs. If you are currently using a separate form for this purpos please let me know. The Office of Regional Counsel (ORC) will need to make a determinati on whether you can use this field in place of your current form. This issue was not rais during the review that was done by the ORC.

Use the far right empty field to notate which samples are field blanks, field duplicates (i. which samples are duplicates of which others), PE samples, and which are blind QC samp (we rarely send double blind QC samples). This information does not transfer onto the la copies.

There is a field to list the sample number that should be used for laboratory QC analyses (mat spike/matrix spike duplicate or spike/duplicate). Please be sure to always designate a sam on a one per 20 samples/per matrix basis per case. Remember that you must supply ex volume for all water samples, except for the organic residential well samples shipped to the C as a RAS/SAS.

Note that the bottom section of the form has a box for the laboratory receiving the samples sign and note whether the custody seal is intact; there has been some confusion on the par some field samplers, who have occasionally filled in this box.

One major change is that the top copy of the form is submitted to the RSCC, with origi signatures, to include in the evidence file. SMO receives the second copy and the lab gets

bottom two copies. Once the lab signs their copies, the lab signs them and returns them to SMO and the Region, so that we will have their original signature for the the evidence file. **PLEASE STRESS THIS TO ALL FIELD SAMLERS, SINCE THIS DOCUMENT IS USED TO TRACE THE SAMPLE FROM COLLECTION TO RECEIPT AT THE LAB AND IS A VERY IMPORTANT DOCUMENT.** You may make a photocopy to keep in your files, but you must send the RSCC the original.

Attached is an example for the forms correctly filled out. Please have samplers review this form carefully prior to using them in the field.

Also attached are instructions prepared by SMO on the use of the forms. Remember that these instructions are general instructions and do not contain Regional requirements; Region V's requirements supercede these instructions (specifically regarding the chain of custody seal numbers being listed, and tracking tag numbers). Please call if you have any questions on Region V's requirements.

Below is a list of things to remember when using these forms:

1) These forms are for exclusive use in the CLP. The old COC forms will still be used for samples shipped to the EPA Central Regional Laboratory (CRL).

2) A TR/COC form must be filled out on a per cooler basis.

3) The top copy with the original signatures is sent to the Region V RSCC.

4) All samples contained in a cooler must be listed on the form, including field blanks, field duplicates, and any performance evaluation (PE) samples sent with field samples.

5) The 'dissolved' metals box on the inorganic TR/COC should not be checked off. Filtered and unfiltered metals samples must be given separate EPA sample numbers, listed separately on the ITR/COC and both should have 'total' metals checked off on the form.

6) Extra sample volume must be sent for all organic and inorganic water samples on a one per twenty sample basis, with the exception of the organic residential well samples sent under a RAS/SAS contract.

7) Each TR/COC must be signed and dated properly.

Again, these forms must be used beginning September 1 and all other revisions of these forms must be returned to me by September 15.

Please call if you have any questions or comments on these forms. Another printing will be done within the next two months, so there is still time to correct problems with the forms.

Attachments

SAMPLER INSTRUCTIONS FOR USE OF MULTI-SAMPLE ORGANIC AND INORGANIC TRAFFIC REPORTS/CHAIN-OF-CUSTODY FORMS EPA FORM #9110-1 (INORGANICS) AND FORM 9110-2 (ORGANICS)

1/20/89
REV. 2/91
REV. 7/91

A. Introduction - Samples and Sample Numbers

The Contract Laboratory Program (CLP) Organic and Inorganic Multi-Sample Traffic Reports/Chain-of-Custody Forms (TRs) document samples shipped to CLP laboratories. They also enable SMO and the Region to track samples and ensure that the samples are shipped to the appropriate contract laboratory. You must use TRs each time you ship Routine Analytical Services (RAS) samples to a CLP laboratory. The TRs may document up to 10 samples shipped to one CLP laboratory under one Case Number and RAS analytical program.

Please note that the TR includes a chain-of-custody record which is located at the bottom of the form. The form is used as physical evidence of sample custody. According to EPA enforcement requirements, official custody of samples must be maintained and documented from the time of collection until the time the samples are introduced as evidence in the event of litigation. You are responsible for the care and custody of the sample until sample shipment.

A sample is considered to be in your custody if any of the following criteria are met:

1. The sample is in your possession or is in your view after being in your possession,
2. The sample was in your possession and then locked up or sealed to prevent tampering, or
3. The sample is in a secured area.

CLP sample types are defined by the RAS analytical program. There are currently three organic/inorganic programs: Low/medium concentration inorganic, low/medium concentration organic, and high concentration organic. Low/medium inorganic samples may be analyzed for Total Metals, Cyanide or both. Low/medium organic samples may be analyzed for Volatile Organics (VOAs), Base/Neutral/Acid (BNAs), Pesticide/PCBs, or any combination of these. High concentration organic samples may be analyzed for VOAs, BNA/Pesticide/PCBs, and Aroclors/toxaphenes. Inorganic samples are documented on Inorganic TRs. Organic and high concentration samples are documented on Organic TRs.

A CLP sample is one matrix - water or soil - never both. The CLP sample is further defined as consisting of all the sample aliquots from one station location, for each matrix and RAS analytical program. For example, let's say you were sampling at Pond A. You plan to collect one water sample and one soil/sediment sample, each to be analyzed for VOAs, BNAs, Pesticide/PCBs, Total Metals and Cyanide. All the bottles for the organic water analyses at this station - VOA vials, BNA jars, and Pesticide/PCB

jars - make up one organic CLP sample, not three. All of the bottles for the organic soil analysis make up the second organic CLP sample. The bottle for inorganic soil analysis makes up the second inorganic CLP sample from Pond A. Even though you have collected a water and a soil for five different analyses from Pond A, you've collected four CLP samples - an organic water, organic soil, inorganic water, and an inorganic soil.

The CLP generates unique Sample Numbers that must be assigned to each organic, and inorganic sample. The unique CLP Sample Numbers are printed at SMO on adhesive labels and distributed to the Regional RSCC as requested. It is your responsibility to assign this critical Sample Number correctly and to transcribe it accurately on the TR.

Organic Sample Numbers are in the format XX123, and have ten labels per strip: four for extractables, two for VOAs, and four blank (extra). (See Attachment 1.) DESTROY THE UNUSED LABELS to prevent duplication of Sample Numbers.

Inorganic Sample Numbers are in the format MXX123 and have seven labels per strip: two for Total Metals, two for Cyanide and three extra (see Attachment 1). Remember that the unique Sample Number must only be used once. DESTROY THE EXTRA LABELS.

Use only the labels provided to the Region in which you are sampling. CLP Sample Numbers are alphabetically coded to correspond with each Region as follows:

Letter Code Organic	Letter Code Inorganic	Region	Letter Code Organic	Letter Code Inorganic	Region
A	MA	I	F	MF	VI
B	MB	II	G	MG	VII
C	MC	III	H	MH	VIII
D	MD	IV	Y	MY	IX
E	ME	V	J	MJ	X

REMEMBER:

- TRs must be used for each Case No. with every shipment of samples to each CLP laboratory.
- Organic samples, high concentration samples, and inorganic samples are assigned separate, unique Sample Numbers. Each sample consists of all the sample aliquots from a sample station location for analysis in one of the three analytical programs.
- A CLP RAS sample will be analyzed as either a water or a soil sample.
- Prevent accidental duplication of Sample Numbers by destroying unused labels.
- Use the Sample Numbers specific to your Region.
- Call SMO (telephone number 703/557-2490 or 703/684-5678) if you have any questions about using TRs.

B. Completing the Form - Case Documentation

Enter the SAS No. (if applicable) and Case No. at the top right of the form. Complete the boxes in the header:

Box No. 1

Project Code/Site Information

Enter the Project Code; Account Code; Regional Information; Non-Superfund Program; Site Name; City, State, and Site Spill Identifier in the designated spaces.

If sampling is not under the Superfund program, enter the name of program, e.g., RCRA, in the box titled "Non-Superfund program".

Box No. 2

Regional Information:

Enter the Region number, the name of your sampling company, and your name and signature in the designated spaces.

Box No. 3

Type of Activity:

Check Funding Level of sampling. Next check the code which describes the task of the sampling mission:

	Funding Level	
SF	-	Superfund
PRP	-	Potential Responsible Party
ST	-	State
FED	-	Federal
	Pre-Remedial	
PA	-	Preliminary Assessment
SSI	-	Screening Site Investigation
LSI	-	Listing Site Investigation
	Remedial	
RIFS	-	Remedial Investigation Feasibility Study
RD	-	Remedial Design
O&M	-	Operations and Maintenance
NPLD	-	National Priorities List
Delete		

Removal

CLEM	-	Classic Emergency
REMA	-	Removal Assessment
REM	-	Removal
OIL	-	Oil Response
UST	-	Underground Storage Tank Response

Box No. 4

Shipping Information:

Enter the date shipped, the carrier for example; Federal Express, Purolator, c Airborne and the airbill number in the appropriate spaces.

Box No. 5

Ship To:

Enter the name of the CLP Laboratory contact (Sample Custodian), laboratory an its full address in the box.

Box No. 6

Preservative

Box provides a list of commonly used preservatives. Please enter the appropriat preservative used in Column D.

Box No. 7

Sample Description

Box provides a list of the description/matrices of samples that are collected Please enter appropriate description in Column A.

C. **Completing the Form - Sample Documentation**

Carefully transcribe the CLP Sample No. from the printed sample labels on the TR i the space provided.

Complete columns A through G to describe the sample:

Column A, Sample Description:

Enter the appropriate sample description code from Box 7.

NOTE: Describe BLANKS as #3 "Leachate" in Column A.

Note: Item #6 "Oil" and Item #7 "Waste" are for RAS PLUS SAS projects onl Do not ship oily samples or waste samples without making prior arrangements wit SMO.

Column B. Concentration:

Organic - If sample is estimated to be low or medium concentration, enter "L". When shipping RAS Plus SAS High Concentration samples (previously arranged with SMO), enter "H".

Inorganic - Enter "L" for low concentration, "M" for medium concentration, and "H" for high concentration (under previous RAS Plus SAS arrangement).

NOTE: Ship medium and high concentration organic and inorganic samples in metal cans.

Column C: Sample Type Composite/Grab

Please enter which type of sample you collected.

Column D: Preservative Used

Please enter preservation used (i.e., HCL, NAOH, HNO_3, H_2SO_4) refer to Box 6 or the reference number of the preservation (1-7,N).

Column E: RAS Analysis:

Check the analytical fractions requested for each sample, for example, VOAs, SVs, and Pesticides are for low/medium concentration organics. Total metals and cyanide are for low/medium concentration inorganics. VOAS, SV/Pesticides and Aroclors are for high concentration organics. Metals, Cyanide, pH/conductivity are for high concentration inorganic samples.

Note: Either total or dissolved metals can be requested for each individual inorganic sample assigned a unique sample number, but not both analyses. ARO/TOX analyses can only be requested for high concentration samples.

Column F: Regional Specific Tracking Number or Tag Numbers.

Enter the Region Specific tracking number or tag number in the space provided.

Column G: Station Location Number:

Enter the station location in the space provided.

Column H: Mo/Day/Year/Time of Sample Collection:

Record the month, day, year, and time in military time (e.g. 1600 hours = 4:00 pm), of sample collection.

Column I: Sampler Initials

Enter your initials

Column J: Corresponding CLP Organic/Inorganic Sample No.:

Enter the corresponding CLP Sample No. for organic or inorganic analysis.

Column K: Designated Field QC

Enter the appropriate qualifier for "Blind" Field QC samples in this column.

Please note that all samples must have a qualifier.

Blind Field QC	Qualifier
Blind Blanks	B
Blind Field Duplicates	D
Blind Field Spikes	S
Blind PE Samples	PE
Not a QC Sample	___

Box Titled "Shipment for Case Complete (Y/N)":

This should reflect the status of the samples scheduled at a lab for a specific Case. When ALL samples scheduled/collected for shipment to a lab for a specific Case have been shipped the Case is complete.

Box Titled "Page 1 of "

Please enter the number of TRs per shipment.

Box Titled "Sample used for Spike and /or Duplicate"

Please enter sample used for spike and or duplicate sample.

Box Titled "Additional Sampler Signatures"

Please record any additional sampler signatures you are unable to record in Box 2.

Box Titled "Chain of Custody Seal Number"

Sampler should enter the Chain of Custody Seal Number if applicable.

Box Titled "Split Samples Accepted/Declined":

Sampler should ask sight owner, PRP, etc. whether they want split samples taken. The split samples are either accepted or declined. Sampler should record their signature and check the appropriate box.

How and when to separate and send Traffic Report/Chain of Custody form copies:

When all paper work has been completed by the sampler and samples are ready to be shipped the bottom two copies of the Traffic Report/Chain of Custody forms should be placed in a plastic bag and taped to the inside of the cooler. The second copy of the Traffic Report/Chain of Custody form must be returned to the SMO within five days of the sample shipment. The top copy is the Regions' copy for their records.

D. Instructions on the Reverse

Instructions summarizing CLP sample volumes, packaging and shipment reporting requirements are printed on the back of the TRs.

APPENDIX F

MOBILE LABORATORY SOP AND MDL STUDY

ESAT REGION V
STANDARD OPERATING PROCEDURES FOR FIELD ANALYTICAL SUPPORT PROJECTS

TITLE:

VOLATILES BY MASS SPECTROSCOPY IN WATER AND SOIL/SEDIMENT

Approvals:

Ziyad Rajabi
Lockheed ESAT Region V
Team Manager

_____

Mel Kaminsky
Lockheed ESAT Region V
FASP Group Leader

_____

Tom Sedlacek
Lockheed ESAT Region V
FASP Chemist

_____

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1 SCOPE, APPLICATION AND LIMITATIONS

1.1 PURPOSE

This Field Analytical Support Project (FASP) method is proposed for use in determining the concentrations of various volatile organic analytes (VOA) in soil/sediment and water samples.

1.2 LIST OF COMPOUNDS

This method is applicable to a wide range of organic [illegible] including the compounds listed in Table 1. Approximate [illegible] quantitation-limits are 5 ug/L for 5 ml waters, 1 ug/L for [illegible] waters and 5 ug/Kg for soils when using the calibration level recommended in section 5.5.

1.3 USER RESTRICTIONS

The method should be used only by trained analysts under the supervision of an experienced chemist.

1.4 ANALYTES IDENTIFIED

The method yields identification and quantitation of the analytes listed in Table 1. Reported values are on as-received basis - no dry weights are used.

1.5 QUALITY CONTROL

This FASP SOP should be used in conjunction with the FASP SOP for quality control (QA-ADIT-00).

1.6 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical is to be viewed as a potential health hazard. From this point of view, exposure to these chemicals is to be reduced to the lowest possible level. The laboratory maintains a current file of Material Safety Data Sheets (MSDS) which cover the safe handling and the associated risks of the chemicals used in this method. It is the employees responsibility to be familiar with this information.

The samples to be analyzed are from known or suspected hazardous waste sites and potentially may contain hazardous organic and/or inorganic materials at high concentration levels. Be aware of the potential hazards associated with the handling and analysis of these samples.

2 SUMMARY OF METHOD

2.1 PURGE & TRAP

Five or twenty-five mL of water sample or five g of soil sam[illegible] with five mL of reagent water is placed in a [illegible] sample is purged with helium onto a trap and is [illegible] GC/MS system

2.2 SAMPLE ANALYSIS

Analysis is performed using a temperature programmed gas chromatograph with a capillary column and a mass selective detector (MSD). Identification is based on comparison of [illegible] tion times and mass spectra between samples and standards. Quantitation is based on the internal standard method. All [illegible] and QC samples and standards within the batch will be quantitated using the same method.

3 INTERFERENCES

Interferences may be minimized by use of purge & trap grade reagents, exhaustive cleanup of glassware, and avoidance of solvents in the laboratory. The analytical system must be demonstrated to be free from contamination under conditions of the analysis by running method blanks. Purge and trap or GC interference by sample carryover may be minimized by use of disposable glassware during sample preparation and employing a bake-out after the purge cycle. Interferences extracted from samples are matrix and site-specific. High concentration levels of volatile compounds require proof that no carry-over has occurred. Exhaustive clean-up of purge & trap system may be required.

4 APPARATUS AND MATERIALS

4.1 ANALYTICAL SYSTEMS

Listed below are GC specifications that meet the requirements of this method. Other GC configurations may be substituted if they meet the method requirements.

4.1.1 Gas Chromatograph
An analytical system complete with a temperature programmable GC and all necessary accessories is required.

4.1.2 Column - 75 m x 0.53 mm I.D. x 3um film DB-624 fused silica capillary column (J&W Scientific).

4.1.3 Purge & Trap system Tekmar 2000LCS and Tekmar Presep II automatic sampler with Supelco VOCARB 4000 trap.

4.1.4 Detector - MSD must be capable of scanning from [illegible] amu every second or less, using 70 volts (nominal) electron energy in the electron impact mode and producing a mass spectrum which meets the tuning criteria (see Table 2).

4.1.5 Data System - a computer system interfaced to the MSD that allows continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the chromatographic program. The computer must have software that allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an extracted ion current profile (EICP). Software must also be available that allows integrating the abundance of any EICP between specified time or scan number limits. The operational data system must be able to flag the data files that have been edited manually by the operator.

4.1.6 Gas Supply - The carrier gas should be ultrapure helium.

4.2 OTHER LABORATORY EQUIPMENT

4.2.1 40 mL VOA vials with teflon lined septa.

4.2.2 Mininert or equivalent vials and septa.

4.2.3 Disposable Pipets - Pasteur, 6 inches long.

4.2.4 Spatulas - Stainless steel, micro and semimicro.

4.2.5 Microsyringes - Assorted sizes.

4.2.6 Balance - Top loading, capable of weighing to 0.01 g, used to weigh samples.

4.2.7 Volumetric Pipets - Class A, 0.5, 1.0, 5, 10, and 25 mL.

4.2.8 Volumetric Flasks - 10, 25, 50, 100 mL.

4.2.9 Leak Detector - GOW-MAC Gas or equivalent leak detector.

4.2.10 Drying Oven - Capable of maintaining temperatures of greater than or equal to 200°C.

5 REAGENTS

5.1 Methanol -purge & trap quality or equivalent.

5.2 Reagent Water - Reagent water is defined as water in which no interference is observed at the retention time above the FASP

Quantitation Limit (FQL) for the analyte of interest. Reagent water may be generated using a carbon filter bed containing activated carbon (Calgon Corporation, Filtrasorb-300 or equivalent) or a water purification system (Milli-Q Plus with Organex Q cartridge or equivalent). The water should be purged with nitrogen prior to use.

5.3 GASES

5.3.1 Helium - Ultra pure or chromatographic grade (always used in conjunction with an oxygen trap).

5.3.1 Nitrogen - Ultra pure or chromatographic grade. Used to purge reagent water.

5.4 STOCK STANDARD SOLUTIONS

Stock standard solutions or analytes should be purchased as manufacturer certified solutions.

5.5 CALIBRATION STANDARDS

Prepare working standards through volumetric dilution of the stock standards with methanol. Prepare calibration standards by spiking the working standard into reagent water. Recommended calibration levels are 1, 2, 5, 10 and 25 ug/L for 25 ml water; 5, 25, 50, 100 and 150 ug/L for 5ml waters and ug/Kg for soils. The working standard should be prepared so that the minimal amount of methanol is spiked into the calibration standard. Excessive methanol degrades both the chromatography and the analyte response. For example, a working standard prepared at [illegible] ug/mL would be spiked at 1, 5, 10, 20 and 30 ul to [illegible] recommended final concentration of the calibration [illegible] soils and 5 ml waters.

A gas working standard may be kept separate from the remaining analytes working standard. The two separate working standards are spiked into water to make the calibration standard. More than two separate working standards are not recommended due to the amount of methanol that would be in the highest concentration standard. Gas standards should be replaced weekly and the other standards should be replaced monthly.

Ketones may be prepared at higher levels than the other analytes. A 5x increase in concentration is usually sufficient to insure these analytes can be detected at all five calibration levels.

All working standards must be stored below 0°C in Teflon-sealed glass mininert vials with no head space.

5.6 MATRIX SPIKE SOLUTIONS

Matrix spike solutions should be prepared by dilution of stock standard solutions with methanol so that samples will be spiked with 50 ng on column. This level corresponds to the recommended third calibration level for 5ml waters and soils. See Table [illegible] recommended MS analytes and recovery limits. The MS is required after every twenty samples.

The Tekmar Precept II will automatically add 1 uL of a MS solution. A 50 ng/uL standard is used to spike at the recommended level.

5.7 SYSTEM MONITORING COMPOUNDS (surrogates)

Surrogate solutions should be prepared by dilution of stock standard solutions so that samples will be spiked with 125 ng on column. See Table 4 for recommended surrogate analytes and recovery limits. One surrogate per sample may be out of control limits.

5.8 INTERNAL STANDARDS

Internal standards should be prepared by dilution of stock standard solutions with methanol. Samples should be spiked with 125 ng on column. See Table 1 for the analytes corresponding to each internal standard. Internal standard areas in the sample must be 50% to 200% of the value in the corresponding [illegible] calibration.

The internal standards and surrogates may be included in the working standard. The Tekmar Precept II will automatically add 1 uL of an internal standard/surrogate solution. A 50 ng/uL standard prepared with both internal standards and surrogates is used to spike at the recommended levels.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples should be collected, handled, preserved and shipped maintaining a chain of custody following current EPA regulations and recommendations at the time of sample collection.

The use of chain-of-custody records as described in the U.S. EPA "CLP Users Guide" (9240.0-1), December, 1988, is required for sample tracking in the field and subsequent internal lab sample tracking. The maximum holding time for soil samples is 14 days. The maximum holding time for waters is seven days. If waters are acid preserved to a pH of less than 2, hold time is increased to 14 days.

7 CALIBRATION

7.1 TUNING

Prior to any analysis the MSD must pass tuning criteria (Table 2) using 50 ng of bromofluorobenzene (BFB). This standard must be analyzed at the beginning of each 24 hour period during which samples or standards are analyzed. The time period for a GC/MS system tune and standards calibration (initial or continuing calibration) begins at the moment of injection of the BFB standard.

7.2 INITIAL CALIBRATION

After an experienced chromatographer has ensured that the entire chromatographic system is functioning properly; i.e., conditions exist such that resolution, retention times, response reporting, and interpretation of chromatographic spectra are within acceptable quality control limits, the GC may be calibrated (Section 11). Using five calibration standards for each target analyte prepared as described in Section 5.5, initial calibration curves are generated for each target analyte (refer to Section 11 for chromatographic procedures).

The percent relative standard deviation (%RSD, see Section 11) is computed to determine the acceptability of the calibration curve. The %RSD must be <30 percent. The initial calibration must be rerun if more than seven analytes fail %RSD limit. Any time the GC/MS system is altered (new column, ion source cleaning, or change in oven temperature program) a new initial calibration curve must be established.

7.3 CONTINUING CALIBRATION

The GC/MS system is checked after every tune with a continuing calibration. The mid-range initial calibration standard is used. The RRF of the continuing calibration is compared to the average RRF of the initial calibration to check the system (see Section 11). The %D between the continuing calibration RRF and the initial calibrations average RRF must be <30 percent. The continuing calibration must be rerun if more than seven analytes fail %D limit. A total of seven analytes, including initial and continuing calibration, may be out of control. If the continuing calibration will not meet required criteria a new initial calibration is required. A continuing calibration remains valid for the 24 hour tune cycle providing the GC system remains unaltered during that time.

8 PURGE & TRAP

A method blank is required for every matrix and for every group of samples. It must be analyzed after every continuing calibration. The method blank is reagent water or sand taken through every step of the analytical sequence to prove systems are clean of contaminants.

8.1 Soil/Sediment Samples, low level.

8.1.1 Add 5 grams of sample to a tared 40 mL VOA vial. [illegible] weight.

8.1.2 The Tekmar Presept II can be programed to automatically spike the correct amount of water, internal standard and surrogate. A total of ten mLs of water is added. Fifty ng of each internal standard and surrogate is recommended.

8.1.3 For every twenty samples a matrix spike is required. The Tekmar Precept II can be programed to automatically spike this sample. Fifty ng of each spike compound is recommended.

8.1.4 Samples with analyte concentrations over the calibration range must be diluted, unless the site specific requirements do not require dilutions to be analyzed. Soil sample size must not be below one gram. If further dilution is required, a medium level analysis must be used.

8.2 Soil/Sediment Samples, medium level.

8.2.1 Add 5 grams of sample to a 40 mL VOA vial.

8.2.2 Add 5 mL of methanol to the vial and cap.

8.2.3 Let the solvent extract the volatile components from the soil for ten minutes. Do not shake the sample as this may release the volatile analytes.

8.2.4 Transfer the solvent layer to a vial with zero headspace.

8.2.5 Add 100 uL of the sample to 5 mL of reagent water.

8.2.6 Transfer the sample to a 40 mL VOA vial.

8.2.7 The Tekmar Presept II can be programmed to automatically spike the correct amount of water, internal standard and surrogate. A total of ten mLs of water is added. Fifty ng of each internal standard and surrogate is recommended.

8.2.8 For every twenty samples a matrix spike is required. The Tekmar Precept II can be programmed to automatically spike this sample. Fifty ng of each spike compound is recommended.

8.2.9 Samples with analyte concentrations over the calibration range must be diluted, unless the site specific requirements do not require dilutions to be analyzed. Methanol extract sample size must not be below one microliter. If a smaller sample size is needed, a further dilution of the extract with methanol is required.

8.3 Water Samples.

8.3.1 Add 5 or 25 mL of sample to a 40 mL VOA vial.

8.3.2 The Tekmar Presept II can be programmed to automatically spike the correct amount of water, internal standard and surrogate. A total of 5 mLs of water is added.

8.3.3 For every twenty samples a matrix spike is required. The Tekmar Precept II can be programmed to automatically spike this sample. Fifty ng of each spike compound is recommended.

8.3.4 Samples with analyte concentrations over the calibration range must be diluted, unless the site specific requirements do not require dilutions to be analyzed. Water sample size must not be below one microliter. If a smaller sample size is needed, a dilution of the water sample with reagent water is required.

8.3.5 The above method analyzes the water sample as if it were a soil. The Tekmar Precept II also has the capability to run water samples directly from the 40 mL VOA vial the sample was in originally. This frees the analyst from measuring the 25 or 5 mL sample size. The water method is only recommended for sites with lower levels of volatile analytes. There is a much greater probability of carry over from sample transfer lines and the common water purge tube with the water method. Samples analyzed as waters cannot be heated. Different five point calibrations, continuing calibrations and blanks are required if the soil and water modes of operation are both used.

9 INSTRUMENTAL ANALYSIS

9.1 INSTRUMENT PARAMETER

9.1.1 Purge & Trap

Standby: 30° C
Preheat: 40° C 2 min.
Purge time: 11 min.

Desorb: Preheat: 240° C
Desorb: 250° C for 6 min.
Bake: 260° C for 4 min.
Valves and lines: 150° C
Purge flow: 30 ml/min.
Purge pressure: 150kPa

9.1.2 GC/MS

Temperature Program: Initial temperature: 45° C for 3 min. 8° C/min. to 190° C, no hold, 10° C/min. to 240° C, no hold. (run time 26.13 min.)

Injector Temperature: 250° C, constant pressure at 13 psi (45°).

Transfer Line Temperature: 250° C

Mass selective detector: 70 volts, 35 to 500 amu mass range, scan time not to exceed 1 second per scan.

Other instruments, columns, and chromatographic conditions may be used if FASP QC criteria are met.

9.2 CHROMATOGRAMS

Computer reproductions of chromatograms that are attenuated to ensure that all peaks on scale over a 100-fold range are acceptable. However, this can be no greater than a 100-fold range. This is to prevent retention time shifts by column or detector overload. Generally, peak response should be >25 percent and less percent of full-scale deflection to allow visual recognition of the various PAH compounds. The following information must be recorded on each chromatogram.

1) Instrument and detector identification.

2) Column phase, length, and I.D.

3) Oven temperature.

4) Injector and Detector temperature.

5) Carrier and make-up gas.

6) Site name.

7) Sample number and matrix.

8) Date and time.

9) GC operator initials.

9.3 TARGET ANALYTE IDENTIFICATION

Qualitative identification is based on retention time and mass spectra as compared to standards.

9.4 ANALYTICAL SEQUENCE

1) Tune; repeat within 24 hours of previous tune
2) Initial or continuing calibration.
3) Associated QC lot method blank.
4) Samples and associated QC matrix spike.

10 CALCULATIONS

10.1 INITIAL CALIBRATION

GC/MS response to target analytes is measured by determining relative response factors (RRF). Calculate the RRF for each target analyte in the initial standard. The data system may be used to make all of these computations.

$$RRF = A_x/A_{is} \times C_{is}/C_x \qquad \text{Equation [illegible]}$$

A_x = Area of the characteristic ion for the [illegible] measured

A_{is} = Area of the characteristic ion for the [illegible] internal standard

C_{is} = Amount of internal standard injected (ng)

C_x = Amount of the compound to be measured injected (ng)

Using the response factors, calculate the percent relative standard deviation (%RSD) for each target analyte at each concentration level using the following equation.

$$\%RSD = SD / \bar{X} \times 100 \qquad \text{Equation 10.1.2}$$

where SD, the Standard Deviation, is given by

$$SD = \sqrt{\frac{\sum (X_i - \bar{X})^2}{N - 1}}$$ Equation [illegible]

where:
X_i = individual response factor (per analyte),
$\bar{X}$ = mean of initial three calibration factors (per analyte),
N = number of calibration standards.

The %RSD must be less than 25.0 percent.

10.2 CONTINUING CALIBRATION

Mid-range standards for all initial calibration target analytes must be analyzed every 24 hours. The maximum allowable percent difference (%D) calculated using the equation below for each analyte must be <30 percent.

$$\%D = (RRF_c - RRF_i) / RRF_i \times 100$$

where: RRF_i = mean Rf from the initial calibration for each analyte
RRF_c = measured Rf from the continuing calibration for the same analyte

10.3 SAMPLE QUANTITATION

Calculate the concentration of the target analytes in the sample using the following equation for internal standards. See Table 1 for internal standards and their associated target analytes. See Table 5 for the recommended characteristic ions and secondary ions. All field and QC samples and standards within the batch will be quantitated using the same method. Samples are calibrated from the continuing calibration.

For soil samples:

$$\text{Concentration in ug/Kg} = (A_x)(I_s)(Df)/(A_{is})(RRF)(W_s)$$

For water samples:

$$\text{Concentration in ug/L} = (A_x)(I_s)(Df)/(A_{is})(RRF)(V_s)$$

where: A_x = area of the characteristic ion for the analyte to be measured

I_s = amount of internal standard injected [illegible]
Df = dilution factor if used
A_{is} = area of the characteristic ion for the internal standard
RRF = response factor from the appropriate continuing calibration for the analyte.
W_s = weight of sample extracted in g
V_o = volume of sample in mL

For soil samples, the results are reported in [illegible] kilogram (ug/Kg) without correction for [illegible] samples will be reported in ug/L. No sample [illegible] corrected because of blank or spike recoveries.

Any target compound found at concentrations of less than one half of the MDL value will be reported as not detected at the MDL value. Any target compound found at concentrations at or above one half of the MDL value will be reported as detected at that concentration and flagged as estimated "J".

QC criteria (as described in FASP QC SOPs) must be met for all analyses. Advisory limits for the matrix spike are presented in Table 3. Internal standard areas in the samples must be 50% to 200% of the value in the corresponding continuing calibration. Surrogate requirements are listed in Table 4. One surrogate may be out of control limits per sample. Samples that fail internal standard or surrogate requirements are considered estimated. Samples with less than ten percent recovery for an internal standard or two or more surrogates must be reanalyzed to confirm results.

11 DELIVERABLES

11.1 FIELD DATA SAMPLE REPORTS

A field data sample report should be available from the laboratory within 24 hours of sample analysis.

11.2 FINAL FASP REPORT

The final FASP report generated for each project should include the following considerations:

11.2.1 A reference to the FASP method used and a comment addressing any unusual occurrences.

11.2.2 A hard copy of all data and summary sheets documenting required QA/QC data (available within 14 days of completion of all FASP analyses for a project).

11.2.3 A data summary of all reportable results with appropriate units clearly specified.

11.2.4 All calculations are preformed using standard good measurement practices in determining significant figures. Results will be rounded for final deliverables.

11.2.5 All sample results are reported using two significant figures.

11.2.6 A statement, by analyte, that initial, and continuing calibration meet required FASP QC criteria.

All results must be annotated (followed by the flag F) by the laboratory to indicate to future data users that FASP techniques were used in sample analysis.

12 SAMPLE AND DATA STORAGE

12.1 DISPOSAL OF SAMPLES

Samples should be disposed of in accordance with established Federal, State, and local regulations and policies after completion of analysis by the analytical team on site. Sample extracts may be disposed of after final FASP field data report submission. The responsibility for proper disposal of samples, glassware and extracts rests with the on-site authority. The mobile laboratory is not licensed to transport hazardous waste.

12.2 RAW AND SUMMARY DATA STORAGE

The lab must maintain a hard copy and computer disk storage of all raw (including instrument printouts and logbooks) and summary data associated with an analytical case for a minimum of 6 months after receipt of the hard copy report by the data user.

12.3 PERMANENT DATA STORAGE

After the 6-month period has elapsed, the laboratory should place all records, including raw data and laboratory notebooks, into permanent storage.

Table 1: Target analytes and associated internal standards.

Fluorobenzene
Dichlorodifluoromethane
Chloromethane
Bromomethane
Vinyl Chloride
Chloroethane
Trichlorofluoromethane
1,1-Dichloroethene
1,1,2-Trichloro-1,2,2-trifluoromethane
Acetone
Carbon Disulfide
Methyl Acetate
Methylene Chloride
trans-1,2-Dichloroethene
Methyl tert-Butyl Ether
1,1-Dichloroethane
cis-1,2-Dichloroethene
2-Butanone
Chloroform
1,2-Dichloroethane
Benzene-d6 (surrogate)
1,1,1-Trichloroethane
Cyclohexane
Carbon tetrachloride
Benzene
Trichloroethene
Methylcyclohexane
1,2-Dichloropropane
Bromodichloromethane
cis-1,3-Dichloropropene
4-Methyl-2-pentanone

Clorobenzene-d5
trans-1,3-Dichloropropene
1,1,2-Trichloroethane
Dibromochloromethane
Toluene
Tetrachloroethene
2-Hexanone
1,2-Dibromoethane
Chlorobenzene
Ethylbenzene
total Xylenes

Styrene
Isopropylbenzene
1,1,2,2-Tetrachloroethane
Bromoform
Toluene-d8 (surrogate)
Bromofluorobenzene (surrogate)

1,2-Dichlorobenzene-d4
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-Chloropropane
1,2,4-Trichlorobenzene

Table 2: Tune parameters, BFB ion abundance criteria

mass 50; 8.0% to 40.0% of mass 95
mass 75; 30% to 66% of mass 95
mass 95; base peak, 100% relative abundance (note mass 174).
mass 96; 5% to 9% of mass 95
mass 173; less than 2% of mass 174
mass 174; 50% to 120% of mass 95
mass 175; 4% to 9% of mass 174
mass 176; 93% to 101% of mass 174
mass 177; 5% to 9% of mass 176

Table 3: Matrix spike recovery

	Water & soil %R
1,1-Dichloroethene	70-130
Trichloroethene	70-130
Benzene	70-130
Toluene	70-130
Chlorobenzene	70-130

Table 4: Surrogate recovery limits

	Water & soil %R
Benzene-d6	70-130
Toluene-d8	70-130
Bromofluorobenzene	70-130

Table 5: Recommended characteristic ions.

	Primary	Secondary
Dichlorodifluoromethane	85	-
Chloromethane	50	[illegible]
Bromomethane	94	[illegible]
Vinyl Chloride	62	[illegible]
Chloroethane	64	[illegible]
Trichlorofluoromethane	101	[illegible]
1,1-Dichloroethene	96	[illegible]
1,1,2-Trichloro-1,2,2-trifluoromethane	101	85, 151
Acetone	43	58
Carbon Disulfide	76	78
Methyl Acetate	43	74
Methylene Chloride	84	49, 51
trans-1,2-Dichloroethene	96	61
Methyl tert-Butyl Ether	73	43, 57
1,1-Dichloroethane	63	65
cis-1,2-Dichloroethene	96	61
2-Butanone	43	72
Chloroform	83	[illegible]
1,2-Dichloroethane	62	64
1,1,1-Trichloroethane	97	99
Cyclohexane	56	69, 84
Carbon tetrachloride	117	119, 121
Benzene	78	
Trichloroethene	130	[illegible]
Methylcyclohexane	83	55, 98
1,2-Dichloropropane	63	65, 114
Bromodichloromethane	83	85, 129
cis-1,3-Dichloropropene	75	77
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	99, 132
Bromoform	173	171, 175
4-Methyl-2-pentanone	43	58
Toluene	91	92
Tetrachloroethene	164	166, 129
2-Hexanone	43	100
Dibromochloromethane	129	208, 206
1,2-Dibromoethane	107	109
Chlorobenzene	112	114
Ethylbenzene	91	106
Xylenes	91	106
Styrene	104	78
Isopropylbenzene	105	120
1,1,2,2-Tetrachloroethane	83	85

Dichlorobenzenes	146	[illegible]
1,2-Dibromo-3-Chloropropane	75	175, 155
1,2,4-Trichlorobenzene	180	[illegible], [illegible]
Benzene-d6 (surrogate)	84	82
Toluene-d8 (surrogate)	98	100
Bromofluorobenzene (surrogate)	95	174
Fluorobenzene (internal standard)	96	70
1,4-Dichlorobenzene-d4 (internal standard)	150	152
Chlorobenzene-d5 (internal standard)	117	119

Stated Method Dection Limits for VOA analysis by Mass Spectrometer

	water ug/L		soil ug/Kg	
	calculated	Reporting	calculated	Reporting
FLUOROBENZENE	ISTD		ISTD	
Dichlorodifluoromethane	1	1	4	5
Chloromethane	1	1	2	5
Vinyl chloride	1	1	11	15
Bromomethane	1	1	16	20
Chloroethane	1	1	8	10
Trichlorofluoromethane	1	1	3	5
1,1,2-Trichloro-1,2,2-trifluoromethane	2	2	3	5
1,1-Dichloroethene	1	1	2	5
Acetone	2	5	9	25
Carbon disulfide	1	1	5	5
Methylene chloride	1	1	8	10
Methyl Acetate	1	1	13	15
trans-1,2-Dichloroethene	2	2	37	50
1,1-Dichloroethane	3	3	6	10
2-Butanone	11	11	9	25
cis-1,2-Dichloroethene	4	4	21	25
Methyl tert-Butyl Ether	1	2	24	25
1,2-Dichloroethane	1	1	2	5
Chloroform	3	3	31	50
BENZENE-D6	1	SURR	3	SURR
4-Methyl-2-pentanone	2	5	8	25
Carbon tetrachloride	2	2	2	5
1,1,1-Trichloroethane	2	2	21	25
Cyclohexane	2	2	21	25
Benzene	1	1	2	5
Trichloroethene	1	1	2	5
Methylcyclohexane	2	2	2	5
1,2-Dichloropropane	1	1	1	5
Bromodichloromethane	1	1	2	5
cis-1,3-Dichloropropene	1	1	3	5
CHLOROBENZENE-D5	ISTD		ISTD	
trans-1,3-Dichloropropene	1	1	2	5
1,1,2-Trichloroethane	1	1	2	5
Dibromochloromethane	1	1	2	5
Bromoform	1	1	3	5
Toluene	1	1	1	5
TOLUENE-D8	1	SURR	1	SURR
Tetrachloroethene	1	1	2	5
2-Hexanone	1	5	7	25
1,2-Dibromoethane	1	1	2	5

Chlorobenzene	1	1	2	5
Ethylbenzene	1	1	3	5
m- &/or p-Xylene	1	1	4	5
o-Xylene	1	1	2	5
Styrene	3	3	3	5
Isopropylbenzene	1	1	2	5
BFB	1	SURR	2	SURR
1,1,2,2-Tetrachloroethane	1	1	3	5
1,2-DICHLOROBENZENE-D4	ISTD		ISTD	
1,3-Dichlorobenzene	1	1	2	5
1,4-Dichlorobenzene	1	1	2	5
1,2-Dichlorobenzene	1	1	2	5
1,2-Dibromo-3-chloropropane	1	1	3	5
1,2,4-Trichlorobenzene	1	1	3	5