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RD/RA PROJECT PLANS
for the
FORMER JUNKER LANDFILL
Hudson, Wisconsin

August 29, 1996

Prepared for
Wisconsin Department of Natural Resources

DAMES & MOORE

2701 International Lane, Suite 210, Madison, WI 53704

Dames & Moore Project No. 33178-002

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1.0 INTRODUCTION

This document presents the management plans for the remedial design (RD) and remedial action (RA) at the Junker Sanitary Landfill Site (JSL) located in the Town of Hudson, St. Croix County, Wisconsin.

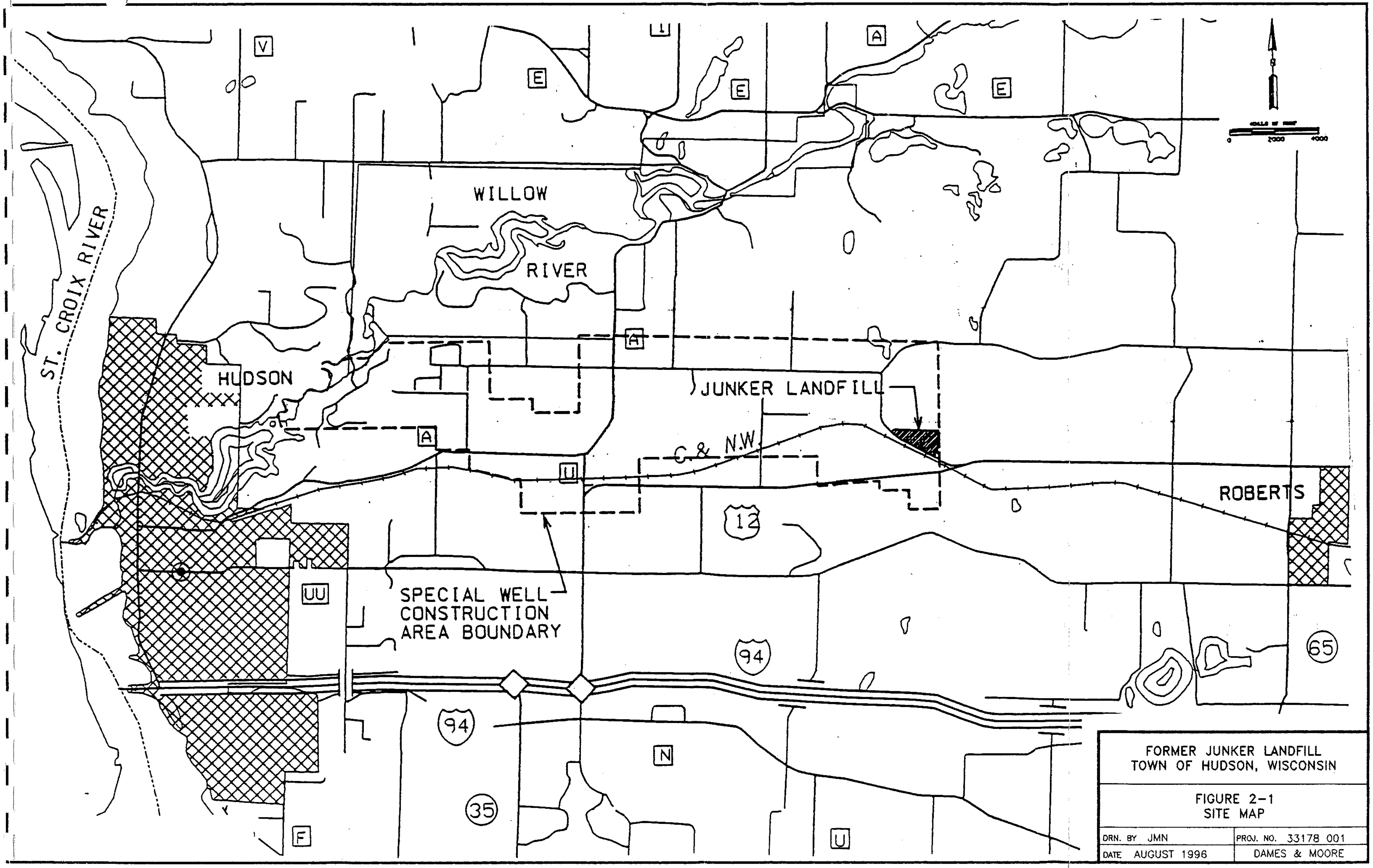
The work plan outlines the policy and organizational structure for completing the RD/RA. It also describes the rationale and approach to the project, summarizes the tasks to be performed, outlines a schedule, and identifies the project personnel and their qualifications.

In addition, concise Quality Assurance Project Plan (QAPP) and Monitoring Program Plan (MPP) as well as a Health and Safety Plan have been included with the work plan.

2.0 BACKGROUND

The Junker Sanitary Landfill (JSL) site is located in southwestern St. Croix County, Wisconsin, in the southeast corner of Section 13, Hudson Township, as shown in Figure 2-1. Originally a gravel pit, landfill operations were initiated in 1972 under Walfred Pilquist. In April 1973, Garry Thompson purchased the landfill and continued its operation. The landfill was leased to Junker Sanitary Landfill, Inc., in 1977. During operation, the site received municipal solid waste, sewage sludges, demolition and commercial/industrial wastes. Since its closure in 1987, the landfill has been covered, regraded, capped and had an active gas extraction system installed. Approximately 200 cubic feet per ~~second~~ minute of landfill gas currently is being extracted and flared.

In 1994-1995, Wenck Associates, Inc., performed a remedial investigation/feasibility study (RI/FS) of the JSL site. Data on groundwater and landfill gas migration were collected and an evaluation of remedial options performed. The WDNR approved several options for remediating contaminant conditions caused by releases of the landfill waste. This work plan has been prepared in conjunction with the Record of Decision (ROD) on the site detailing the approach to complete these proposed remedial actions.



FORMER JUNKER LANDFILL
TOWN OF HUDSON, WISCONSIN

FIGURE 2-1
SITE MAP

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3.0 QUALIFICATIONS OF PERSONNEL

3.1 DAMES & MOORE PERSONNEL

Dames & Moore with the assistance of Short, Elliott and Hendrickson, has assembled a team experienced in the various requirements of this project. The project organization and key individuals of the Dames & Moore project team are indicated on Figure 3-1. The project will be managed out of the Madison Dames & Moore office, with assistance for field activities and planning from the Minneapolis Dames & Moore office.

Project leadership and primary staff will be composed of personnel familiar with anticipated activities. Our project team will provide experience in hydrogeologic analysis, environmental engineering, risk assessment, and remedial design. Brief descriptions of key project team members follow, with detailed Curriculum Vitae located in Appendix A.

Principal-In-Charge

Mr. Christopher E. Weggemann will serve as the Principal-In-Charge. He will be responsible for overseeing the project in Dames & Moore's Minneapolis office and providing the resources required to ensure a successful project. Mr. Weggemann has 10 years of environmental consulting experience.

Project Manager

Mr. David P. Trainor, will serve as Project Manager. He is the Managing Principal-In-Charge of the Madison office and has more than 16 years of experience in the environmental field. Mr. Trainor has managed numerous environmental projects throughout the United States, including RCRA, CERCLA, and state programs. Mr. Trainor will be responsible for overall technical supervision and quality assurance/quality control.

Project Hydrogeologist

Dr. Robert S. Guthrie, will serve as the Project Hydrogeologist and will manage day-to-day project investigation sampling, analysis and report preparation. Dr. Guthrie will evaluate the geologic and hydrologic information collected during field investigations. Dr. Guthrie has 10 years of professional environmental experience performing and managing hydrologic investigations, including remedial investigations and feasibility studies.

Project Engineering Manager

Mr. Gary L. Boley, will serve as the Project Engineering Manager. Mr. Boley has more than 25 years of project development, management and engineering experience. He has been responsible for the design and construction of major public and private projects, varying in size from a few hundred thousand to more than $200 million.

Staff Geologist

Mr. Dirk W. Leemkuil, will serve as the Staff Geologist. His duties include supervising field project activities. Mr. Leemkuil has 8 years of environmental consulting experience where he has specialized in performing subsurface investigation; design, installation and operation of remediation systems; and management of personnel services related to the performance of these activities.

Design Engineer

Mr. J.P. Walker will be the project Design Engineer. He has 17 years of experience in the design and construction of civil and environmental engineering projects. A majority of the projects have been in the waste management discipline.

Staff Engineer

Ms. Julie Hoffman will be the Staff Engineer. She will assist with the design and implementation of this project. Ms. Hoffman has 3 years of process engineering experience. She has assisted with the design of landfill gas extraction and groundwater remediation systems.

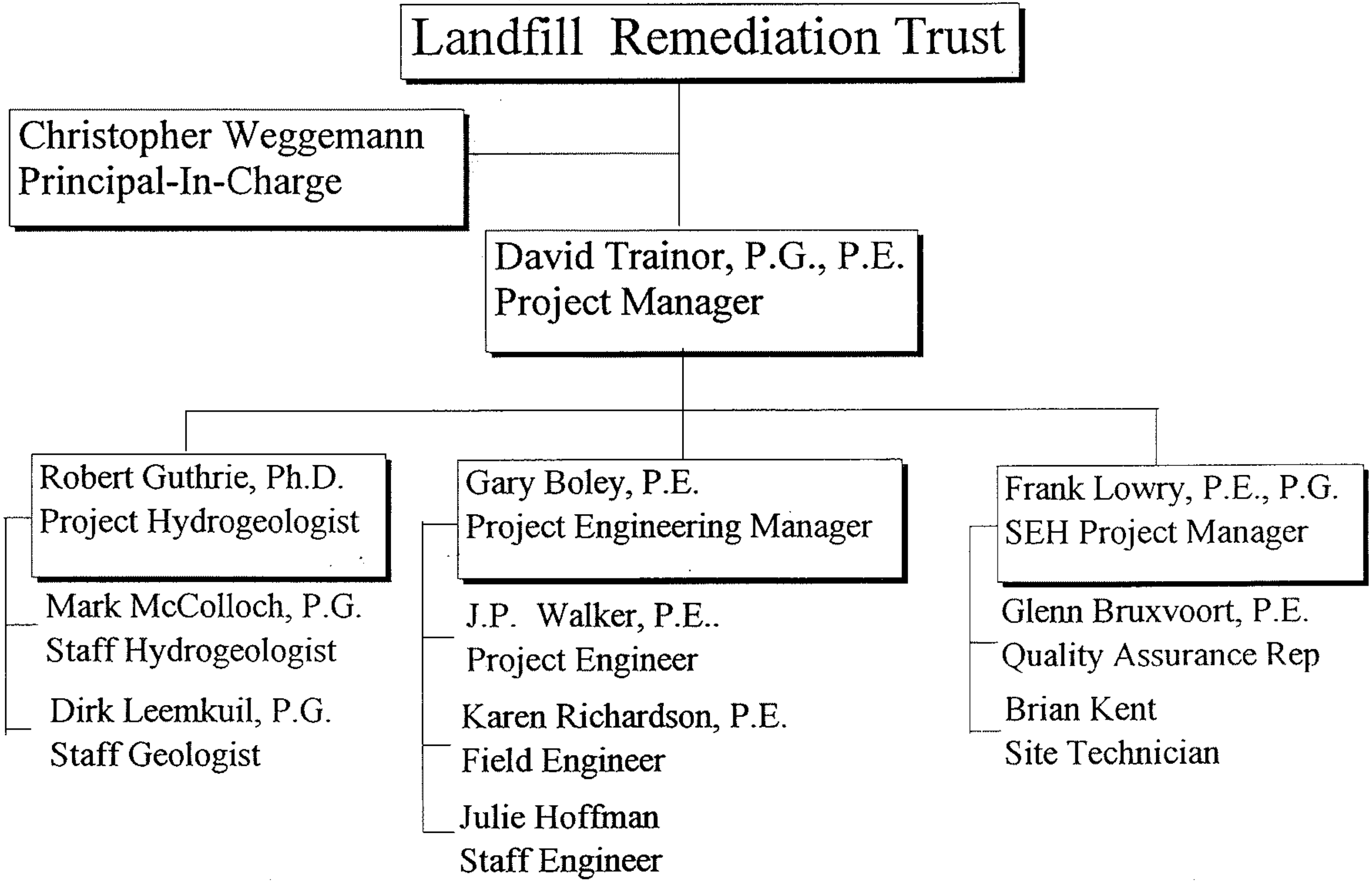
Field Engineer and Health & Safety Officer

Ms. Karen Richardson will provide construction oversight and serve as the Health and Safety Officer. She has 12 years experience as a civil and environmental engineer in the design and construction of environmental and highway engineering projects.

Staff Hydrogeologist

Mr. Mark McColloch will be staff hydrogeologist. Mr. McColloch has seven years of experience in providing environmental services. He has conducted numerous site investigations requiring the identification and characterization of soil and groundwater contamination at solid waste disposal sites.

Figure 3-1: Project Organization - RD/RA
Junker Landfill Site



4.0 SCHEDULE

A project schedule has been developed based on the estimated duration of the tasks outlined in Sections 6.0 and 7.0 of this plan. The project schedule is provided as Figure 4-1 and contains tasks to be performed during 1996 and 1997. The schedule identifies target start dates and completion dates. Several activities are contingent on other project goals. These items have special symbols that are referenced at the base of the table.

The schedule is contingent on a number of factors. Review by the WDNR for each major document is assumed to be four weeks. Inclement weather may cause delays in the various field activities. However, the intent of the schedule is to meet the deadline requirements stipulated in the Consent Decree.

Figure 4-1: PROJECT SCHEDULE
Junker Sanitary Landfill
Remedial Design and Remedial Action

	1996																						1997											
TASK (Week Ending)	8/2	8/9	8/16	8/23	8/30	9/6	9/13	9/20	9/27	10/4	10/11	10/18	10/25	11/1	11/8	11/15	11/22	11/29	12/6	12/13	12/20	12/27	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
Task 1-RD/RA Plans																																		
1/A-Description & Qualification of Personnel	✓	░			▓																													
1/B- Schedule for Completion of Tasks	✓	░			▓																													
1/C-Pre-Design Investigative Work Plan - Draft/Final	✓	░			▓																													
1/C - Pre-Design Investigation Activites																																		
1. Radius of influence-gas extraction system						░	▓																											
2a. Groundwater VOC sampling @ Town Hall								░		x	▓																							
2b. VOC & Inorganic sampling of groundwater in SWCA								░		x	▓																							
3. VOCs measurements at gas extraction wells & probes								░		x		▓								▓														
4. Evaluate Seeps								░		x		▓				▓				▓			▓	▓	▓	▓	▓	▓	▓	▓	▓	▓	▓	▓
5. Define extent of clay ✓								░		x	▓																							
6. Survey of landfill area					░	▓																												
7. Install gas probes @ N,E,& S boundary										░	▓																							
8. Excavate refuse N of landfill & regrade subsidence areas ♦								░		▓																								
9. Leachate Mound Analysis (well installation & pumping test)																									░	▓								
10. Obtain Access Agreements			░				▓																											
1/D-Investigative Waste Plan	✓	░			▓																													
1/E-Draft/Final Community Relations Plan	✓	░			▓																													
Install GAC filters												░										▓												
GAC System Sampling														░	X								▓	+										
Task 2-Remedial Design																																		
2/A-Health & Safety Plan					▓																													
2/B-Quality Assurance Project Plan					░															▓														
2/C-Design Plans																																		
1. Construction & Operation Schedule												░								▓														
2. Draft Design, Plans & Specs												░								▓							○	●						
3. Operation & Maintenance Plan																																		
4. Draft Design, Cost Estimate												░								▓														
5. Construction Quality AssurancePlan																																		
2/D-Monitoring Program Plan					░															▓														
2/E-Design Phases (see 2/C)																																		
2/F-Additional Studies																																		▓
2/G-Progress Meetings														▓		▓		▓																
Task 3 - Remedy Construction																													░	▓				
1. Preconstruction Meeting																												▓						
2. Prefinal Inspection																															▓			
3. Final Inspection																															▓			
Task 4 - Progress Reports												▓				▓				▓			▓	▓	▓	▓	▓	▓	▓					
Task 5-Constr Completion Report																																		
1. Draft Construction Completion Report																																	▓	
2. Final Construction Completion Report																																		▓
Task 6-Remedy Documentation																																		
1. Draft Completion Remedy Report																																		⊠
2. Final Completion Remedy Report																																		⊠

░ Target initiation of activity

▓ Target completion of activity

X Initial GAC sampling of effluent will occur within 15 working days of installation

\+ Annual sampling of GAC systems hereafter

○ Bid Documents

● Bidding (assumes 3 week bid period)

⊠ Due 14 years after construction completion document

♦ Assumes survey (I/C 6.) is complete within specified time, and no weather delays occurs.

5.0 INITIAL EVALUATION

5.1 SITE DESCRIPTION

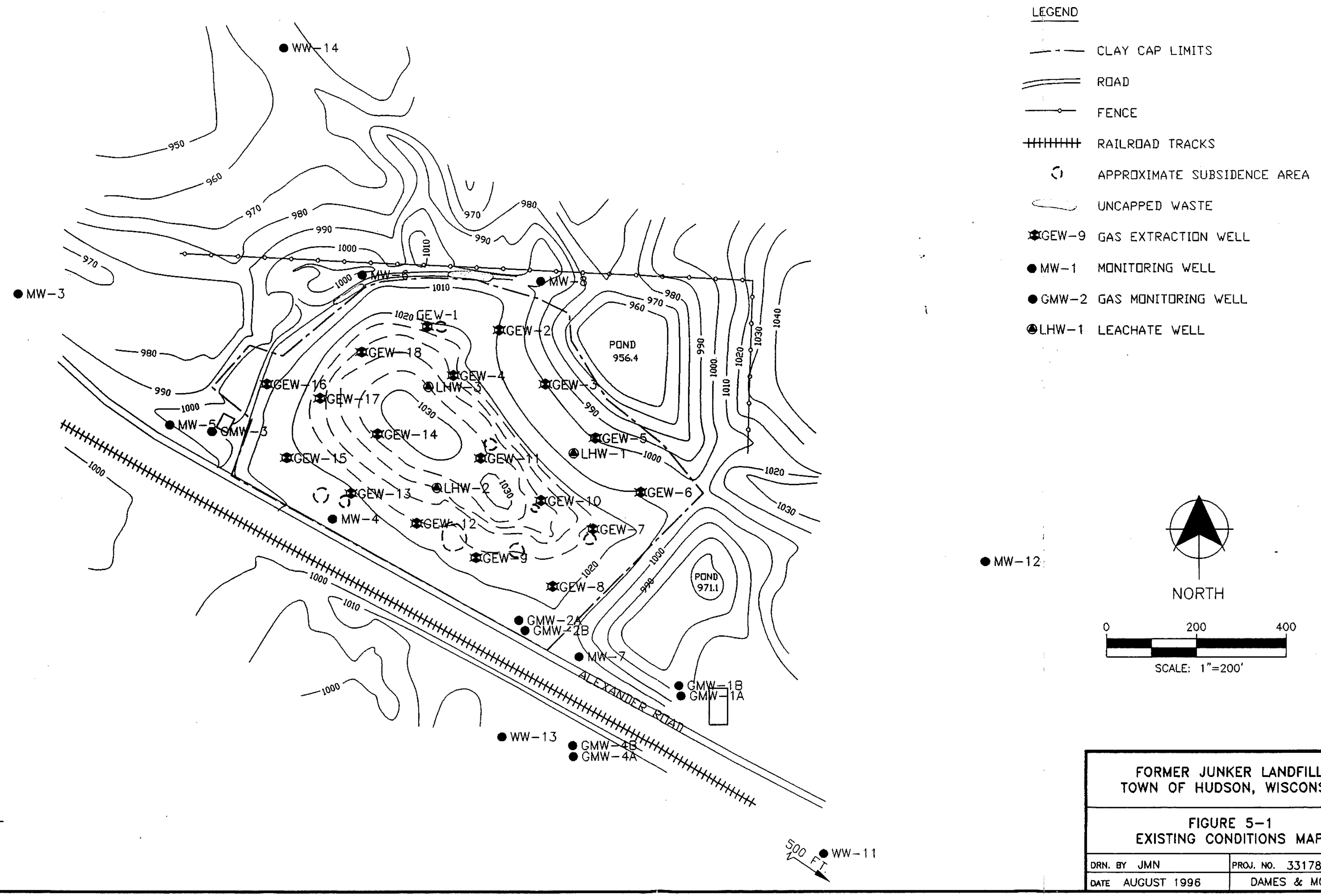
The site is located on approximately 40 acres in the SE¼ of the SE¼ and the SW¼ of the SE¼ of Section 13, Township 29 North, Range 19 West, St. Croix County, Wisconsin. Municipal and industrial/commercial wastes, and sewage sludge and demolition wastes were disposed in the 15-acre fill area. The wastes were placed in the landfill from north to south, but there is no documentation of placement method.

The site is bordered on the west and south by Alexander Road, on the east by a residential farmstead and on the north by property owned by the Girl Scouts of America (Rolling Ridges Girl Scout Camp). The Chicago and North Western Railroad (Union Pacific) runs parallel to Alexander Road, along the southwest side.

The site currently has a Chapter NR 504 cap, an active gas extraction system and a leachate extraction system. There is also a series of gas monitoring probes, groundwater monitoring wells and various monitoring points associated with the gas and leachate extraction systems, as shown in Figure 5-1. The leachate extraction system, installed as a dual unit in conjunction with the gas system, is not currently operated.

The cap consists of two feet of clay, one and one-half feet of grading material and six inches of topsoil. The permeability of the clay barrier was documented during construction to be less than 1×10^{-8} cm/sec for discrete samples. The landfill site is well vegetated, with surface water runoff directed toward one of two sedimentation ponds adjacent to the landfill.

The gas extraction system consists of 18 gas extraction wells, a blower and flare, which are operated to control methane gas migration from the JSL site while minimizing oxygen intrusion. Gas extraction well EW-16 currently does not operate, as it is not located in the waste. The extracted gas flows through a header system and is destroyed by a ground flare. Condensate collected from the gas extraction system is stored in a 4,000 gallon holding tank, located at the flare station. Although the extraction system is currently being monitored, landfill gas has been measured in monitoring probes located southeast of the property.



FORMER JUNKER LANDFILL
TOWN OF HUDSON, WISCONSIN

FIGURE 5-1
EXISTING CONDITIONS MAP

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5.2 GEOLOGY

The surficial sediments at the JSL site consist of sandy ice-contact stratified drift of Late Wisconsinan Superior Lobe glacial till. Unconsolidated material of glacial origin extends from 40 to 100 feet deep below these sediments. The underlying bedrock is the Prairie du Chien Group, a fractured dolomite, which may be up to 300 feet thick. In places, it may be capped by the St. Peter's Sandstone. The surficial sediments are the likely conduit for off-site migration of landfill gases.

5.3 HYDROGEOLOGY

Groundwater is used extensively for residential, commercial, industrial and agricultural purposes. Potable water is obtained primarily from the upper portion of the Prairie du Chien formation. Regional groundwater flow in the area of the site is west-northwest toward the St. Croix River. Residences and businesses within the SWCA receive bottled water. This SWCA was formed in 1991 when volatile organic contaminants were detected in private groundwater well samples.

6.0 WORK PLAN OBJECTIVES AND RATIONALE

The objectives of the remediation of the JSL site are to develop a community relations plan, achieve and maintain safe drinking water in the vicinity of the landfill, restore groundwater to standards over time and prevent unsafe levels of landfill gas from migrating off landfill property. These objectives are divided into the categories Community Relations Plan, Groundwater, Water Supply, Landfill Gas Migration, Seep Investigation and Access Agreements.

6.1 COMMUNITY RELATIONS PLAN

The community relations plan (CRP) for this project will explain activities at the JSL site, including remedial design, planned or existing remedial activities, the schedule, or any changes to the original remedy. The final CRP has been submitted with this document.

6.2 GROUNDWATER

The program whereby bottled water is delivered to the residences accepting the service will continue. Kandiyohi Bottled Water Company of Willmar, Minnesota is the bottled water supplier and Jensen Distributors of River Falls, Wisconsin distributes the water. Currently, 54 residences receive bottled water at their request.

Determination of the extent of VOC migration in the groundwater will begin by sampling the well at the Town Hall. If VOCs are found in the Town Hall well, sampling of additional wells will determine the migration of contaminated groundwater, as stipulated in the Scope of Work.

Prior to repairs and extension of the clay cap, further definition of the waste boundary is required. The area north of the landfill which is currently not under the clay cap will be excavated. This excavated material will be placed in areas on the landfill where subsidence has occurred. The existing clay cap will be removed in subsided areas, excavated material will be placed to reestablish grades, and the clay cap restored. Improvements in the existing drainage swale along Alexander Road are recommended, to allow surface water flow and prevent ponding in low-lying areas along the swale. Prior to developing design plans for this effort, the extent of clay previously removed for construction of the gas extraction system (1992) will be confirmed. This will be accomplished through advancing approximately 200 shallow soil probes within 3-6 feet a pump test will be performed to determine the proper pump design for leachate pumps. If the leachate exists as describe din the Wenck report, additional gas extraction wells will likely be placed in the area of the mound. Within these wells (as well as other extraction wells in the vicinity), leachate pumps will be added to the wells, designed for discharge to the condensate collection system.

* most include evaluation, of headersystem + effects of gas system

Balancing and upgrading the current system through addition of gas extraction wells and gas monitoring probes should eliminate the need for a vapor barrier system at the southeast corner of the landfill. During gas extraction well installation, items such as header pipe integrity will be investigated. If necessary, piping will be repaired, and sloped to aid draining of condensate and leachate from the lines. In addition, valves may be installed along the header pipe length to assist in vacuum measurements and discerning where header pipe head loss occurs. Currently, the valve assemblies on the gas extraction wells are being replaced by SEH with assemblies that are also able to pump leachate.

go back through Gas Control Report

The approximate locations of soil borings for determining the extent of clay (along the gas header pipe), additional gas monitoring probes and the gas extraction wells are shown in Figure 6-1. Note that the placement of the gas extraction wells will depend on the results of the radius of influence test and the VOC landfill gas measurements, and thus, is subject to modification.

6.5 SEEP INVESTIGATION

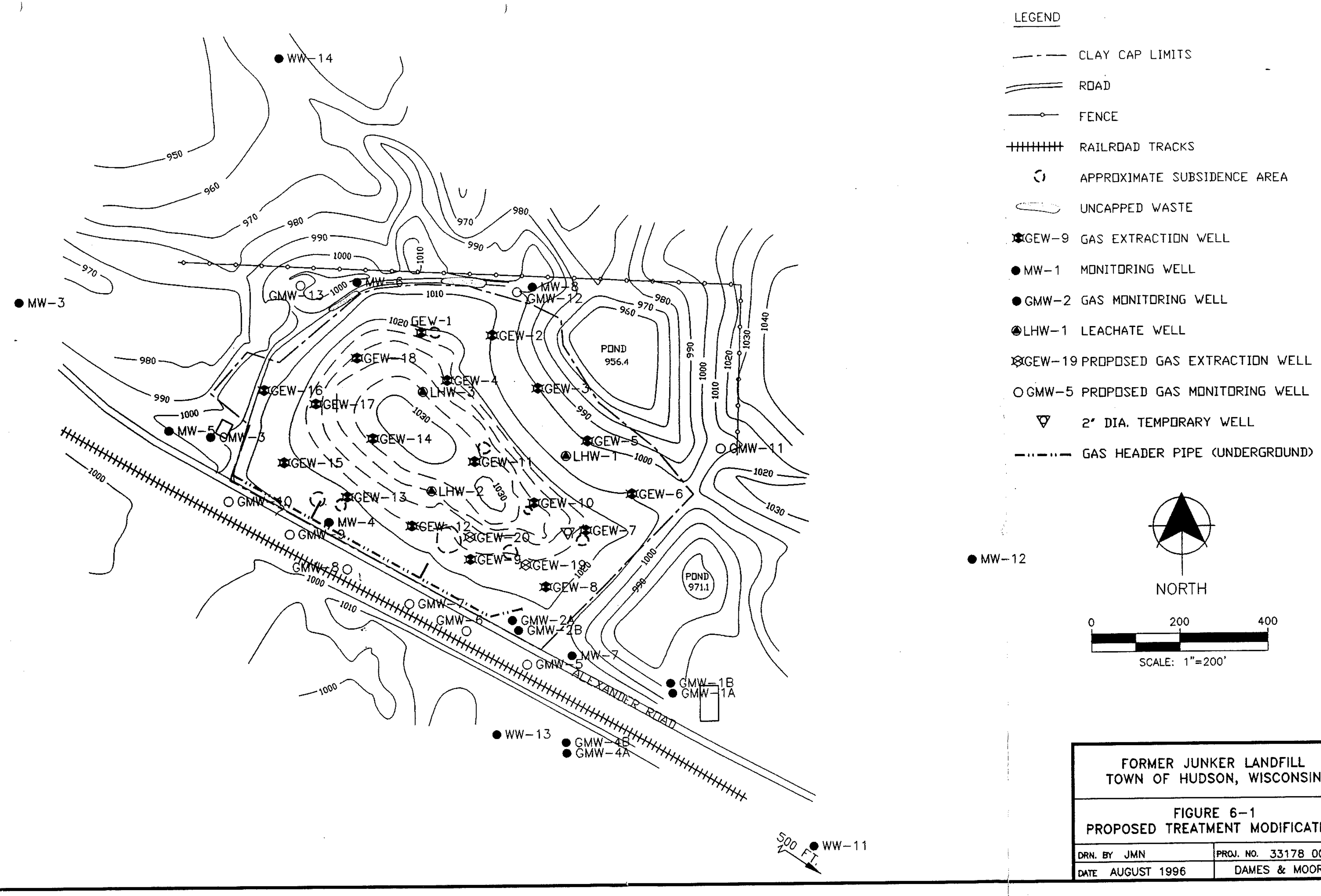
A site inspection at the landfill will also be performed. If any seeps are detected, samples will be collected for determination of chemical makeup, including VOCs, SVOCs and RCRA metals. In addition, each sample will be monitored on site for temperature, pH and conductivity. A monthly site inspection for seeps will be incorporated into the Operation and Maintenance Plan. If seeps are contaminated with VOCs, SVOCs or metals, pan or suction lysimeters will be installed as part of the design project.

6.6 ACCESS AGREEMENTS

The Landfill Remediation Trust was unable to get voluntary access to the JSL site from the current site owner. A lawsuit was filed in St. Croix County Circuit Court on August 16, 1996 in an attempt to obtain a court order authorizing necessary access to the landfill site. This court order also requests that a deed restriction is placed on said property to prevent future development of the property that would damage or interfere with the landfill cap or engineering controls at the site.

Access agreements have been obtained from six of the twelve owners of private property that are currently on the routine monitoring route for SEH personnel. A copy of these agreements and the court order was forwarded to the WDNR on August 14, 1996. Verbal permission for property access was obtained from the other six residents.

Future access agreements will include obtaining a railroad right of way from Union Pacific Railroad, and a highway right of way from the Town of Hudson. Other property owners who have not been identified, and who will require access, will be contacted and a formal access agreement developed, if possible.



FORMER JUNKER LANDFILL
TOWN OF HUDSON, WISCONSIN

FIGURE 6-1
PROPOSED TREATMENT MODIFICATION

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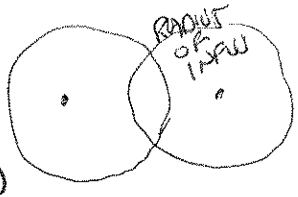
FILE: C:\DWG\JUNKER\FIG6-1

7.0 INVESTIGATIVE WORK PLAN

The items detailed under the investigative work plan will be discussed according to the chronological order of execution, as detailed on the Project Schedule shown in Figure 4-1. Dames & Moore has developed a Health and Safety Plan to cover the items discussed in this section. A photocopy of the Health and Safety Plan is located in Appendix D.



7.1 RADIUS OF INFLUENCE OF GAS EXTRACTION WELLS

not really

To identify the optimum location for additional extraction wells, the radius of influence of each existing well will be determined. The initial determination will occur prior to the installation of additional monitoring probes. All extraction wells will be turned off and each well tested. A series of measurements will be used to determine static and differential pressures. Gas extraction systems are designed to have overlapping radii of influence. Thus, if vacuum is detected at an adjacent well, sufficient overlap is present. This work is scheduled to begin the week ending September 6, 1996. The information gathered from this study will be used in conjunction with VOC analyses to determine the optimum location for additional gas extraction wells. Vacuum measurements in the shallow gas vents installed at the time of cap installation may also be used to check radii.

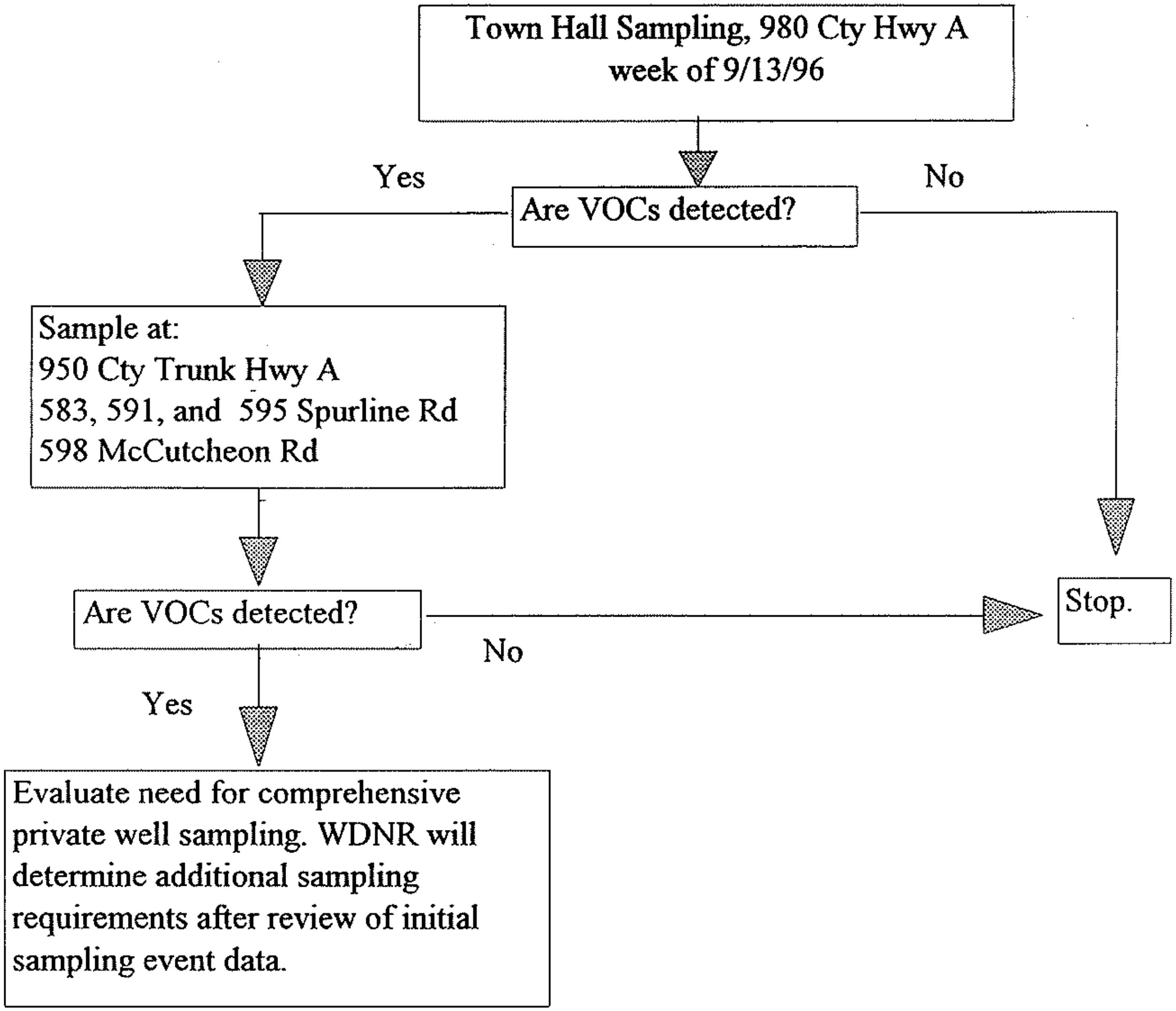
We expect that arrangements will be occurring wk of 3/9

7.2 GROUNDWATER VOC SAMPLING

A groundwater sampling event during February 1995 at the Town Hall in Hudson Township, at 980 County Road A, resulted in a detection of VOCs. This sampling event will be repeated the week ending September 20, 1996 to determine if VOCs are still present. Approximately two weeks turnaround is required to obtain results of the groundwater analyses. If VOCs are detected in the Town Hall well, additional groundwater sampling will be conducted at downgradient wells as required by the Scope of Work. These potential wells are outside the current boundary of the SWCA. The wells proposed for sampling if VOCs are detected in the Town Hall well sample are 950 County Trunk Highway A, 583 Spurline Court, 591 Spurline Court, 595 Spurline Court and 598 McCutcheon Road. If these well samples yield detections of VOCs, additional wells will be sampled, as detailed in Figure 7-1.

Figure 7-1: Decision Tree for Groundwater Sampling

Junker Sanitary Landfill Project



will include
in LTM

All samples will be collected from sampling taps located near the pressure tank and always prior to any treatment device. Prior to collection of the sample, water will be flushed out of the sampling tap for ten minutes. The samples will be sent to Northern Lake Service, Inc., for VOC analysis. Dames & Moore has a Standard Operating Procedure (SOP) for collecting samples for volatile organic analysis (SOP 440) and sampling from private wells (SOP 330A). The SOPs are located in Appendix E. Additional groundwater sampling is planned for homes within SWCA Sections 13, 14, 15, 22, 23, and 24 selected for installation of GAC filters. Details of that sampling are discussed in Subsection 7.10.

7.3 VOC MEASUREMENTS AT GAS EXTRACTION WELLS AND GAS PROBES

Three sampling events for landfill gases are planned. The initial sampling of landfill gas will be conducted on the gas extraction wells. The second and third sampling events will involve the monitoring probes located adjacent to the landfill property. The first sampling event is scheduled for the week ending September 20, 1996 and will include only the gas extraction wells. Samples will be collected in Tedlar bags and analyzed onsite using a field gas chromatograph (GC). The field GC is capable of analyzing all compounds with ionization potentials less than 11 mV (see SOP 260). The standard calibration gas used with the field GC will contain those compounds previously identified in the blower gas and those identified in the ROD as compounds of concern. This list is provided in Section 9.0, along with the ionization potentials of these compounds. Calibration gases for the field GC will be obtained through Scott Specialty Gases. Of the seventeen gas extraction well samples, four samples will be collected using SUMMA vacuum canisters and sent to Quanterra Environmental Services in the City of Industry, California for confirmation of GC results. These four samples will include a low VOC well, two high VOC wells and a field blank. The location of the SUMMA canister samples will be determined by the results of the field GC analyses. The SOPs for collecting gas samples with Tedlar bags (SOP 500) and with SUMMA canisters (SOP 501) are located in Appendix E. The second sampling event is scheduled for the week ending October 18, 1996, but is dependent on the installation of the additional gas probes. The third sampling event is scheduled for the week ending December 13, 1996. The second and third sampling events will require SUMMA vacuum canisters for all gas monitoring probes outside the landfill. The samples will be sent to Quanterra for analysis. Quarterly gas monitoring of the gas probes will continue as long as VOCs are detected and will be incorporated into the O&M Plan for the site.

So the 2nd + 3rd do not
include the GEWs

7.4 EVALUATE SEEPS

do this earlier.

A visual inspection of the site is planned for the week ending September 20, 1996. At that time, a thorough seep investigation will occur. Any seeps will be sampled in accordance with SOP 340, surface water sampling, shown in Appendix E. At the time of collection, temperature, pH and conductivity will be measured, according to SOP #240, 220 and 230, respectively. Seep samples will be analyzed for VOCs and RCRA metals. A monthly investigation of the site for seeps and chemical analysis of any seeps, will be incorporated into the O&M plan for the site. If seep analysis shows the presence of VOCs or metals, pan or suction lysimeters will be installed in the seep areas. The lysimeter design will be incorporated into the design package for this site.

(What happened to semi VOCs - see pg 6-4

7.5 DEFINE THE EXTENT OF CLAY TO THE SOUTH

One recommendation for the design of the remedial system at the JSL site was to extend the clay cap to the south in the area of the gas header pipe. It is necessary to first determine the area where the clay cap is deficient. A soil probe, the JMC "Backsaver," will traverse the area around the header pipe, shown on Figure 6-1, to determine whether clay is present. Approximately 200 soil probe borings are planned within 3-6 feet of the as-built header pipe location. The probe will be used to determine both vertical and lateral extent of the clay layer. The "Backsaver" soil probe is advanced manually and provides a clean core sample for analysis. SOP 320A for the JMC "Backsaver" soil probe is included in Appendix E. This investigation is planned for the week ending September 20, 1996. If it is determined that there are areas deficient in compacted clay, replacement of this layer will be incorporated into the design plans for the site.

7.6 SURVEY OF LANDFILL AND SURROUNDING AREA

A survey of the landfill property and an area extending in a 400-foot radius around the property by BRW, Inc., of Minneapolis, MN will establish current topography. The Wisconsin State Plane coordinate grid system will be used. In addition, the existing gas extraction wells will be added to the survey. The survey is necessary to determine the extent of the settlement areas noted on Figure 5-1 and is scheduled for the week ending August 30, 1996. Measurement of the subsidence areas will be used in designing the plan for excavating waste north of the landfill. This item is discussed further in Subsection 7.8.

7.7 INSTALL GAS PROBES AT THE NORTH, EAST AND SOUTH BOUNDARIES

The Scope of Work requires installation of gas probes along the southern boundary of the landfill to more accurately assess the gas migration pattern to the south-southwest. However, no gas probes exist on the north and east boundaries of the landfill. It is not known whether gas is migrating in either of these directions. Monitoring wells MW-6, MW-8 and MW-12 will be monitored for landfill gases during the VOC determination at the existing gas extraction wells and probes. If this evaluation indicates the presence of landfill gases to the north or east, installation of gas probes along the north and east boundaries will be implemented. The approximate location of the proposed gas probes to the north, east and south of the landfill is shown in Figure 6-1. Every other one of the gas monitoring probes along the southern boundary will be nested, with screens set at key depths. The possible depths of the screens include the top of bedrock, below the depth where VOCs and/or methane gas are originally detected during drilling, and above or below the water table. Additional nested gas probes may be installed within the landfill. The nested probes will be used to determine where landfill gases are being produced (deep or shallow) and assist with understanding the effectiveness of the current gas extraction system to remove the source landfill gases. A description of the gas monitoring probe installation is provided in SOP 110A in Appendix E. The target for gas probe installation is the week ending October 4, 1996. These gas probes will also be used to maintain the balance of the gas extraction system, and will be incorporated into the Operation and Maintenance Plan.

for methane too - may be hard to distinguish VOCs in gas vs VOCs coming off water.

(5,6,7,8,
(9,10

Screened at highest methane detection

7.8 EXCAVATE REFUSE NORTH OF LANDFILL

Initial plans indicated that the clay cap would be extended north of the landfill to cover the two areas identified as containing uncapped waste. Instead, it is recommended that these two areas be excavated and the refuse used as general fill for the subsidence areas noted on Figure 5-1. The volume of the subsidence areas will be determined from survey data. Plans for filling and regrading the subsidence areas will be conducted. The target date for beginning the excavation of refuse is the week ending October 25, 1996. It will also be necessary to backfill the areas north of the landfill and restore the access road.

7.9 LEACHATE MOUND AND PUMP TEST

The Wenck RI presented data on the existence of leachate in several of the gas extraction wells in the southeast quadrant of the landfill. This data was extrapolated to show the existence of a leachate mound in this area with as much as 24 feet of saturated waste within its bounds. This mound has been implicated as one of the potential sources of landfill gas southeast of the site. Part of the planned remedial responses include sealing potential sources of infiltration to prevent leachate buildup. A minimum of two leachate wells will be drilled in the area of the leachate source. One well will be a pumping well and the other a leachate monitoring well. A leachate pumping test is planned for early Spring 1997 to determine the true dimensions and the hydraulic characteristics of the leachate mound. (why so late - do this in Oct/Nov.

The proposed gas extraction wells to be installed in Spring 1997 to capture landfill gas migration southeast of the site will likely be located within the area of the greatest mapped saturated leachate thickness. A special well screen to allow gas flow but also optimize leachate flow without sediment buildup will be installed in the subject gas well boring. This well screen will be selected to be compatible with the backfill material specified in NR 504.08 (2)(e), Wisconsin Administrative Code. If the mound is present as indicated by Wenck, the proposed gas extraction wells will be fitted with pumps and valve assemblies capable of extracting both gas and leachate extraction.[1] Due to the time constraints involved, the design documents for the gas extraction wells will assume that the leachate mound exists, and include the leachate pumps and valve assemblies as part of the design. If it is found that the leachate mound does not exist, these items will be removed from the bid documents.

In the event that methane migration conditions do not improve, or groundwater shows further degradation, the WDNR will require leachate pumping from gas extraction wells located within the area of the mound.

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[1] The existing gas extraction wells in the area of the leachate mound are currently being retrofitted by SEH with new valve assemblies. These new valve assemblies will be capable of extracting both gas and leachate, in the event leachate is necessarily withdrawn.

7.10 INSTALL GAC FILTERS

The GAC filter installation will be offered to the Norflex Facility, and all private residences and businesses located in Sections 13, 14, 15, 22, 23, and 24 that are also within the Special Well Construction Area (SWCA) of St. Croix County. GAC filters are currently being used in other areas of Hudson Township. Dames & Moore has obtained the design information through DCOMM for the approved GAC system. Dames & Moore has begun work to identify a supplier that could provide the same GAC system. Once a supplier is identified, the approval application will be filed with DCOMM. In addition to the application, DCOMM requires sales brochures (if proposed), a labeling system, and an instruction and maintenance manual. Dames & Moore is seeking approval of two different sized systems. The required GAC system in each home will be determined by the results of VOC testing of the groundwater. The current approval of the GAC system is detailed in Appendix C.

Groundwater within the subject SWCA must also be sampled to determine whether there are any residences that require pretreatment for removal of substances that reduce the efficiency of GAC systems. Every home within SWCA Sections 13, 14, 15, 22, 23, or 24 will be sampled for analysis of VOCs, total iron, iron bacteria and coliform bacteria. In addition, every tenth home will be sampled for analysis of hardness, alkalinity and total dissolved solids. This sampling event is scheduled in conjunction with the sampling at the Town Hall during the week ending September 20, 1996. The analytical data, detailing organic and inorganic contaminants, will be reviewed by Bob Schaeffer, WDNR Water Supply. Mr. Schaeffer is responsible for granting approval of the GAC systems and any pretreatment devices. The description of the plan for installing the filters and the location of wells within the SWCA are located in Appendix C. The installation of GAC filters is scheduled to begin October 18, 1996 with completion by January 1, 1997.

Section VIII.E.2 of the Consent Decree requires Settling Defendants to offer to install and maintain, at their expense, point-of-entry granulated activated carbon filters, that have been approved by WDNR and WDILHR in each home and business within the Special Well Construction Zone that is located in Sections 13, 14, 15, 22, 23 or 24 or the Town of Hudson, T29N, R19W, St. Croix County, Wisconsin, and in the Norflex Inc. Facility at 720 Norflex Drive in the Town of Hudson.

To meet his requirement the Settling Defendants propose to implement a program to ensure this offer is made to each home in the area and any new construction that would be eligible for this offer. It is anticipated that the Settling Defendants and/or their agents will request to meet informally with Town of Hudson Board members to determine the best program to meet this requirement.

One of the options currently being considered by the Settling Defendants is to propose to the Town a program whereby as part of the building inspection process or as part of the issuance of an occupancy permit the Town official responsible for such inspection or permit would contact the project manager for the Trust and inform him of the new residence or business and then the Trust would send a standard form to the new owner outlining the offer under the terms as set forth in the consent decree. The owner of the residence or business would then have to indicate on the form whether or not he/she wanted a GAC and then return that form to the project manager. The filter would then be installed in accordance with the protocol that is approved by WDNR and DILHR for GAC filter installation.

Will the well owners know the sample results

As indicated, an informal discussion will need to be held with the Town to see if such a program is acceptable and also to incorporate any necessary additions into their local building/permitting ordinances or procedures as may be necessary.

It should be noted that such a program would not address any issues with respect to subsequent purchasers of the residences or businesses in the SWCA for which Settling Defendants are required to offer GAC filters. To the extent that a home or business that has a filter is sold, any notices about change out of filters or maintenance, etc., will be conveyed to the new owner.

However, if a current owner declines the offer for a filter and subsequently sells the home it may be impossible for the Settling Defendants to know that there is a new owner and that another offer of a GAC filter should be made. There would be no Town official involved in a standard home sale. Therefore, the Settling Defendants propose discussing this issue with Town Board representatives informally and also potentially with local realtors, or sending a mailing to local realtors asking that they voluntarily notify Settling Defendants when a house in the SWCA defined above that does not contain a filter is sold.

Another alternative may be to see if local realtors would be receptive to keeping application forms for filters and providing them to prospective purchasers prior to closing.

The Settling Defendants will have a plan in place and submitted for WDNR approval prior to the commencement of the GAC filter installation which plan will include a standard form to send to all affected homes and businesses initially, as well as a program or plan for how to deal with new construction and transfers of property. Given the various issues involved and the necessity for Town Board input, and possibly even realtor input, a final program could not be established by the date this submittal was due.

8.0 INVESTIGATIVE WASTE PLAN

8.1 MANAGEMENT OF INVESTIGATION-DERIVED WASTES

The disposal of materials generated during site remediation will be based on the following rationale:

- The materials generated are intrinsic elements of the site and should not be separated from the site; and
- Remedial action will not contribute to further environmental degradation, nor should a threat to public health or safety be presented.

At the former Junker Sanitary Landfill, the bulk of construction derived wastes will consist of previously disposed landfill refuse. It is anticipated that the amount of soil waste derived from drilling of gas extraction wells, gas monitoring probes and disturbance of the existing cap during remedial construction will be negligible because it will be utilized for regrading subsidence areas. Management and disposal of these waste materials are defined in the following sections.

8.2 SOIL

Soil cutting wastes generated from the advancement of soil borings near the gas header pipe will be collected in drums at each boring location. A visual inspection will be performed to determine the condition of the cuttings. If material is not contaminated, it will be deposited on site. If material appears contaminated (i.e. apparent sludges or strong odors), a sample will be collected and analyzed for VOCs and SVOCs, following USEPA Method 8021 and 8270, respectively. Based on the results of this analysis, further testing for hazardous waste classification by TCLP analysis may be needed. If TCLP testing is performed, the waste will remain drummed until the TCLP results are available, and proper waste disposition procedures will be followed.

8.3 DRILL CUTTINGS

Drill cuttings, if derived, will be collected in drums. Drums will be filled no less than 10 inches from the top, to prevent rupture in freezing temperatures. The contents of each drum will be clearly labeled, and each boring will be numbered and invoiced. As with the soil cuttings, no drums will contain mud from multiple borings. Drill cuttings will be visually inspected and/or field screened. If visual inspection reveals that the drill cuttings are clean, this material will be deposited on site. If visual inspection reveals the cuttings may potentially be hazardous waste, then these cuttings will be segregated and samples analyzed for VOCs and SVOCs, following methods 8021 and 8270, respectively. Based on the results of these analyses, a suitable disposal method will be selected.

8.4 EXCAVATED REFUSE

Most of the construction derived waste materials will be refuse excavated as a result of remedial activities. The area north of the landfill that has been identified as containing uncapped waste will be excavated and used as backfill for the subsidence areas. Additional refuse will be excavated during the installation of additional gas extraction wells and trenches for laterals and header pipe and condensate conveyance lines.

During excavation, waste materials will be visually inspected. If materials are encountered that appear potentially hazardous, it will be segregated and stockpiled on plastic. Samples of this material will be characterized by TCLP analysis. If the results of this analysis yield hazardous concentrations, the material will be handled and properly disposed off-site at a licensed facility.

Materials that are not visually suspect, and materials stockpiled as potential hazardous wastes, but yield TCLP concentrations below threshold limits, will be disposed on-site in the subsidence areas. These areas will have the compacted clay cap replaced. If necessary, an additional cell will be located on the site, filled with the excavated waste, graded, and covered with a compacted clay cap.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The purpose of the sampling program at the JSL site is threefold: to characterize the nature of groundwater within SWCA Sections 13, 14, 15, 22, 23, and 24 of St. Croix County; to characterize the nature of the leachate seeps on the landfill property; and to characterize the nature of VOC components in the landfill gas. The groundwater sampling data will be used to determine what size GAC filter system is necessary to adequately treat the water and whether inorganic substances are present that require pretreatment. The data obtained from leachate seeps (if any are identified) will be used to assess the risk of ecological impact. Finally, the VOC characterization of the landfill gases will be used to determine if migration patterns of VOC gases can be linked to leachate production within the landfill.

Table 9-1 shows the proposed analytical program for the JSL site. For each sample matrix, Table 9-1 provides the field and laboratory parameters; the number of sample points and sampling rounds; and the total number of investigative samples, field duplicates, field blanks and trip blanks to be collected. For each sample matrix and analysis, the appropriate sample bottles, preservatives and holding times are indicated in Table 9-2. The proposed analyte lists and their respective Method Detection Limits (MDLs) are provided in Tables 9-3 (gaseous) and 9-4 (aqueous).

This discussion is separated into two sections, the landfill investigation activities, and groundwater investigation activities. Following WDNR request, this information includes some aspects of the Quality Assurance Project Plan and Monitoring Program Plan. Other quality assurance procedures and protocol not presented in this document that are part of the quality assurance program for this investigation (i.e., chain-of-custody, calibration checks, etc.) are contained within the discussion on quality assurance procedures in the Field Sampling and Analysis Plan (Appendix 7) of the original Wenck RI Work Plan.

9.1 LANDFILL INVESTIGATION

Several investigative activities are planned for the Junker Landfill site. Among them are investigating the extent of VOCs in the landfill gas on the landfill and surrounding properties; leachate seep identification and analysis; determining the extent of the clay layer to the south;

Table 9-1
Remedial Design/Remedial Action Analytical Program

field or lab?

all GEW probes

Sample Matrix	Field Parameters	Laboratory Parameters	Sampling Points (a)	Sampling Rounds (b)	Total Samples	Field Duplicates	Field Blank	Trip Blank	Matrix Total (a)
Gas	GC Screening	VOCs VOCs	2 22	1 2	2 44	1 4	1 2	0 0	4 50
Surface Water^c	pH,temp, conductivity	VOCs/SVOCs Metals	4 4	1 1	4 4	1 1	1 1	1 1	7 7
Groundwater (d)	pH, temp, conductivity	VOCs Total Fe, Fe Bacteria, Coliform Bacteria Hardness, Alkalinity, Total Dissolved Solids	10 87 9	1 1 1	10 87 9	1 8 1	1 1 1	1 1 1	13 97 12

Should be 87.

(a) Matrix spike/matrix spike duplicate (MS/MSD) samples are required for aqueous organic analysis. Samples designated for MS/MSD analysis will be collected with extra volumes at a frequency of one per group of 10 or fewer investigative samples. Triple the normal sample volumes will be collected for VOCs and double the normal volumes will be collected for extractable organics.

(b) The first round of groundwater analytical data will be used to assess the VOC contamination at the Village Hall and provide baseline VOC information at the new residential wells. During the same sampling event, total iron, iron bacteria and coliform bacteria will be analyzed on all samples. Total hardness, alkalinity and total suspended solids will analyzed on every tenth sample. The inorganic parameters will be used to assess the pretreatment requirements, if any, prior to granular activated carbon filtration. The first round of gas analytical data will be used to assess the VOC levels in landfill gases at the gas extraction wells. The following rounds of gas analytical data will be used to assess the seasonal VOC levels in the landfill gases that have migrated off-site, as detected in the gas monitoring probes.

what about "every home w/in JWCA"

(c) The actual number of surface water samples will depend on the number of seeps identified during the landfill inspection.

(d) One trip blank will be submitted for each cooler shipped to the laboratory containing water samples for VOC analyses.

GC-lowest
mdl's -not
ascertain

Table 9-2
Sample Bottles, Preservatives, and Holding Times

Matrix	Analysis	Method	Bottles	Preservatives	Holding Time
Private Groundwater	VOCs	EPA 8021	2 X 40 ml glass vials	1:1 HCL to pH<2; Cool 4°C	14 days from collection
	Iron Bacteria, Total Coliform	1130, 301.1	1 X 100 ml polyethylene bottle	None	Analyzed within 48 hours from date of collection
	Total Hardness, Total Iron	ICP	1 X 1L polyethylene bottle	HNO3 to pH<2; Cool 4°C (unfiltered samples)	6 months
	TDS	EPA 160.1	1 X 50ml	Cool 4°C	Analyze within 7 days from date of collection
	Total Alkalinity	EPA 310.2	1 X 50ml	Cool 4°C	Analyze within 14 days from date of collection
	pH	Field	1 X 4 oz. glass	NA	Analyze immediately
	Temperature	Field	1 X 4 oz. glass	NA	Analyze immediately
	Specific Cond.	Field	1 X 4 oz. glass	NA	Analyze immediately
Surface Water (in addition to analyses listed above)	SVOCs	EPA 8270	2 X 1L amber glass bottles	Cool 4°C	7 days to extraction: 40 days analysis
	Metals (dissolved)	ICP	1 X 1L polyethylene bottle	HNO3 to pH<2; Cool 4°C (filtered samples)	6 months
Landfill Gas	VOCs	EPA TO-14	6L Stainless Steel Canister	NA	Analyze within 14 days from date of collection

Note: Surface water sampling represents collection, if present, of leachate surface seeps on the landfill site.
Sample preservatives will be added immediately upon sample collection for all analyses except metals.
Metal samples to be filtered will be done immediately following collection and before adding preservative for dissolved metals.

Table 9-3
Gaseous Analytes and Corresponding Method Detection Limits (MDLs)
Junker Sanitary Landfill Project

Compound	Report Limits (ppbv)	MDL (ppbv)	Compound	Report Limits (ppbv)	MDL (ppbv)
Freon 12	2.0	0.30	Styrene	2.0	0.52
Chloromethane	4.0	0.70	Bromoform	2.0	0.26
Freon 114	2.0	0.23	1,1,2,2-Tetrachloroethane	2.0	0.42
Vinyl Chloride	2.0	0.26	Benzyl Chloride	10.0	3.2
Bromomethane	2.0	0.98	4-Ethyltoluene	2.0	0.34
Chloroethane	4.0	1.7	1,3,5-Trimethylbenzene	2.0	0.31
Trichlorofluoromethane	2.0	0.19	1,2,4-Trimethylbenzene	20.0	0.26
1,1-Dichloroethene	2.0	0.77	1,3-Dichlorobenzene	2.0	0.39
Carbon Disulfide	10.0	0.72	1,4-Dichlorobenzene	2.0	0.60
Freon 113	2.0	0.20	1,2-Dichlorobenzene	2.0	0.51
Acetone	10.0	1.1	1,2,4-Trichlorobenzene	20.0	9.3
Methylene Chloride	2.0	0.68	Hexachlorobutadiene	2.0	0.61
trans-1,2-dichloroethene	2.0	0.88	cis-1,3-Dichloropropene	2.0	0.52
1,1-Dichloroethane	2.0	0.39	4-Methyl-2-pentanone	10.0	2.2
Vinyl Acetate	10.0	0.45	Toluene	2.0	0.31
cis-1,2-Dichloroethene	2.0	0.95	trans-1,3-Dichloropropene	2.0	0.52
2-Butanone (MEK)	10.0	2.4	1,1,2-Trichloroethane	2.0	0.66
Chloroform	2.0	0.26	Tetrachloroethene	2.0	0.38
1,1,1-Trichloroethane	2.0	0.30	2-Hexanone	30.0	14
Carbon Tetrachloride	2.0	0.32	Proylene	10	(a)
Benzene	2.0	0.61	1,3- Butadiene	10	(a)
1,2-Dichloroethane	2.0	0.84	2-Propanol	20	(a)
Trichloroethene	2.0	0.25	Chloropropene	10	(a)
1,2-Dichloropropane	2.0	0.47	Hexane	10	(a)
Bromodichloromethane	2.0	0.21	Tetrahydrofuran	10	(a)
Dibromochloromethane	2.0	0.12	Cyclohexane	10	(a)
1,2-Dibromoethane	2.0	0.29	1,4-Dioxane	10	(a)
Chlorobenzene	2.0	0.26	Ethanol (b)	---	---
Ethylbenzene	2.0	0.25	Methy tert-Butly Ether	10	(a)
Total Xylenes	2.0	0.36	Heptane	10	(a)

Notes: (a) these compounds are Add-ons for Quanterra. Method Detection Limits can be established if detection limits are below those listed for the Report Limits.
(b) Ethanol not analyzed by Ouanterra; 5/96 blower sample yielded ND

Table 9-4
Laboratory Method Detection Limits (MDLs) and Lower Quativative Limits (LOQ) for Groundwater and Surface Water Samples

VOCs by Method EPA 8021

Analyte	MDL (μg/L)	LOQ (μg/L)	Analyte	MDL (μg/L)	LOQ (μg/L)
Dichlorodiflouromethane	0.151	0.522	Chlorobenzene	0.180	0.622
Chloromethane	0.188	0.648	1,1,1,2-Tetrachloroethane	0.152	0.525
Vinyl Choloride	0.110	0.338	Ethylbenzene	0.366	1.26
Bromomethane	0.155	0.534	m\p-xylene	0.636	2.19
Chloroethane	0.138	0.477	ortho-xylene/styrene	0.690	2.38
Trichlorofluoromethane	0.402	1.39	Isorprpylbenzene	0.423	1.46
1,1-Dichloroethene	0.204	0.704	Bromoform	0.120	0.415
Methylene Chloride	0.448	1.54	1,1,2,2-Tetrachloroethane	0.271	0.935
trans-1,2-Dichloroethene	0.394	1.36	1,2,3-Trichloropropane	0.271	0.935
1,1-Dichloroethane	0.127	0.439	n-Propoylbenzene	0.404	1.40
2,2-Dichloropropane	0.292	1.01	Bromobenzene	0.333	1.15
cis-1,2-Dichloroethene	0.303	1.05	1,3,5-Trimethylbenzene	0.675	2.33
Chloroform	0.132	0.455	2-Chlorotoluene	0.352	1.21
Bromochloromethane	0.128	0.442	4-Chlorotoluene	0.675	2.33
1,1,1-Trichloroethane	0.138	0.474	tert-Butylbenzene	0.410	1.41
1,1-Dichloropropene	0.358	1.23	1,2,4-Trimethylbenzene	0.400	1.38
Carbon Tetrachloride	0.279	0.961	sec-Butylbenzene	0.418	1.44
1,2-Dichloroethane	0.128	0.443	p-Isopropyltoluene	0.422	1.46
Benzene	0.312	1.07	1,3-Dichlorobenzene	0.293	1.01
Trichloroethene	0.288	0.993	1,4-Dichlorobenzene	0.312	1.07
1,2-Dichloropropane	0.141	0.487	n-Butylbenzene	0.678	2.34
Bromodichloromethane	0.111	0.386	1,2-Dichlorobenzene	0.678	2.34
Dibromomethane	0.096	0.331	DBCP	0.459	1.58
cis-1,3-Dichloropropene	0.134	0.461	1,2,4-Trichlorobenzene	0.337	1.30
Toluene	0.335	1.15	Hexachlorobutadiene	0.288	0.960
trans-1,3-Dichloropropene	0.154	0.533	Naphthalene	0.574	1.98
1,1,2-Trichloroethane	0.136	0.468	1,2,3-Trichlorobenzene	0.329	1.10
1,3-Dichloropropane	0.320	1.11	Tert-butyl-methylether	0.394	1.36
Tetrachloroethene	0.307	1.06	Isopropylether	0.311	1.07
Dibromochloromethane	0.122	0.387	Tetrahydrofuran (a)	1.3	4.4
EDB	0.094	0.325	Methyl Ethyl Ketone((MEK)	1.0	(b)

Notes: (a) MDL and LOQ for Tetrahydrofuran based on 3400 GC
(b) MDL for MEK based on lowest calibration standard; MEK will only be analyzed in surface water (seep) samples

SVOCs by EPA Method 8270

Analyte	MDL (µg/L)	LOQ (µg/L)	Analyte	MDL (µg/L)	LOQ (µg/L)
Acenapthene	0.71	2.3	2,4-Dinitrotoluene	0.71	2.3
Acenaphthylene	0.71	2.3	2,6-Dinitrotoluene	0.74	2.5
4-Aminobiphenyl	0.67	2.3	Diphenylamine	0.70	2.3
Aniline	0.60	2.0	1,2-Diphenylhydrazine	1.0	3.3
Anthracene	0.60	1.9	Fluoranthene	0.60	1.9
Benzidine	2.4	8.4	Fluorene	0.61	1.9
Benzo [a] anthracene	0.57	1.8	Hexachlorobenzene	0.60	2.0
Benzo [a] pyrene	0.64	2.0	Hexachlorobutadiene	0.54	1.8
Benzo [b] fluoranthene	1.9	6.3	Hexachlorocyclopentadiene	1.2	4.0
Benzo [g,h,i] perylene	0.78	2.5	Hexachloroethane	0.43	1.4
Benzo [k] fluoranthene	0.60	1.9	Indeo [1,2,3-cd] pyrene	0.78	2.5
Benzoic Acid	2.8	9.3	Isophorone	0.72	2.4
Benzyl Alcohol	1.2	4.1	2-Methylnaphthalene	0.68	2.3
Bis (2-chloroethyl) ether	0.67	2.2	2-Methylphenol	0.76	2.5
Bis (2-chloroethoxy) methane	0.80	2.7	3 & 4-Methylphenol	1.3	4.3
Bis (2-ethylhexyl) phthalate	0.82	2.6	N-nitroso-di-n-propylamine	0.67	2.2
Bis (2-chloroisopropyl) ether	0.71	2.4	N-nitrosodi-n-butylamine	0.75	2.5
4-Bromophenyl-phenyl ether	0.57	1.9	N-nitrosodimethylamine	0.47	1.6
Butylbenzylphthalate	0.43	1.4	N-nitrosopiperidine	2.0	7.1
2-Chlorophenol	0.65	2.2	N-nitrosodiphenylamine	0.70	2.3
4-Chloro-3-methylphenol	0.70	2.3	Naphthalene	0.88	2.8
1-Chloronaphthalene	0.59	2.0	1-Naphthylamine	0.43	1.4
2-Chloronaphthalene	0.78	2.6	2-Naphthylamine	0.65	2.2
4-Chloroaniline	0.75	2.5	2-Nitroaniline	0.59	2.0
4-Chlorophenyl-phenyl ether	0.65	2.2	3-Nitroaniline	0.75	2.5
Chrysene	0.68	2.2	Nitrobenzene	0.71	2.4
Di-n-butyphthalate	0.74	2.5	2-Nitrophnol	1.3	4.3
Di-n-octylphthalate	0.43	1.4	4-Nitroaniline	0.69	2.3
Dibenzo [a,h] anthracene	0.70	2.2	4-Nitrophenol	0.43	1.4
Dibenzofuran	0.70	2.3	Pentachlorobenzene	0.63	2.1
1,2-Dichlorobenzene	0.45	1.5	Pentachloronitrobenzene	0.62	2.1
1,3-Dichlorobenzene	0.41	1.4	Pentachlorophenol	0.65	2.2
1,4-Dichlorobenzene	0.48	1.6	Phenanthrene	0.63	2.0
3,3'-Dichlorobenzidine	0.90	3.1	Phenol	0.45	1.5
2,4-Dichlorophenol	0.77	2.6	Pyrene	0.59	1.9
2,6-Dichlorophenol	0.76	2.5	Pyridine	3.1	10
Diethylphthalate	0.81	2.7	1,2,4,5-Tetrachlorobenzene	0.69	2.3
2,4-Dimethylphenol	0.51	1.6	2,3,4,6-Tetrachlorophenol	0.58	1.9
Dimethylphthalate	0.77	2.6	1,2,4-Trichlorobenzene	0.61	2.0
p-(Dimethylamino) azobenzene	0.53	1.7	2,4,5-Trichlorophenol	0.74	2.5
4,6-Dinitro-2-methylphenol	0.41	1.3	2,4,6-Trichlorophenol	0.86	2.9
2,4-Dinitrophenol	5.1	18			

Metals by ICP		
Analyte	MDL (μg/L)	LOQ (μg/L)
Arsenic	1.4	4.8
Copper	2.2	7.9
Iron, Total	17	57
Lead	58	210
Manganese	1.3	4.4
Zinc	12	12

installing gas probes along the southern boundary; excavating the uncapped waste north of the landfill; and performing a leachate mound analysis. Of these activities, the VOC analysis of landfill gases and leachate seep analysis require laboratory and/or field analyses. The installation of the gas probes will involve field analyses only.

9.1.1 VOCs in Landfill Gases

a. Sampling Plan

Dames & Moore has proposed collecting gas samples from the seventeen operating gas extraction wells for field gas chromatography (GC) analysis. These samples will be collected in Tedlar bags, as specified in SOP #500 of Appendix E, and then analyzed on the field GC. From the 17 operating gas extraction wells, three additional samples will be collected in SUMMA canisters, as specified in SOP #501 of Appendix E, and analyzed by gas chromatography/mass spectrometry (GC/MS) methods at Quanterra Environmental Services, City of Industry, California. These four samples will include two samples from a well (one sample and one duplicate) identified through field GC analysis to contain "high" levels of VOCs; and one sample from a well that contains "low" levels of VOCs. In addition, a field blank will be prepared with high purity air. This sampling plan is detailed in Table 9-1. The second round of testing will determine VOC levels in the gas monitoring probes on the landfill and surrounding properties. This sampling event will analyze samples from the existing gas monitoring probes on the landfill and surrounding property and the proposed gas probes along the south boundary of the landfill. All gas monitoring probe samples will be collected in SUMMA canisters and analyzed by Quanterra. As detailed in Table 9-1, for every 10 gas samples collected, a duplicate will also be collected. A field blank will be prepared using high purity air. Trip blanks will not be collected for air analyses. A chain-of-custody form will be completed for each sample collected for laboratory analyses.

really only 2 GEWS will be confirmed by lab

if lab confirmation GEWS in rd 2 with Summa Can

b. Field GC Analysis

A Photovac Field GC will be used for the field analysis of landfill gas samples during the first round of testing. The compounds that have been identified as primary components of the landfill gases generated at the JSL site and their corresponding ionization potentials are listed in Table 9-5. The Photovac Field GC is equipped to identify compounds with ionization potentials less than 11eV. Consequently, it is not able to detect the Freon compounds. Calibration gases for the remaining compounds will be ordered from Scott Specialty Gases. Every time the instrument is turned off and then turned on again, a calibration must be performed. The operating and calibration procedures are detailed in SOP #260, located in Appendix E.

c. Analysis by Quanterra

Quanterra Environmental Services will analyze the confirmatory gas samples from the gas extraction wells, and all samples collected from the gas monitoring probes. Quanterra is prepared to identify volatile organic compounds in air samples by GC/MS, using EPA Method TO-14. Table 9-3 provides a list of the TO-14 compounds and their corresponding Report Limits and Method Detection Limits. Quanterra has not established a Method Detection Limit for tetrahydrofuran. The lowest concentration standard used for tetrahydrofuran is 10 ppbv. Thus, this number is also reported as the MDL. The SOPs for determination of VOCs in Air Samples By GC/MS are included in Appendix F.

d. Monitoring Plan

Sampling for VOCs in landfill gases will continue on a quarterly basis in the gas monitoring probes as long as VOCs are detected in probes outside the landfill. Monitoring for VOCs in these gas probes will be added to the current O&M Plan for the site, and submitted with the Design Plans for this site.

9.1.2 Leachate Seep Analysis

a. Sampling Plan

Samples for the seep analysis will be collected in accordance with the Surface Water Sampling SOP, #340, located in Appendix E. In addition, temperature, pH, and specific conductivity will be recorded at the time of sampling. Procedures for these items are located in Appendix E, SOP #240, #220, and #230, respectively. For every 10 seep samples, a duplicate sample will be pulled. A trip blank will be included for every cooler shipped to the lab for analysis, as detailed in Table 9-1. A chain-of-custody form will be completed for each sample collected.

Table 9-5
Ion Potentials(IPs) for the Major Volatile Constituents of Blower Gas

Analyte	IP (eV)	Analyte	IP (eV)
Freon 12	11.75	m,-Xylene	8.44
Vinyl Chloride	9.99	o-Xylene	8.56
Chloroethane	10.97	Styrene	8.40
Freon 11	11.77	1,3,5-Trimethylbenzene	8.39
Methylene Chloride	11.28	1,2,4-Trimethylbenzene	8.27
1,1-Dichloroethane	11.06	Acetone	9.69
cis-1,2-Dichloroethene	9.65	2-Propanol	10.12
Benzene	9.24	2-Butanone MEK	9.54
Trichloroethene	9.45	Hexane	10.18
Toluene	8.82	Tetrahydrofuran	9.45
Tetrachloroethene	9.32	Cylcohexane	9.88
Chlorobenzene	9.07	4-Ethyltoluene	10.0
Ethyl Benzene	8.76	Heptane	9.90

Notes: IP values , except 4-Ethyltoluene, from DHHS (NIOSH) Publication No. 94-116; value for 4-Ethyltoluene from laboratory personnel at Quanterra Environmental Services.
Gases listed on this table include: those listed as yielding the highest sample concentrations from the landfill gas extraction system blower in the August 20, 1996 ROD Final Remedial Action - Junker Landfill, and other gases detected during from the 4/95, 7/95, 10/95, 1/96, or 5/96 blower gas sampling events.

b. Analysis by Northern Lake Service, Inc.

All samples collected during the seep investigation will be analyzed by Northern Lake Service, Inc., or their subcontractor, Wisconsin State Hygiene Lab. Samples will be tested by gas chromatography for VOCs and SVOCs, according to methods 8021 or 8270B/625. Metals analyses will be performed by inductively coupled plasma-atomic emission spectroscopy (ICP).

c. Monitoring Plan

Monitoring for seeps on the landfill property will continue on a monthly basis throughout the project. After project completion, the frequency for monitoring of landfill seeps will be reevaluated.

9.1.3 Gas Probe Installation

a. Sampling Plan & Analysis

Ambient air will be monitored during the installation of gas probes for methane and volatile organic compounds. Methane will be detected with a combustible gas indicator. Organic compounds will be detected with a photoionization detector (PID). Although specific compounds will not be identified with the use of a PID, general information regarding the presence of VOCs will be obtained. Using the combustible gas indicator and the PID during drilling will optimize both the depth of the probe and the placement of the screen.

9.2 GROUNDWATER INVESTIGATION

9.2.1 VOC Analysis of Private Well Samples

a. Sampling Plan

Initial sampling will occur during the week of September 16, 1996 and will include the Town Hall at 980 County Highway A and all residences within SWCA Sections 13, 14, 15, 22, 23, and 24 of St. Croix County. Samples for VOC analyses will be collected in accordance with the Private Well Sampling SOP, #330A, and VOA Sample Collection SOP, #440, located in Appendix E. The system will be purged for a minimum of ten minutes. After purging, the sample will be collected from a sampling port located prior to any treatment device. In addition, temperature, pH, and conductivity will be recorded at the time of sampling, according to SOPs #240, 220, and 230, respectively. For every 10 groundwater samples collected, a duplicate will also be collected. A trip blank will be included for every cooler shipped to the lab for analysis, as detailed in Table 9-1. A chain-of-custody form will be completed for each sample collected.

b. Analysis by Northern Lake Service, Inc.

Samples will be analyzed for the presence of VOCs using EPA Method 8021. The MDLs for the analytes tested are listed in Table 9-4. All samples will be analyzed by Northern Lake Service, Inc.

c. Monitoring Plan

Private well samples will be collected on an annual basis from all private wells within SWCA Sections 13, 14, 15, 22, 23, and 24. In addition, ten homes will be monitored quarterly to determine the average efficiency of the carbon units and assist with determining adequate carbon changeout times.

9.2.2. Inorganic Analysis of Groundwater Samples

a. Sampling Plan

Samples will be collected from all private wells within SWCA Sections 13, 14, 15, 22, 23, and 24, as specified earlier. Samples for inorganic analyses will be collected prior to any treatment device. All homes will have samples collected for coliform bacteria, iron bacteria and total iron. In addition, every tenth home will have samples collected for hardness, alkalinity and total dissolved solids. Sampling frequency, duplicates, trip blanks and field blanks is detailed in Table 9-1. A chain-of-custody form will be completed for each sample collected.

b. Analysis by Northern Lake Service, Inc.

The inorganic analyses performed will include coliform bacteria, iron bacteria, total iron, hardness, alkalinity, total dissolved solids, and field screening of pH, temperature and conductivity. Iron and hardness will be analyzed by ICP. Total alkalinity will be analyzed by titration, EPA Method 310.2. Total dissolved solids will be analyzed according to Filterable Residue EPA Method 160.1. Coliform bacteria and iron bacteria will be analyzed by the Wisconsin State Hygiene Lab. The results of the inorganic analyses will be used to indicate if pretreatment of the water is required before it passes through the GAC system. The MDLs for each analyte are listed in Table 9-4. All samples will be analyzed by Northern Lake Service, Inc., or their subcontractor, Wisconsin State Hygiene Lab.

APPENDICES

APPENDIX A

CURRICULUM VITAE FOR PROJECT PERSONNEL

Curriculum Vitae

GARY L. BOLEY, P.E.

Title	Senior Consultant Principal-In-Charge
Expertise	Energy & Power Project Development Waste-to-Energy/IPP Project Development & Management Environmental Permitting Solid Waste Management Contract/Claims Resolution
Academic Background	University of Pittsburgh, PA -Master of Public Works Administration, 1975 University of Wisconsin -Platteville, WI -BSCE, 1969 Other: Financial Management Development Program, Wharton School Manager of Managers Executive Program, Strategic Management Group Total Quality Executive Program, Philip Crosby College
Registration	Registered Professional Engineer - WI

Experience

A Senior Executive/General Manager with extensive experience in project development, design, and consultation. His accomplishments include the creation, staffing and management of organizations for the development, engineering, permitting and implementation of major projects. He has negotiated complex full service (design, build and operate) contracts that led to the financing of over $1 billion of construction.

Management & Facility Operations

- Organized, staffed and managed the initial start-up and operations of major waste-to-energy co-generation facilities.

- Established and managed a multi-disciplined process and power plant engineering department that included Process, Electrical, I & C, Mechanical, Project Engineering Management, Facility Start-up, Environmental Permitting and R & D units.

- Managed facility design, construction and operating cost estimates, facility predicted performance and establishing guarantees (including facility environmental emissions) for full service product offerings. Major projects developed total approximately $1 billion in construction and $1 billion in total contract operations income.

Facility Environmental Permitting

- Established and managed the environmental and power plant permitting of waste-to-energy facilities, (air, water, solid waste, FAA, and PURPA).

- Managed the environmental and power plant permitting of waste-to-energy facilities in several States, (air, water, solid waste, FAA, and PURPA).

Solid Waste Management, Energy & Technology Studies

- Directed the development and negotiated the agreements to allow the U.S. EPA and Environment Canada to use of the CRRA Mid-Connecticut facility for a detailed performance test program of a "state-of-the-art" municipal waste combustion and emission control system. The research project resulted in a joint corporate and government technology test program for the purpose of establishing Federal New Source Performance Standards for municipal waste combustors.

- Project Director for the evaluation of stack emissions from coal fired and waste-to-energy facilities.

- Project Director for a national survey of waste ash utilization research activities in the U.S.

- Project Director for the evaluation of utilizing wood pallets as fuel for steam and/or electrical generation.

- Provided technical review of advanced fluidized bed combustion technology evaluation for solid and/or waste fuels.

- Managed a multi-million dollar R & D technology licensing and development program as member of the Corporate R & D budget review and funding committee.

- Managed the redevelopment of a European mass burn system for the US market.

- Directed the development of a municipal solid waste ash stabilization process and an ash ferrous and non-ferrous metal recovery system.

- Directed the development of a municipal waste CO control optimization program to minimize the emission of CO and NOX and the control of boiler tube corrosion.

- Provided technical reviews of advanced fluidized bed combustion and emission control system development for municipal and hazardous waste combustion facilities for the European markets.

Contract Negotiation & Claims Resolution

- Lead Negotiator for a Fortune 500 company in the recovery of a multi-million dollar construction delay insurance claims including claims from Lloyds of London and a business interruption insurance claim from a U.S. insurer.

- Provided management and technical services to resolve contract dispute for a major U.S. corporation (client claim in excess of $50 million).

- Negotiated and/or managed power and waste related technology transfer and licensing agreements with international firms located in The Netherlands, Germany, Italy, United Kingdom and Japan.

- Provided management and technical services to resolve contract dispute for a major U.S. corporation (client claim in excess of $50 million).

Project Development

- Developed municipal projects including landfills, waste recycling systems, and major public works facilities.

- Directed the development of a predictive plant performance model and financial performance model for alternate fueled electrical generation facilities.

- Project Director of a US EPA demonstration project to evaluate refuse milling and landfill feasibility and environmental impacts from such practices.

- Directed the application for, and received funding from the U.S. Department of Energy through Argonne National Laboratories. Managed a project development team to evaluate the technical and economic feasibility and environmental impacts of firing municipal biomass in over 20 solid fueled industrial and utility boilers.

- Directed the development, negotiated all necessary agreements and acquired funding for a technology demonstration project to evaluate test firing RDF (Refuse Derived Fuel) in combination with stoker coal in an Industrial Boiler. The project team consisted of a Fortune 500 food processor (boiler owner & operator), a major international boiler supply company, the University of Wisconsin, private contractors and the City of Madison. The project resulted in the first successful commercial sale of RDF to an industry.

- Directed the development, acquired funding and implemented a waste-to-energy project utilizing new technologies to pre-process municipal waste to produce RDF for firing in a utility boiler in combination with pulverized coal, gas and/or oil. The project demonstrated a first-of-a-kind waste process and fuel feed system. This was the first U.S. project to successfully sell RDF to a private utility.

Expert Witness Experience

- Provided expert witness testimony for St. Croix County, Wisconsin with regard to a waste-to-energy facility contract dispute. Services provided included the technical and economic viability and the facility's ability to meet environmental compliance The claim was withdrawn immediately after the deposition in the favor of the client.

- Provided expert witness testimony for a Fortune 100 company on the air and solid waste permitting process with regard to waste-to-energy facility environmental permitting process. The court ruled in the favor of the client.

- Provided expert witness testimony for a confidential Fortune 50 company during a dispute mediation process regarding a large process facility's ability to meet contract guarantees.

Professional History

Senior Consultant, Principal in Charge, Dames & Moore - 1994 to Present
Principal, Environmental Technology Associates - 1992 to 1994
Vice President Engineering, ABB Combustion Engineering, Inc. - 1981 to 1992
Director-Engineering, Wisconsin Solid Waste Recycling Authority - 1980 to 1981
Chief - Special Projects Section, City of Madison, WI - 1969 to 1980

Citizenship

United States

Countries Worked In

United States

Language Proficiency

English

Professional Affiliations

National Society of Professional Engineers
American Society of Civil Engineers
American Public Works Association
International Solid Waste Association

Curriculum Vitae

ROBERT SCOTT GUTHRIE, Ph.D.

Title Senior Geologist

Expertise Soils, Surficial Geology, Site Characterization, Site Investigations, & Litigation Support.

Experience Litigation Support and third-party reviews for a variety of environmental projects. Expert witness testimony on leachate infiltration for a landfill insurance coverage case was cited in an August 1995 decision by the Minnesota Supreme Court.

- Minnesota Refinery; Devised and performed a hydrogeologic site characterization investigation for a portion of the Prairie du Chien aquifer impacted by an extensive pentachlorophenol/petroleum plume; worked with the MPCA project hydrogeologist through all phases of the investigation; collected information on joint orientations, bedding place apertures, springs and solution cavity distribution; set up down well video surveys, performed monitoring well packer tests, and directed geophysical surveys to identify optimal locations for recovery well placement; performed pump tests on five wells to determine hydraulic properties and contaminant concentrations for final system design; reduced data and prepared reports for all phases of the hydrogeologic investigation.
- Minnesota Military Facility, Minnesota; Performed a seismic refraction survey to determine the depths for an irregular bedrock surface for the placement of groundwater interceptor trenches; met the MPCA officials to review workplans and proposed actions, worked with engineers to design an effective system; assisted with calculations for final system design.
- Minnesota Window Manufacturer; Managed a groundwater remediation project consisting of dual carbon modules with a 300 gpm filtration capacity for the removal of pentachlorophenol and associated wood-treating carrier solvents; incorporated safety features into the system and expanded and upgraded the groundwater and river monitoring program to utilize cost-effective innovative technologies and equipment; devised and directed a bioremediation feasibility study and field test that led to the design and installation of a permanent bioremediation system; prepared reports, annual budgets, and met with state and local regulatory officials.
- Minnesota Landfill (State Superfund Site); Directed and reported on treatment system maintenance activities for former industrial landfill site.
- Performed investigations for four landfill sites in Nebraska. Work involved the logging of all cores review and interpretation of hydrogeological information, collection of ground water samples, and testing for anaerobic gases.
- Six field seasons of soil survey mapping in Minnesota and Indiana for four county surveys.

Technical Conferences/ Publications

Wayne, W.J., and Guthrie, R.S,. 1993, Permafrost and Cryodeflation around the Wisconsin ice margin in the mid continent region of North America: Proceedings of the Sixth International Permafrost Conference, Beijing, China, P. 694-699.
Guthrie, R.S., 1992, Cryodefation and the development of the Iowan Erosional Surface east of the Great Plains: Geological Society of America Abstracts with Programs, p. 43.

Guthrie, R.S. and Marston, R.A., 1986. Relict (Late Tertiary) pediments and alluvial fans, Sierra Boca Grande, northwestern Chihuahua: in Geology of South-Central New Mexico, El Paso Geol. Soc. Field Trip, p. 49-51.
Dyer, R., Chavez-Quirarte, R., and Guthrie, R.S., 1987, Mesozic evolution of Trans-Pecos and Chihuahua Tectonic Belt: Geological Society of America Abstracts with Programs, p.650.

Academic Background	Ph.D. in Geology, University of Nebraska, 1990 M.S. in Geology, University of Texas at El Paso, 1987 B.S. in Geology, Winona State University, Minnesota, 1983 B.S. in Agriculture (Soil Science), Purdue University, 1979
	Field Camp, Indiana University Field Station, Cardwell, Montana, 1983 SEPM diagenesis (geochemical) short course; Austin, Texas, 1985 Juneau Icefield Research Program, Juneau, Alaska, Summer 1985 Groundwater Flow Through Fractured Media: Madison, Wisconsin, 1993 Basic Hazardous Materials Workshop: Bloomington, Minnesota, 1993 Using ModFlow and ModPath: Groundwater Modeling with a Graphical Interface: Madison, Wisconsin, 1995 OSHA Training 20 CFR Part 1910-120(e) (40 hours), 1988; eight hour refresher, 1989-1996.
Citizenship	United States
Language Proficiency	English, Spanish
Professional Affiliations	National Ground Water Association AMQUA (American Quaternary Association) Minnesota Association of Professional Soil Scientists Registered Ground Water Professional in the State of Iowa

Curriculum Vitae

JULIE A. HOFFMAN

Title
Staff Engineer

Expertise
Chemical Engineering
Environmental Engineering

Academic Background
M.S., Civil and Environmental Engineering, University of Wisconsin-Madison, 1996.
B.S., Chemical Engineering, Michigan Technological University, 1990.

Experience

Ms. Hoffman has experience with fundamental chemical engineering process design and environmental remediation systems. Prior experience includes quality production of compounded plastic.

- Performed an initial cost estimate for iron reactive barrier technology at a PCE-contaminated groundwater site.
- Determined gas well rates, condensate production, and pressure drops for a landfill gas extraction system.
- Performed O&M services at soil and groundwater remediation sites.
- Performed construction oversight at landfill gas extraction and groundwater remediation sites.
- Investigated effects of groundwater on the performance of granular iron used in the iron reactive barrier technology.
- Authored Air Permit to Operate for polymer compounding unit.
- Managed product improvement and process optimization for a polymer compounding facility.

Professional History
Staff Engineer, Dames & Moore, Madison, WI, 1996 - Present
Associate Process Engineer, Shell Chemical Company, Belpre, OH, 1990 - 1993

Citizenship
United States

Countries Worked In
United States

Language Proficiency
English

Certifications
OSHA 29 CFR 1910.120

Publications
Hoffman, J.A., 1996. Inorganic Chemistry Effects on the Physical Performance of Granular Iron Used in Reactive Barriers. *Master's Thesis*, University of Wisconsin-Madison, January 19, 1996.

Curriculum Vitae

DIRK W. LEEMKUIL, C.P.G., P.G.

TITLE: Project Geologist

EXPERTISE: Soil and Ground-Water Investigation and Evaluation
Geotechnical Investigation and Evaluation
Environmental Investigation and Evaluation
Volatile Organic Remediation
Landfill Engineering, Siting, Closure and Monitoring

EXPERIENCE: Provide geologic and hydrogeologic investigation and evaluation for many varied clients throughout Wisconsin, Minnesota and the upper Midwestern United States. Joined Dames & Moore in 1995

Project Manager/Project Geologist/Principal Investigator

- Conducted geologic and hydrogeologic investigation for evaluation of contaminant extent at numerous leaking underground storage tank sites. Investigation included extensive subsurface exploration, design and installation of ground-water monitoring wells and piezometers, aquifer parameter testing and detailed geologic cross-section mapping. Clients include: UNOCAL, Pillsbury, Ford Motor Company.

- Conducted phase II soil and ground-water characterization and evaluation for property transactions. Investigation includes subsurface exploration, design and installation of ground-water monitoring wells and soil and ground-water analysis. Evaluation of soil and bedrock stratigraphy, hydrogeology, and soil and ground-water geochemistry and contaminants. Projects conducted for Rockwell International and Pillsbury.

- Conducted numerous phase I environmental site assessments per guidelines defined by the American Society for Testing and Materials, Designation: E 1527-94 process. Clients include: Honeywell, Illingworth Corporation, Chrysler Realty Corporation, Medtronic Incorporated and private property owners.

Over ten years experience in mining, engineering and environmental engineering fields.

Senior Project Manager, Northern Environmental Technologies, Incorporated, Milwaukee, Wisconsin and Minneapolis, Minnesota (1990-1995)

- Managed and conducted successful completion of numerous soil and ground-water contaminant investigations.

- Conducted geologic and hydrogeologic characterization studies, underground storage tank site assessments, soil and ground-water exploration and design and installation of soil and ground-water remediation systems including: vapor extraction, ground-water extraction systems and bio-remediation.

- Conducted engineering design and monitoring for landfill closures. Tasks included: surveying, plan and specification preparation, construction inspection and documentation for compliance with Wisconsin Department of Natural Resources regulations.

- Conducted numerous phase I an II environmental site assessments for Northwestern Mutual Life Insurance Company and Norwest Bank, and environmental assessment worksheets and environmental impact statements for various St. Paul and Minneapolis metropolitan area municipalities and the Metropolitan Council Waste-Water Services.

Geologist/Engineering Technician, Larson Engineers, Milwaukee, Wisconsin (1986-1990)

- Conducted geotechnical, geologic and hydrogeologic investigation and assessment for siting sludge disposal landfill for the Milwaukee Metropolitan Sewage District. Tasks included: soil exploration, ground-water monitoring well and piezometer installation, and geotechnical soil sampling in the Pleistocene Oak Creek Formation Stratigraphic Unit of Wisconsin.

- Conducted hydrologic investigation and design of storm-water collection system for new State of Wisconsin prison in Racine County, Wisconsin.

- Member of a construction engineering and inspection team conducting materials and construction inspection for Interstate Highway 94 renovation. Tasks included: surveying, concrete pavement and bridge inspection and documentation for construction standards, and unit cost evaluation and assessment.

Geologist, Anaconda Minerals Company, Denver, Colorado (1981-1984)

- Conducted reconnaissance and site specific exploration for economic deposits of precious metals. Tasks included: sampling, logging and mapping potential deposits in the southwest district of the United States. This included Colorado, Nevada, Arizona, and Southern California.

- Conducted extensive exploration of disseminated gold ore deposit at South Mountain in Summitville, Colorado. Tasks included: chip collection and logging on reverse circulation drilling operation, chip logging, underground mapping and three-dimensional modeling of gold ore deposit in South Mountain.

ACADEMIC BACKGROUND: B.S., Earth Science, University of Wisconsin, River Falls, 1976
Engineering Professional Development, University of Wisconsin, Madison, 1990
Graduate Studies, Hydrogeology, University of Wisconsin, Milwaukee, 1992

CITIZENSHIP: United States

PROFESSIONAL REGISTRATION:
Professional Geologist: Wisconsin, 1995
Professional Geologist: Indiana, 1993
Professional Geologist: Tennessee, 1993
Certified Professional Geologist: AIPG, 1993

PROFESSIONAL AFFILIATIONS:
National Ground-Water Association
Minnesota Ground-Water Association
Wisconsin Ground-Water Association
Wisconsin Chapter AIPG
Minnesota Chapter AIPG

ADDITIONAL TRAINING:
OSHA 40-Hour and Eight-Hour Hazardous Waste Site Workers, Health and Safety Training, 1995
Wisconsin Department of Industry, Labor and Human Relations, Certified Underground Storage Tank Site Assessor, 1996
State of Utah, Certified Ground Water and Soil Sampler, 1994

Curriculum Vitae

MARK S. McCOLLOCH, P.G.

Title

Project Geologist

Expertise

Hydrogeology
Geology
Geological Engineering

Academic Background

M.S., Geological Engineering, University of Missouri - Rolla, Rolla, Missouri, 1991
B.S., Major in Geology, Minor in Mathematics, University of Iowa, Iowa City, Iowa, 1986

Registration

Professional Geologist, State of Wisconsin

Experience

Mr. McColloch is a project level geologist in the Dames & Moore Madison, Wisconsin office. He has conducted numerous site investigations at RCRA, CERCLA, LUST, and solid waste disposal sites. He has interpreted geologic conditions, prepared site investigation reports, evaluated potential remedial alternatives, and assisted with the design and installation of remedial systems. Mr. McColloch is currently serving as Project Manager on several projects which have included the design, installation, and operation of remedial systems.

- Evaluated potential remedial alternatives, and assisted in design and installation of air sparging and bioventing remediation system for a petroleum contaminated site in Janesville, Wisconsin.
- Completed site investigation and evaluation of potential remedial responses for hydraulic oil release inside a building. Subsequently assisted in design and installation of bioventing and product recovery remediation system, La Crosse, Wisconsin.
- Completed site investigation, evaluated remedial alternatives, and coordinated excavation of contaminated soils for two underground storage tank sites in Westfield, Wisconsin.
- Completed a site investigation, evaluated remedial alternatives, and designed and installed a soil vapor extraction system inside a building for a VOC contaminated site in La Crosse, Wisconsin. Subsequently coordinated the collection of data during one year of operation.
- Evaluated remedial alternatives, performed aquifer performance test, and capture zone analysis. Subsequently designed and installed groundwater extraction system, and prepared construction documentation report at a bulk petroleum storage facility, McFarland, Wisconsin.
- Performed Clay Borrow Investigation and prepared documentation report for Holtz & Krause Landfill, Wausau, Wisconsin.
- Supervised the removal of three underground storage tanks, excavation of contaminated soil, and collection of soil samples for closure at a former service station, Lake Geneva, Wisconsin.
- Supervised the removal of two underground storage tanks, performed site assessment, and prepared Tank Closure Documentation report. Subsequently completed a site investigation, and evaluated remedial alternatives at a former service station, Fall River, Wisconsin.

- Coordinated and supervised closure activities for two former hazardous waste storage units, and prepared RCRA closure report at a former metal fabricating facility, DeWitt, Iowa.
- Performed aquifer test and pilot test for design of leachate extraction system at a paper mill sludge landfill, Eau Claire, Wisconsin.
- Supervised drilling activities for a hydrogeologic investigation. Subsequently performed clay borrow investigation, and prepared clay borrow site documentation report for capping a closed City of Madison landfill.
- Supervised of drilling activities for proposed foundry landfill. Subsequently performed construction oversight of clay borrow excavation and placement during landfill construction, Waupaca, Wisconsin.
- Supervision of drilling activities for a subsurface investigation and coordinated groundwater extraction well installation and aquifer performance test for groundwater remediation system for former trailer manufacturer, Edgerton, Wisconsin.
- Field supervisor for Phase II of a RCRA Facility Investigation at the former Chicago Incinerator Facility. Assisted in report preparation of RFI Draft and Final Reports.
- Performed Clay Borrow Investigation and construction oversite of waste placement and clay cap installation for the Mineral Point Roaster Waste Remediation Project, Mineral Point, Wisconsin.
- Supervised installation of groundwater extraction well which included logging over 100 feet of cored bedrock and assisted in aquifer performance test, Dane County Landfill No. 1, Verona, Wisconsin.
- Performed Phase I and Phase II Environmental Site Assessments for a papermill in Ladysmith, Wisconsin. Assisted in site inspection and report production.
- Performed construction oversite of Refuse Hideaway Landfill Cap Restoration and Improvements Project, Middleton, Wisconsin. Supervised clay placement, dredging of a sedimentation basin, and construction of riprap lined drainages.
- Performed Clay Borrow Investigation for Rhinelander Landfill, Rhinelander, Wisconsin. Located and documented potential clay sources and prepared report.
- Drilling supervision of soil borings in and around excavated surface impoundments for chemical analysis during a RCRA closure operation at a chemical plant in Ohio.
- Drilling supervision for a hydrogeologic investigation for a solid waste facility near Madison, Wisconsin. Drilling activities included installation of monitoring wells and rock coring in combination with groundwater zone sampling using inflatable packers.
- Performed field work for a subsurface investigation at a Naval Ordinance plant in Minnesota. Duties included installation of monitoring wells and a groundwater extraction well, groundwater sampling, and assisting in a five day aquifer test.
- Performed field work for Phase II of a Remedial Investigation at a CERCLA site in New York contaminated with PCBs.
- Performed groundwater sampling at CERCLA, RCRA, and at solid waste facilities at numerous sites in Wisconsin, Michigan, Illinois, New York, Indiana, Minnesota, and Missouri.

Professional History

Project Geologist, Dames & Moore, Madison, Wisconsin, May 1991 to present.

Hydrogeologic Technician, RMT, Inc., Madison, Wisconsin, 1987 to 1989.
Graduate Teaching Assistant, University of Missouri, Rolla, Missouri, 1989 to 1991.

Citizenship	United States
Countries Worked In	United States
Language Proficiency	English
Professional Affiliations	Association of Groundwater Scientists and Engineers National Ground Water Association

Curriculum Vitae

KAREN B. RICHARDSON, P.E.

Title

Project Manager

Expertise

Civil and Environmental Engineering
Transportation Engineering
Construction Management

Academic Background

B.S., Civil and Environmental Engineering, University of Wisconsin-Madison, 1984
Post graduate courses taken at UW-Madison in Solid and Hazardous Waste Engineering, Special Topics in Environmental Studies, Soils and Environmental Pollution, and Waste Geotechnics.

Registration

Licensed Professional Engineer - Wisconsin

Experience

Design, construction oversight, and operation and maintenance of remediation systems and transportation projects. Remedial projects include landfill capping projects and gas extraction systems, groundwater pump and treat systems, soil vapor extraction systems, thermal desorption, biopiling, and bioventing systems. Transportation projects include interstate highway, rural and urban highways, and bridges and box culverts.

- Prepared design plans, specifications and bid documents, managed the bidding process, and provided construction oversight of the Mineral Point Roaster Waste Remediation project. This project involved remediating approximately 100,000 cubic yards of mining wastes which had been polluting the adjacent stream for over 80 years. Design and construction included coordination with the Pecatonica Rail Commission. At completion of the project, the stream was clean enough to support trout.

  This project was awarded a Civil Engineering Achievement Award by the Wisconsin section of the ASCE in 1994, and was one of twenty projects that were nominated for the nationwide ASCE Outstanding Project award.

- Prepared design plans for an urban highway project, and provided construction oversight for urban transportation projects.

- Assisted in preparation of design plans and specifications for landfill final closure plans at two sites. Both sites included gas extraction systems and composite final soil cover with geomembranes.

- Prepared a Spill Prevention and Countermeasures Plan for a manufacturing facility.

- Prepared Stormwater Pollution Prevention Plans for manufacturing facilities.

- Prepared design plans and specifications and managed construction for a leachate extraction system at a closed papermill sludge landfill.

- Prepared design plans, specifications and bid documents for pump and treat groundwater remediation systems. These systems include groundwater treatment, soil vapor extraction, and offgas treatment with catalytic oxidation.

- Managed construction oversight of soil excavation projects involving petroleum contaminated soils.

- Investigated potential soil remediation technologies for a dioxin-contaminated site and prepared a Remedial Action Plan to recommend a cost-effective approach. Managed site through WDNR closure of soils and groundwater.
- Supervised a staff of seven in the design and preparation of highway projects.
- Prepared construction plans and specifications for several highway projects, including two large interstate resurfacing projects.
- Managed the construction of several highway facilities, including a large grading project, several bridges and box culverts, and an interstate resurfacing project.
- Managed a project which involved converting a railroad/roadway grade separation to an at grade crossing. This project included coordination with the Wisconsin DOT Bureau of Harbors and Rails.
- Represented the Department of Transportation at public informational meetings.

Professional History

Project Manager, Dames & Moore, Madison, Wisconsin, 1991 - present
Design Supervisor, Wisconsin Department of Transportation District 1, Madison, Wisconsin, 1989 - 1990.
Transportation Engineer, Wisconsin Department of Transportation District 1, Madison, Wisconsin, 1984 - 1989.

Citizenship

United States

Countries Worked In

United States

Language Proficiency

English

Conferences

Presented a poster display at the Sixteenth Annual Madison Waste Conference in Madison, WI entitled "The Mineral Point Roaster Waste Remediation Project"

Curriculum Vitae

DAVID P. TRAINOR, P.E., P.G.

Title	Managing Principal-In-Charge/Senior Engineer
Expertise	Environmental/Waste Management Hydrogeology Geotechnical Engineering
Academic Background	M.S., Civil and Environmental Engineering, University of Wisconsin, Madison, 1983 B.S. Civil Engineering, Ohio State University, 1978 B.S., Geology, Ohio State University., 1975
Registration	Professional Engineer: Wisconsin, Michigan, California, Idaho, Iowa Professional Geologist: Wisconsin

Experience

Directs a range of environmental projects, including feasibility/plan of operation landfill siting studies, RI/FS programs, groundwater assessments, remedial design, and construction management.

- Oversaw design and construction of a landfill gas extraction system for an abandoned sanitary landfill; Tomah, Wisconsin.
- Provided expert testimony for a food processing company siting a solid waste disposal facility.
- Provided expert testimony for a defendant for insurance claims for disposal of lead contaminated foundry sand; Milwaukee, Wisconsin.
- Oversaw investigation, developed remedial options, and directed remedial design for soils contaminated with manufactured gas plant waste (Northern States Power); Ashland, Wisconsin.
- Provided Agency negotiation, consultant review and oversight of an investigation and remedial options analysis for an abandoned sanitary landfill; Rice Lake, Wisconsin.
- Directed remedial design and remedial action oversight, including final cover and landfill gas control, for an abandoned municipal waste landfill; Wausau, Wisconsin.
- Directed remedial design activities, including final cover and landfill gas control, for an abandoned municipal waste landfill; Rhinelander, Wisconsin.
- Performed a groundwater assessment, negotiated Agency approval for a selected remedial option, and directed construction management of a leachate extraction system for a paper waste landfill; Eau Claire, Wisconsin.
- Directed preparation of design plans and specifications, and construction management for remediation of 200,000 cubic yards of mining wastes under the Wisconsin Environmental Repair Program; Mineral Point, Wisconsin.
- Directed work plan development, negotiated USEPA approval, and directed the investigation for an NPL abandoned landfill site; Tomah, Wisconsin.

- Directed investigation and remedial design activities for groundwater contamination from a former truck-trailer manufacturing operation; Edgerton, Wisconsin.

- Prepared and implemented USEPA-approved RCRA facility investigation work plan for a hazardous waste incinerator (CWM Chemical Services); Chicago, Illinois.

- Directed preparation of Plan of Operation for a 3.5 million cubic yard sanitary landfill, including expert testimony before the Waste Facility Siting Board; Madison, Wisconsin.

- Directed preparation of plans and specifications for landfill cover restoration, state Superfund site; Madison, Wisconsin.

- Directed a remedial investigation and feasibility study for groundwater remediation options for an abandoned landfill; Dane County, Wisconsin.

- Directed a remedial investigation for a wood treatment facility; Reed City, Michigan.

- Negotiated language for a voluntary consent order and directed investigation for a landfill remedial investigation (PRP group); Madison, Wisconsin.

- Coordinated design and construction of a landfill gas extraction system; Madison, Wisconsin.

- Directed preparation of a Feasibility Study and hydrogeologic assessment for a 1.5 million cubic yard industrial landfill; Wisconsin.

- Coordinated investigations and developed remediation options for several abandoned city sanitary landfills; Madison, Wisconsin.

- Developed a Feasibility Study for a 4 million cubic yard sanitary landfill, and provided expert testimony at a contested-case hearing; Madison, Wisconsin.

- Supervised subsurface investigations and prepared recommendations for remediation of two chlorinated hydrocarbon spill sites; Wisconsin manufacturing facilities.

- Supervised subsurface investigations and prepared hydrogeologic reports for several closed municipal landfill sites; Wisconsin.

- Prepared RCRA facility investigation work plan for a large military defense contractor (Hamilton Standards); Windsor Locks, Connecticut.

- Supervised investigations and developed remedial designs for several tank release sites; Wisconsin and Michigan.

- Developed remediation options for PCB-contaminated soils at an aluminum manufacturing plant; Kentucky.

- Coordinated investigation and developed design for a large demolition waste landfill facility; Portage County, Wisconsin.

- Developed an environmental and economic assessment for a county siting a hazardous waste facility; Minnesota.

- Prepared closure verification report for hazardous waste handling facilities in Wisconsin (APV Crepaco) and Illinois (Chemical Waste Management).

- Managed and performed an environmental assessment for acquisition of an abandoned power plant; Oklahoma.
- Performed environmental audits for AT&T; Waterloo and Dubuque, Iowa.
- Prepared feasibility/plan of operation report for a PCB transformer salvage facility; Juneau, Wisconsin.
- Designed a vacuum extraction system for remediation of an underground gasoline spill at a service station; Madison, Wisconsin.
- Designed and supervised construction of clay-lined earthen impoundments with dewatering facilities for foundry process sludge for a large industrial foundry facility; Defiance, Ohio.
- Devised geotechnical testing programs of various waste materials generated from paper manufacturing processes.
- Provided geotechnical analysis and recommendations for repair of a failure in a clay liner sidewall for a sanitary landfill; Minneapolis.
- Designed and implemented a modified multi-unit triaxial device to study the effects of leachate permeants on clay soils.
- Designed and provided construction documentation, kiln dust disposal facility; Alpena, Michigan.
- Designed and provided construction documentation, sanitary landfill; Minneapolis.
- Designed and provided construction documentation, foundry waste landfill; Milwaukee.
- Performed hydrogeological assessment of a solvent spill for an underground storage tank; South Bend, Indiana.
- Determined stability and projected settlements of embankments for bridge foundations; Idaho.
- Designed foundation and retaining structure recommendations for various commercial, industrial and transportation facilities; Idaho, Oregon and Washington.
- Designed foundation systems for residential, commercial and industrial buildings constructed on problem soils; San Francisco Bay area.
- Developed recommendations for the repair of residential structures damaged by soil expansion and settlement; San Francisco Bay area.
- Analyzed static and dynamic seacliff erosion and provided setback recommendations for a coastal development; Aptos, California.

Professional History

Managing Principal-in-Charge/Senior Engineer, Dames & Moore, Madison, WI, 1987-Present
Geotechnical Project Engineer, RMT, Inc., Madison, WI, 1983-1984; 1985-1987.
Geotechnical Project Engineer, Northern Engineering and Testing, Boise, ID, 1984-1985.
Staff Engineer, Terratech, Inc., San Jose, CA, 1978-1981.

Citizenship United States

Countries Worked In United States

Language Proficiency

English

Professional Affiliations

American Society of Civil Engineers
International Society for Soil Mechanics and Foundation Engineering
American Institute of Professional Geologists

Certifications

OSHA 40-hour Hazardous

Publications & Presentations

Co-author, "Groundwater Remediation at a DeInk Landfill," TAPPI Environmental Conference, 1994.

Author, "Isotope Aging to Determine Methane Gas Sources," Geological Society of America, National Conference, 1992.

Author, "Current Status of Environmental Assessments," Government Institutes Seminar, Madison, 1992.

Author, "RCRA Corrective Action - 1990," paper presented to the Minnesota State Bar Association, Minneapolis, 1990.

Author, "Investigation and Remediation of a Printing Solvent Release," paper presented at the short course Detection and Corrective Action for Leaking Underground Storage Tanks, Department of Engineering-Professional Development, University of Wisconsin, Madison, 1989.

Author, "Moisture and Saturation Effects on Hydraulic Conductivity Testing," paper presented at the ninth annual Madison Waste Conference, 1986.

Co-author, "Use of Foundry Quenched Slag as a Drainage Medium," paper presented at the ninth annual Madison Waste Conference, 1986.

Co-author, "Case Studies in Constructive Use of Foundry Wastes for Landfill Construction," paper presented at the American Foundrymen's Society Casting Conference, 1987.

Curriculum Vitae

J.P. WALKER, P.E.

Title	Senior Engineer
Expertise	Civil and Environmental Engineering/Waste Management
Academic Background	B.S., Civil and Environmental Engineering, University of Wisconsin-Madison, Wisconsin., 1977
Registration	Professional Engineer: Wisconsin, Minnesota and Virginia

Experience

Mr. Walker has more than 17 years in the design and construction of civil and environmental engineering projects. These projects have included the preparation of plans and specifications for pipeline projects, landfill designs including the use of geomembrane, clay liners and caps, leachate extraction systems and gas extraction systems. Mr. Walker has also designed groundwater extraction systems using pump and treat technology, and soil vapor extraction systems.

Solid Waste Management Projects

- Managed the design and installation of NR 500 final cover and liner systems, landfill gas management systems, leachate management systems, and stormwater management systems for landfills in Wisconsin. Final cover and liner system components include soil layers and geosynthetics. Landfill management systems included horizontal and vertical extraction wells, header pipes, condensate drains, storage tanks and loadout facilities, blower units, and flare units, consisting of candlestick flares and enclosed flares. Major emphasis was placed on preparing design plans and construction specifications, coordinating construction management, and preparing detailed construction documentation reports. The Wisconsin landfills that included the work described above are:
    - Truax Landfill
    - Holtz-Krause Landfill
    - Rhinelander Landfill
- Managed the design and construction for a foundry waste landfill for a foundry in Wisconsin. The project included construction management services and documentation of construction. Major components include clay liner, leachate collection system including lift station, underground storage tanks with loadout facilities, stormwater management system included sedimentation basins, access road, and final cover systems consisting of soil layers. Two of three phases have been constructed, with the final phases scheduled for construction in 1996 and 1997. Current work includes evaluation for horizontal expansions.
- Provided independent technical review of a permit modification to a sanitary landfill in Illinois. As a technical adviser to a local developer, Mr. Walker reviewed detailed designs for a leachate management system, landfill gas management system consisting of horizontal collection pipes and vertical extraction wells, header pipe systems, condensate management systems and enclosed flares.
- Prepared plans and specifications for a multi-phased landfill closure system; Henrico County, Virginia. Design features included geomembrane cap with composite drainage layer, slurry wall, leachate extraction wells and lift station, and a passive gas venting network..

- Prepared plans and specifications for a vertical expansion to an existing landfill which consisted of developing separate disposal cells for municipal solid waste and incinerator ash; Montgomery County, Maryland. Design features included geonet, geomembrane liner and composite drainage layer at the base of the vertical expansion and a geomembrane cap and composite drainage layer in the final cover system.

- Prepared plans and specifications for an enclosed solid waste transfer facility capable of handling 1000 tons of waste per day; Craven County, North Carolina. Design features included facility layout, grading plans and structural details for access roads, surface water drainage control features, wash-water management system, scale installation and a public drop off area.

- Prepared plans and specifications for a horizontal expansion of an existing landfill; Newport News, Virginia. Design features included a composite liner system consisting of geomembrane atop a clay liner and a synthetically lined leachate storage lagoon.

- Prepared a Plan of Operation, Construction Quality Assurance Plan, Contingency Plan and Closure Plan for a 2.0 million cubic yard sanitary landfill; Newport News, Virginia.

- Designed a landfill closure system for a municipal solid waste landfill; Franklin, Virginia. Design features included a geomembrane cap and composite drainage layer.

- Functioned as a technical adviser to a County government during the permitting and design process of a full-service vendor for a horizontal expansion to an existing landfill; Prince George's County, Maryland. Design features included a composite liner system, drainage layers consisting of synthetic drainage nets and granular material, and a leachate extraction system.

- Designed a horizontal expansion to an existing zone of saturation landfill; Franklin, Wisconsin.

- Prepared Integrated Solid Waste Management Plans for Adams, Juneau, Langlade, and Ozaukee Counties in Wisconsin. The Plans included waste generation and characterization data, analyses of waste management alternatives, and cost analyses.

- Designed and implemented the closure of a private landfill; Darlington, Wisconsin.

- Designed and implemented the construction of a clay lined landfill; Darlington, Wisconsin.

- Designed and implemented a horizontal expansion of an existing sanitary landfill; Middleton, Wisconsin.

Environmental Remediation Projects

- Managed the design and installation of a groundwater remediation system at a petroleum storage site in Wisconsin. The design effort included the preparation of design plans and construction specifications. System installation was completed in 1995.

- Managed the design and installation of a groundwater remediation system at a nuclear power plant in Michigan.

- Managed an underground storage tank removal/replacement project for the Air National Guard which involves the testing, permitting, tank removal/replacement and site remediation in Wisconsin. Contract specifications were prepared for tanks removals, soil testing, and tank replacements. Currently performing investigation of specific tank locations to determine environmental impacts. Future remediation work may be required.

Curriculum Vitae

CHRISTOPHER E. WEGGEMANN, AIA

Title	Managing Principal
Expertise	Phase I and Phase II Environmental Site Assessments, Compliance Audits, Asbestos/Lead Issues, and Building Design & Construction Management

Experience

Directs a range of environmental projects, including Phase I and Phase II environmental assessments, soil and ground water monitoring and remediation programs, and compliance audits. In addition, Mr. Weggemann is actively involved in building design and construction management issues.

Mr. Weggemann is the Managing Principal of Dames & Moore's Minneapolis office, therefore is also actively involved with business devopment and client interaction.

- Project Director for a multi-site groundwater investigation. This investigation was initiated to investigate groundwater impact to adjacent and downgradient properties from former underground storage tanks.

- Project Director for a Phase II Assessment of a manufacturing facility in Durham, North Carolina. This project was conducted to identify the extent of potentially affected soil and groundwater in several areas of concern identified in the Phase I Environmental Site Assessment. Project included the preparation of a sampling work plan, report preparation and interacting with the regulatory agencies.

- Project Manager for the installation of a long term air monitoring program for a distribution facility in the State of Wisconsin. This project included coordination with the state agencies for monitor placement and sampling parameters, preparation of a sampling plan, and submittal of quarterly reports.

- Project Manager for a multi site Phase I Environmental Site Assessment totaling thirty-five sites over ten states and seven countries. The sites consisted of manufacturing facilities with a variety environmental and compliance issues. This project also included conducting Phase II investigations on seven of the sites with issues ranging from petroleum contamination to wastewater discharge.

- Designed project specifications for the removal of lead-based paint (LBP) from several steel bridges. This work included an assessment of the existing coating system, project specifications and testing during the removal of the LBP.

- Developed a comprehensive remediation program for the removal of lead oxide dust from an industrial facility. This project included establishing a cleanup criteria, development of a work plan, and managing the remediation contractor.

- Has conducted several Indoor Air Quality (IAQ) studies for the review of existing buildings air quality and HVAC effectiveness.

- Provided the development of many asbestos related project related designs from occupied buildings. This work has included building surveys, removal specifications, construction management, operation and maintenance programs and general consulting on asbestos issues.
- Provided expert witness testimony in litigation involving construction disputes, property damage issues and property tax reduction protest.
- Conducted detail studies of new material application and their effect on the existing structure with respect to the indoor air quality. This work has included monitoring and environmental chamber test for volatile organic compounds (VOC), carbon monoxide (CO), molds and fungi growth.
- Conducted a comprehensive renovation of the Gold Concourse, gates 1-9 at the Minneapolis/St. Paul International Airport. This project required continuous passenger access to all areas of the concourse, therefore required the installation of a detailed scaffolding system to support the work.

Technical Conferences/ Publications

Environmental Information Association (EIA), March 1994
Presented a seminar titled "Lead Based Paint Abatement from Steel Structures". This Presentation discussed the methodologies, cost and current trends associated with the remediation of lead based paint from steel structures, including bridges, tanks, towers, etc.

Canadian Chapter of the National Asbestos Council, May 1992
Presented a discussion comparing the National Building Code of Canada and the Uniform Building Code in the United States and their relationship projects involving hazardous materials.

Weggemann, C.E., "Keeping it in Place - O&M Program can Control Asbestos," Minnesota Real Estate Journal, February, 1991.

National Asbestos Council, September 1989
Presented a paper discussing HVAC surface contamination and its relationship to the ambient air. This presentation was bases on an actual project work that was conducted by Mr. Weggemann and discussed the results.

Academic Background

Bachelor of Architecture, University of Kansas, 1985
Bachelor of Environmental Design, University of Kansas, 1984

Registrations/ Certifications

National Council of Architectural Review Boards (NCARB) Certification
Registered Architect - Kansas, Minnesota, Wisconsin and Illinois
AHERA Certified Building Inspector and Management Planner
AHERA Certified Project Designer
State of Illinois Certified Asbestos Project Designer

Citizenship United States

Language Proficiency English

Professional Affiliations American Institute of Architects (AIA)

Glenn Bruxvoort, P.E. — Senior Project Manager

Education

Bachelor of Science
Civil Engineering
Calvin College
Grand Rapids, Michigan
(1975)

Masters of Civil and
Environmental Engineering
University of Wisconsin
Madison (1987)

Professional Registration

Professional Engineer in Wisconsin, Michigan, and Minnesota

Professional Certifications

40 Hour Health and Safety Certifications

8 Hour annual H & S Refresher

Annual Medical Monitoring Certified

Nuclear Density Testing Certification

Professional Associations

American Society of Civil Engineers

National Groundwater Association

General Background

Geotechnical Engineer with experience in landfill design and construction, foundation design and construction, remedial design and construction, slope stability evaluation, construction materials testing and construction document reports. Experience in pre-purchase site assessments, underground storage tank closure assessments, remedial investigations, corrective action plans, and remedial systems construction.

Experience

Experience prior to joining SEH includes six years of consulting engineering.

Representative projects include:

Moccasin Mike Landfill - Superior, Wisconsin. Responsible for design, bidding and construction for vertical expansion. Project components included evaluation of existing leachate collection system and evaluation of alternatives, design of leachate pumping system, design of access road, design of composite cap and design of active extraction system.

Pine _____ Sanitary Landfill - St. Paul Minnesota. Clay borrow investigation for clay to be used in the construction of Phase I to III. Slope stability analysis for marsh excavation. Resident Project Representative during construction of Phase __ clay liner, HDPE lysimeter, and leachate collection system. Prepared construction documentation report for submittal to MN Pollution Control Agency.

Klim__ Bulk Oil - Independence, Wisconsin. Initiated Emergency Response to reported petroleum release. Vapor extraction system installed within three days of initial contact. Responsible for continued investigation and monitoring of groundwater.

WDOT Highway 29 Improvements - Shawano County, Wisconsin. Geotechnical engineer responsible for designing and implementing exploration program for over 30 miles of highway expansion. Made recommendations in accordance with WDOT guidelines.

Jenni__ - Eau Claire, Wisconsin. Responsible for design and construction of vapor extraction system to remediate soils contaminated with isopropanol and acetone. Expert witness testimony regarding cause of release. Site closeout achieved.

Carnation Company - Eau Claire, Wisconsin. Geotechnical exploration for three facility expansions. Recommendations for site work, foundations, slabs, and lateral earth pressures.

Dow Sludge Detwatering Facility Closure Cap - Midland, Michigan. project involved constructing composite cap over sludge ponds. Responsible for coordination and documentation of testing on clay cap and HDPE cover and preparing construction documentation report for submittal to MI DNR.

New Richmond Landfill - New Richmond, Wisconsin. Design and construction of clay cap and monitoring system to meet the requirements of a consent order. Also included the design of a passive gas extraction system with the ability to be converted to an active gas extraction system.

"Quality Assurance for Geotechnical and Construction Materials Laboratories." QA/QC for laboratory testing of strength and indicator tests of soil, aggregate, concrete and asphalt. Implement and apply software for laboratory applications. Developed software for clay liner leachate compatibility study.

Milwaukee Metropolitan Sewage District - Milwaukee, Wisconsin. Developed testing program to determine geotechnical properties of sludge products for use in designing land-based disposal options. Involved in designing and constructing a bench-scale apparatus including instrumentation. Developed software for compiling and interpreting data. Also used apparatus for determining geotechnical properties of paper mill sludge.

Bay City Bus terminal - Bay City, Michigan. Performed Phase I pre-purchase site assessment and recommended Phase II investigation. Implemented Phase II investigation to determine extent of contamination from UST. Implemented remediation action to obtain closure on the site, prior to site development.

Northwoods Sanitary Landfill - Cameron, Wisconsin. Active gas extraction design, gas flare design and leachate loadout facility specifications. Construction documentation of soils and lift station work for Phase I construction.

Pevan Transfer and Storage - Chippewa Falls, Wisconsin. Remedial investigation and corrective action plan for Diesel Fuel Release. Recommended and cosigned combined excavation/thermal treatment and vapor extraction/bio-venting for remediation.

AJ Manufacturing - Bloomer, Wisconsin. Remedial investigation and design for petroleum release. Evaluated potential impact on nearby municipal well. Designed combination SVE/air sparging as the remedial alternative.

Marquart Motors - Chippewa Falls, Wisconsin. Implemented Phase I assessment for proposed purchase and remodeling of facility. Implemented Phase II through Phase IV including WDNR site closure prior to remodeling. Coordinated site activities with SEH Architectural services.

Park Ridge Distributing - Eau Claire, Wisconsin. Implemented Phase I remedial investigation through remediation (asphalt incorporation) for UST release. Groundwater was impacted but able to obtain WDNR site closure.

Frank J. Lowry, P.E., P.G. — Project Manager

Education

Bachelor of Science
Civil Engineering
University of Wisconsin
Platteville (1988)

Bachelor of Science
Geology, University of
Wisconsin Platteville (1984)

Professional Registration

Professional Engineer in Wisconsin

Professional Geologist in Wisconsin

Qualified Hydrogeologist in Wisconsin

Professional Certifications

40 Hour Health and Safety Certifications

8 Hour annual H & S Refresher

Annual Medical Monitoring Certified

Nuclear Density Testing Certification

Professional Associations

Association of Groundwater Scientists and Engineers

American Society of Civil Engineers

General Background

Project Engineer/Hydrogeologist with experience in aboveground and underground storage (AST/UST), solid waste, environmental site assessments and other geological/hydrogeologically related projects. Frank services as project manager for many petroleum investigation/cleanup projects. Frank's experience with USTs includes removal assessments, remedial investigations and remedial design. Other geologic/hydrogeologically related experience includes landfill design, gas and groundwater monitoring well design, soil testing and classification, and rock core drilling and logging. This experience lends itself to performing a wide variety of environmental projects.

Experience

Project Engineer/Hydrogeologist for

Augusta Oil and Feed - Augusta, Wisconsin. Responsible for a Phase I Remedial Investigation for a petroleum release, followed by design, bidding and construction oversight of thermal treatment and a sparge/SVE remedial system.

St. Joseph's Hospital - Chippewa Falls, Wisconsin. Responsible for a Phase I Remedial Investigation, infield SVE design, and bidding and installation of SVE facilities leading to project closeout.

Baker Oil Company - Hawkins, Wisconsin. Responsible for Phase I Remedial Investigation at a UST site and an AST bulk fueling facility.

Northside Motors - Menomonie, Wisconsin. Responsible for Phase I Remedial Investigation and remedial design for a sparge/SVE system.

Colfax, Wisconsin. Groundwater monitoring well installation and hydraulic conductivity testing for an Infield Condition's Report followed by plans and specifications, bidding, construction oversight, and construction documentation for closure of a solid waste landfill.

Stanley, Wisconsin. Plans, construction oversight, and construction documentation for closure of a solid waste landfill.

St. Croix River Valley Landfill. Monitoring well installation and groundwater investigation for completion of a Groundwater Investigation Report.

Bruce, Wisconsin. Environmental Contamination Assessment including leachate beadwell design and construction observation at an abandoned papermill sludge and municipal waste landfill.

Superior, Wisconsin. Engineer for an Environmental Contamination Assessment for a municipal landfill.

Superior, Wisconsin. Engineer for operations at the municipal landfill, including lift station design, construction oversight, and an Initial Site Report for explanation.

Mond__, Wisconsin. Plans and specifications and construction documentation for closure of a solid waste landfill and an infield Conditions Report for design and construction documentation of a demolition landfill.

Polk County, Wisconsin. Phase I Hazardous Materials Investigations for CTH M and CTH E reconstruction.

Solid Waste Recovery Systems, Inc. Small demolition landfill design, construction documentation and monitoring.

Wisconsin Department of Transportation District 2. Phase I Hazardous Materials Investigations for STH 59 Improvements.

US Small Business Administration - Cornell, Wisconsin. Environmental site assessment for a wood treatment facility.

Cadott, Wisconsin. Phase I Environmental Site Assessment for industrial park development.

Northwest Airlines - Hibbing, Minnesota. Environmental site assessment for a proposed engine repair facility in Hibbing, Minnesota.

Minnesota Aquafarms. Hydrogeologic assessment for a fish farming operation in Chisholm, Minnesota.

Town of Hallie, Wisconsin. Municipal well location study for a potable water supply.

Caddot, Wisconsin. Municipal well location study for a potable water supple.

Caddot, Wisconsin. Wellhead protection plan for the municipal well field.

Christopher Haller — Senior Technician

Education

Bachelor of Science
Environmental Science
Mount Senario College
(1991)

Specializations

- Site Remediation
- Operation and Maintenance
- Remediation Equipment
- Construction Oversite
- Site Construction
- Equipment Installation

Training

40 Hour OSHA Hazardous Waste Operations Course
WI UST Site Assessment 1994

40 Hour Health and Safety Certifications

8 Hour annual H & S Refresher

Annual Medical Monitoring Certified

Michigan Industrial Wastewater Treatment Course 1, 1992

Michigan Wastewater Treatment Plant Operator Certification A-2b, B-3b

General Background

Senior Technician with experience in preparation and review of plans and specifications, construction observation for a variety of soil venting, air sparging and groundwater recovery systems. Also experienced in remedial excavations, site remediation design, site study and remedial alternative testing.

Responsible for development of operation and maintenance procedures, training of entry level technical staff in field testing, system monitoring and maintenance.

Experience

Altoona Landfill Passive Gas Extraction Site - Altoona, Wisconsin. Installation of landfill lateral extraction trench and piping. Monitoring methane migration and study for effects of the passive extraction trench system. Study and test active methane gas extraction by performing a series of vacuum pilot tests on gas extraction well within the landfill.

Olson Motor Clinic - Bloomer, Wisconsin. Responsible for soil vapor extraction installation, bidding, contract administration and onsite construction oversite during installation of soil vapor extraction system.

Chippewa County Highway Department - Bloomer, Wisconsin. Responsible for soil vapor extraction installation, bidding, contract administration and onsite construction oversite during installation of (SVE) system. Development and supervisor of the operation and monitoring schedule for the SVE system. System includes catalytic combustion air treatment.

Northwestern Wisconsin Electric Company - Frederic, Wisconsin. Site study including work plan and implementation of work plan for air sparge pilot test and soil vapor extraction pilot test. Design of remedial system and site supervision for installation.

Experience prior to joining SEH includes two years of consulting.

West Bend Landfill, Active Gas Extraction - West Bend, Wisconsin. Responsible for operation, maintenance, and troubleshooting, of LPG fueled ground flair. Troubleshoot, monitor, and maintain electrical controls of system.

Mobil Oil Corporation - Upper Midwestern Region, MI, WI, IL, IN, OH. Remediation systems design review, installation, construction observation, operation, monitoring and supervision of technical staff for over 40 remediation sites, including air sparging, soil vapor extraction and groundwater recovery systems.

Wayne Recycling and Reclamation EPA Super Fund, Remediation Site - Columbia City, Indiana. Site study, remedial alternatives testing, and remedial design. Plans and specifications for combination groundwater recovery, air sparge injection and soil vapor extraction. Work plans for removing and cleaning of multiple above ground storage tanks.

ARCO Pipeline - Indiana, Ohio. Site study, remedial alternatives testing, and site remediation of a petroleum contamination site.

Rockwell International - Detroit, Michigan. Coordinate remedial excavation and on-site contract administration and site supervision of a petroleum contamination site.

Wisconsin Central Railroad Co. - Eastern Wisconsin. Site supervisor on a multi-site petroleum underground storage tank removal project. Include Phase I site work and reporting of Phase I results.

Complete Auto Transit, Inc. - Janesville, Wisconsin. Field instrumentation of design changes to remediation system including start-up and trouble shooting of system construction for petroleum reducation. Supervision of operation and maintenance staff for remediation system.

Brian L. Kent — Environmental Technician

Education

Bachelor of Arts
Environmental Science
Biology
Mount Senario College
(1994)

Specializations

Site Remediation
Operation and Maintenance
Construction Oversite
Site Construction
Equipment Installation

Training

40 Hour OSHA Hazardous Materials Training per OSHA 29 CFR 1920.120

Certified for use of Troxler Electronics Laboratories Nuclear Testing Equipment

General Background

Technical with experience in review of plans and specifications, construction observation for a variety of soil venting, air sparging, and groundwater recovery systems. Also experienced in remedial excavations, site remediation design, and remediation alternative testing.

Responsible for implementation of operation and maintenance schedule on all existing remediation systems which includes groundwater, soil, and vapor sampling. Duties also include preparation of quarterly/annual progress reports and Construction Documentation Reports, and Operations, Maintenance, and Monitoring Plans for all remediation systems.

Responsible for coordination and performance of soil vapor extraction, air sparging and groundwater pump pilot tests.

Experience

Responsible for design, installation, modifications, environmental compliance, data interpretation, and reporting for over 25 remediation systems.

Northwestern Wisconsin electric Company - Frederic, Wisconsin. Responsible for construction oversight for the installation of an air sparge, groundwater, and air recovery system. Groundwater treatment utilized Biological Activate Treatment System (BATS) technology.

Rihn Oil - Bloomer, Wisconsin. responsible for construction oversight for the installation of an air sparge/soil vapor extraction biopile system. System includes active venting of previously excavated petroleum contaminated soil stockpiled and covered with a polyethylene liner.

Deiss Sanitation - River Falls, Wisconsin. Responsible for the supervision for the installation of a vacuum enhanced free product and groundwater recovery system. Treatment utilizes volatization of contaminants via air stripping technology.

St. Croix River Valley Landfill - Osceola, Wisconsin. Responsible for setting modifications to optimize extraction of methane gas from under existing office and maintenance buildings. Active methane venting was initiated to reduce explosive hazard in working environments.

Village of Kendall - Kendall, Wisconsin. Responsible for oversite on excavation of petroleum contaminated soils and onsite construction oversite of soil vapor extraction and groundwater treatment piping. Responsibilities included inspection of HDPE fusion, manifest destiny of over 6000 tons of contaminated soil to thermal treatment, inspection of well design and construction, and overall specification details.

Augusta Oil and Feed - Augusta, Wisconsin. Responsible for oversite of excavation of petroleum contaminated soils and onsite construction oversite of soil vapor extraction and air sparge piping layout. Conducted soil vapor extraction and air sparge pilot tests to determine the effectiveness and design parameters.

APPENDIX B

GAC SYSTEM DETERMINATION MATRIX

21 homes with a (b) or (c) footnote.

Table B-1
GAC System Determination for Private Wells Within SWCA
Page 1 of 2

RESIDENCES RECEIVING BOTTLED WATER

		Analyte (μg/L)		*GAC System*	
Address	**Sample Date ***	**TCE**	**PCE**	**JL-100**	**JL-300**
942 Alexander Road	8/5/94	5.6	0.66	✓	
Troop House 965 Alexander Road	3/1/95	0.3	0.3 J	✓	
919 100th Girl Scout Troop	8/4/94	<0.06	<0.05	✓	
Caretaker Girl Scout Camp	8/4/94	<0.06	<0.5 (a)	✓	
Day Camp Girl Scout Camp	8/4/94	<0.06	<0.05	✓	
Windy Acre Girl Scout Camp	8/4/94	1.3	0.63	✓	
947 Bakken Road	8/11/94	4.1	0.52	✓	
954 Bakken Road	8/11/94	2.2	<0.5 (a)	✓	
957 Bakken Road	8/10/94	3.2	0.76	✓	
888 Chippewa Path	8/10/94	<0.06	<0.05	✓	
920 Chippewa Path	8/11/94	<0.06	<0.05	✓	
935 Chippewa Path	8/4/94	<0.06	<0.05	✓	
981 County Road A	3/2/95	<0.2	<0.2	✓	
612 E. Highway 12	(b)				
890 E. Highway 12	3/2/95	30 E	<0.2		✓
888 E. Highway 12	8/8/94	21	<0.5 (a)		✓
898 E. Highway 12	8/5/94	28	<0.05		✓
877 Kingsway Road	(b)				
878 Kingsway Road	(c)				
881 Kingsway Road	8/6/94	1.6	<0.5 (a)	✓	
756 Holden Lane	8/8/94	7.8	<0.5 (a)	✓	
766 Holden Lane	8/11/94	<0.06	<0.05	✓	
767 Holden Lane	8/10/94	<0.6 (a)	<0.05	✓	
772 Holden Lane	8/3/94	<0.06	<0.05	✓	
779 Holden Lane	8/3/94	<0.06	<0.05	✓	
780 Holden Lane	(c)				
783 Holden Lane	8/3/94	8.3	<0.5 (a)	✓	
792 Holden Lane	8/9/94	6.8	<0.5 (a)	✓	
917 LaBarge Road	8/3/94	<0.6 (a)	<0.5 (a)	✓	
923 LaBarge Road	6/21/95	<0.2	<0.2	✓	
929 LaBarge Road	8/4/94	0.66	<0.5 (a)	✓	
940 LaBarge Road	8/11/94	<0.06	<0.05	✓	
948 LaBarge Road	8/4/94	<0.06	<0.05	✓	
953 LaBarge Road	8/4/94	8.4	0.72	✓	
959 LaBarge Road	8/8/94	8.2	0.56	✓	

These have filters from Norlake

Table B-1
GAC System Determination for Private Wells Within SWCA
Page 2 of 2

Has a new well been installed since 3/95? check with Pat.

RESIDENCES RECEIVING BOTTLED WATER					
		Analyte (μg/L)		*GAC System*	
Address	**Sample Date ***	**TCE**	**PCE**	**JL-100**	**JL-300**
963 LaBarge Road	3/2/95	0.9 J	0.3 J	✓	
677 McCutcheon Road	5/7/90	1.4	ND	✓	
695 McCutcheon Road	8/3/94	<0.6 (a)	<0.05	✓	
696 McCutcheon Road	8/3/94	2.9	<0.5 (a)	✓	
712 McCutcheon Road	8/4/94	1.4	<0.5 (a)	✓	
756 McCutcheon Road		(b)			
757 McCutcheon Road	8/11/94	1.7	<0.5 (a)	✓	
763 McCutcheon Road	8/3/94	8.6	<0.5 (a)	✓	
771 McCutcheon Road	8/4/94	<0.06	<0.05	✓	
775 McCutcheon Road	8/10/94	4	0.54	✓	
783 McCutcheon Road	8/11/94	2.3	<0.5 (a)	✓	
786 McCutcheon Road	3/1/95	1	<0.2	✓	
794 McCutcheon Road	8/10/94	4.9	<0.5 (a)	✓	
720 Norflex Drive	6/21/95	1	<0.2	✓	
985 Scott Road	8/3/94	<0.2	<0.05	✓	
981 Tanney Lane	8/9/94	5.2	<0.5 (a)	✓	
982 Tanney Lane	3/2/95	5 J	0.6 J	✓	
994 Tanney Lane	3/2/95	<0.2	<0.2	✓	
997 Tanney Lane	8/10/94	<0.06	<0.05	✓	
998 Tanney Lane	8/3/94	<0.2	<0.05	✓	
1001 Tanney Lane	9/27/94	<0.06	<0.05	✓	
848 Yellowstone Trail	8/4/94	<0.06	<0.5 (a)	✓	

Tome 57

Notes: * Most recent sample date analytical results for TCE and PCE only.
Not final determination for GAC System; will be updated based on September 1996 sample analysis results.
SWCA = Special Well Construction Area.
All values reported in μg/L.
(a) Value reported as below laboratory quantitative limit.
(b) No record of sampling; Appendix N in Remedial Investigation Report Junker Landfill prepared by Wenck Assoc., Revised July 1995.
(c) No one home during 8/94 sampling event.
J Value should be considered an estimate due to minor QC deviations, method biases, or proximity to the instrument detection limit.
E Concentration exceeded upper calibration concentrations of the initial calibration curve. A dilution is then performed to bring the concentration within the reliable quanification range. Value is estimated, and likely biased high.

add 932 Chippewa Path
877 Kingsway
878 Kingsway
882 Kingsway

Table B-2
GAC System Determination for Private Wells Within SWCA
Page 1 of 2

This has filter from Norlake

881

RESIDENCES NOT RECEIVING BOTTLED WATER					
		Analyte (µg/L)		*GAC System*	
Address	**Sample Date ***	**TCE**	**PCE**	**JL-100**	**JL-300**
948 Bakken Road	(c)				
961 Bakken Road †	8/3/94	2.5	< 0.5 (a)	✓	
962 Bakken Road	8/3/94	3.4	< 0.5 (a)	✓	
970 Bakken Road	8/28/94	< 0.6 (a)	< 0.5 (a)	✓	
921 Chippewa Path	(c)				
935 Chippewa Path	(c)				
928 County Road A	3/1/95	< 0.4	< 0.2	✓	
965 County Road A	3/2/95	< 0.2	< 0.2	✓	
980 County Road A (Town Hall)	3/6/95	< 0.2	< 0.2	✓	
606 E. Highway 12	(b)				
818 E. Highway 12	8/29/94	27 (d)	< 0.05	(d)	(d)
887 E. Highway 12	8/14/94	< 0.06	< 0.05	✓	
892 E. Highway 12	8/9/94	< 0.06	< 0.05	✓	
882 Kingsway Road	(c)				
761 Holden Lane	(c)				
773 Holden Lane	(c)				
787 Holden Lane	(b)				
790 Holden Lane	(b)				
891 LaBarge Road	(b)				
932 LaBarge Road †	3/1/95	10	1	✓	
671 McCutcheon Road	8/5/94	< 0.6 (a)	< 0.5 (a)	✓	
690 McCutcheon Road	8/29/94	< 0.06	< 0.05	✓	
692 McCutcheon Road	(b)				
704 McCutcheon Road	8/11/94	1.1	< 0.5 (a)	✓	
718 McCutcheon Road - Barn	8/4/94	< 0.6 (a)	< 0.5 (a)	✓	
763 McCutcheon Road - House	8/4/94	< 0.6 (a)	< 0.5 (a)	✓	
760 McCutcheon Road	(b)				
767 McCutcheon Road	(b)				
795 McCutcheon Road	8/3/94	6.9	1.8	✓	
812 McCutcheon Road	8/11/94	< 0.06	< 0.05	✓	
832 McCutcheon Road ††	(b)				
981 McCutcheon Road	8/11/94	< 0.06	< 0.05	✓	
977 Scott Road	(b)				
993 Scott Road	8/3/94	< 0.2	< 0.05	✓	
986 Tanney Lane	(c)				

Notes: * Most recent sample date analytical results for TCE and PCE only.
Not final determination for GAC System; will be updated based on September 1996 sample analysis results.
SWCA = Special well Construction Area.
All values reported in μg/L.

- † Refused service per Wenck, RI Work Plan, December 1994.
- †† Labeled Windy Acres on map.
- (a) Value reported as below laboratory quantitative limit.
- (b) No record of sampling; Appendix N in Remedial Investigation Report Junker Landfill prepared by Wenck Assoc., Revised July 1995.
- (c) No one home during 8/94 sampling event.
- (d) Duplicate sample - TCE = <0.06, PCE = <0.05; GAC system determination will be made based on 1996 sample results.
- J Value should be considered an estimate due to minor QC deviations, method biases, or proximity to the instrument detection limit.
- E Concentration exceeded upper calibration concentrations of the initial calibration curve. A dilution is then performed to bring the concentration within the reliable quanification range. Value is estimated, and likely biased high.

APPENDIX C

GAC FILTER INSTALLATION PLAN PUBLIC EDUCATION AND INFORMATION GAC SYSTEM INSTALLATION

1.0 INTRODUCTION

This Alternate Water Supply Plan has been prepared in response to a request from the WDNR during discussions on preparation of this Work Plan. The Landfill Remediation Trust retained Dames & Moore to prepare this plan. The Consent Decree requires the Landfill Remediation Trust submit a plan to install and maintain point-of-entry granulated activated carbon filters that have been approved by WDNR and the Wisconsin Department of Commerce (DCOMM) in each home and business within the Special Well Construction Zone that is located in Sections 13, 14, 15, 22, 23, or 24 of the Town of Hudson, T29N, R19W, St. Croix County, Wisconsin, and in the Norflex Inc. facility at 720 Norflex Drive in the Town of Hudson. Dames & Moore proposes the use of the JL-100 or JL-300 filter, manufactured for Landfill Remediation Trust.

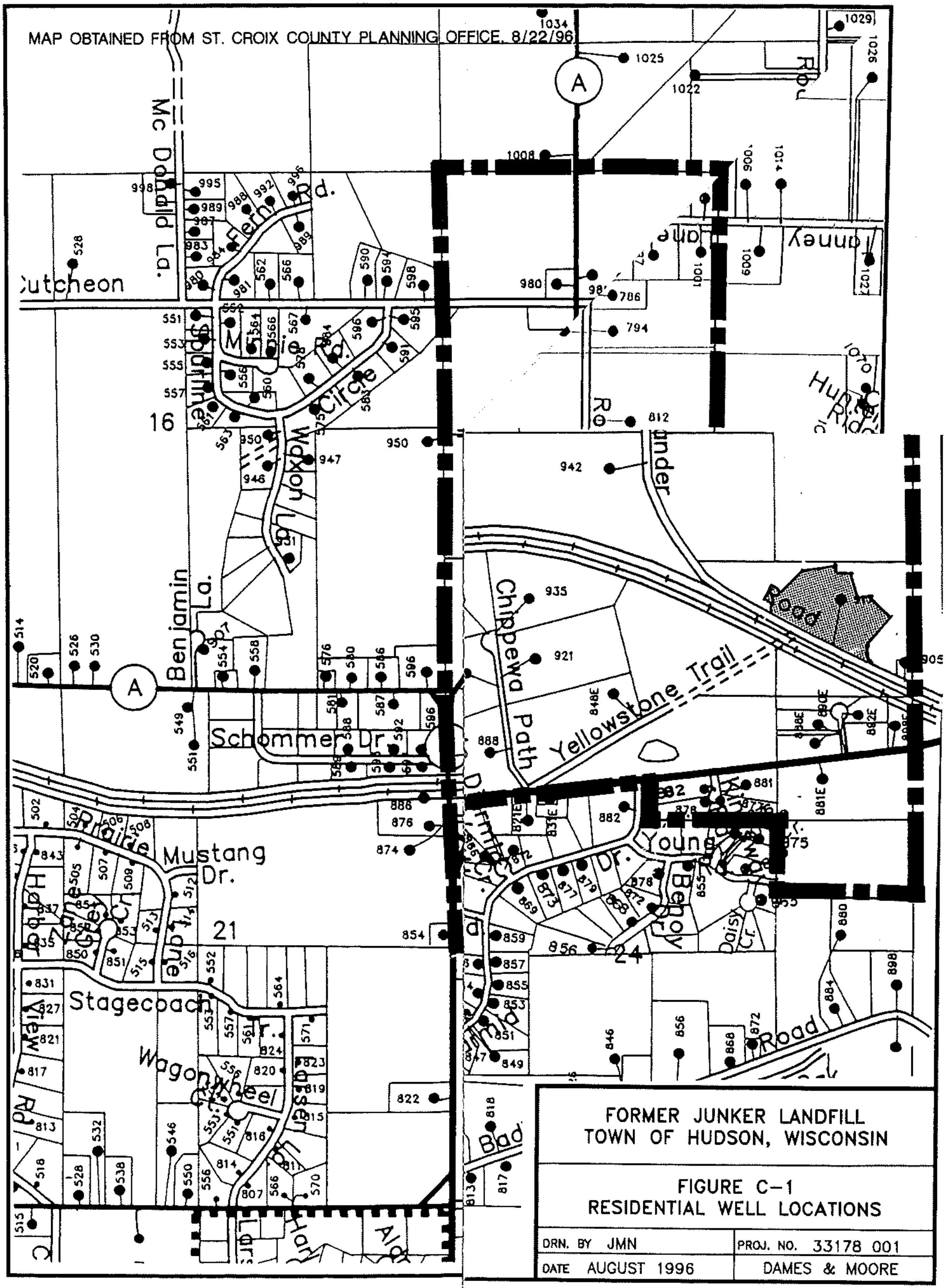
1.1 BACKGROUND

Current practice includes the delivery of bottled water to every private residence and business within Sections 13, 14, 15, 22, 23, or 24 that are also located within the SWCA that desires it. The Landfill Remediation Trust will continue to supply bottled water to these residences until the approved GAC filters are installed.

Figure C-1 is a map from the St. Croix County Planning Office that shows the locations of all residences and businesses. This map was updated with current SWCA information and the location of future residences as indicated by the Township of Hudson Building Inspector. The Landfill Remediation Trust is proposing to use the JL-100 or JL-300 GAC filters as the more permanent water supply replacement. Units similar to the JL-100 and JL-300 filter systems have been approved by the Wisconsin Department of Commerce (DCOMM) and the Wisconsin Department of Natural Resources (WDNR) for use on water supplies in other areas of Hudson Township. Efforts are underway to receive approval of the JL-100 and JL-300 system in the aforementioned area of Hudson Township.

The JL-100 and JL-300 point of entry GAC filters consist of two tanks filled with granular activated carbon. The tank size in the JL-100 system is 8.4 inches in diameter, 43.6 inches tall and contains 1.25 cubic feet of activated carbon. The JL-300 tank is 14.2 inches in diameter, 46.2 inches tall and contains 3.61 cubic feet of carbon. Various appurtenances connect the two-tank filter system to the water supply piping.

MAP OBTAINED FROM ST. CROIX COUNTY PLANNING OFFICE, 8/22/96



FORMER JUNKER LANDFILL
TOWN OF HUDSON, WISCONSIN

FIGURE C-1
RESIDENTIAL WELL LOCATIONS

DRN. BY JMN	PROJ. NO. 33178 001
DATE AUGUST 1996	DAMES & MOORE

FILE: C:\DWG\JUNKER\FIG1,2-1,7-2

2.0 PUBLIC EDUCATION AND INFORMATION

The public will be informed of the decision to install the GAC filters and when the installation is scheduled to proceed through the plan outlined in the Community Relations Plan for this site. The plan for disseminating this information is through one of several Project Fact Sheets mailed to each homeowner; an Availability Session where an open house is held that enables community members to talk one-on-one with project team members and agency personnel; and a personal letter addressed to each home or business owner that describes the installation schedule and announces the previously mentioned availability session, followed by a personal phone call to arrange the installation. After the installation is complete, each well owner will be contacted to discuss the annual maintenance requirements and answer any questions they may have.

Section VIII.E.2 of the Consent Decree requires Settling Defendants to offer to install and maintain, at their expense, point-of-entry granulated activated carbon filters, that have been approved by WDNR and WDILHR in each home and business within the Special Well Construction Zone that is located in Sections 13, 14, 15, 22, 23 or 24 or the Town of Hudson, T29N, R19W, St. Croix County, Wisconsin, and in the Norflex Inc. Facility at 720 Norflex Drive in the Town of Hudson.

To meet his requirement the Settling Defendants propose to implement a program to ensure this offer is made to each home in the area and any new construction that would be eligible for this offer. It is anticipated that the Settling Defendants and/or their agents will request to meet informally with Town of Hudson Board members to determine the best program to meet this requirement.

One of the options currently being considered by the Settling Defendants is to propose to the Town a program whereby as part of the building inspection process or as part of the issuance of an occupancy permit the Town official responsible for such inspection or permit would contact the project manager for the Trust and inform him of the new residence or business and then the Trust would send a standard form to the new owner outlining the offer under the terms as set forth in the consent decree. The owner of the residence or business would then have to indicate on the form whether or not he/she wanted a GAC and then return that form to the project manager. The filter would then be installed in accordance with the protocol that is worked out with WDNR and DILHR for GAC filter installation. Well would be sampled and owner will get a copy of the results.

As indicated, an informal discussion will need to be held with the Town to see if such a program is acceptable and also to incorporate any necessary additions into their local building/permitting ordinances or procedures as may be necessary.

It should be noted that such a program would not address any issues with respect to subsequent purchasers of the residences or businesses in the SWCA for which Settling Defendants are required to offer GAC filters. To the extent that a home or business that has a filter is sold, notices about change out of filters or maintenance, etc., will be conveyed to the new owner.

However, if a current owner declines the offer for a filter and subsequently sells the home it may be impossible for the Settling Defendants to know that there is a new owner and that another offer of a GAC filter should be made. There would be no Town official involved in a standard home sale. Therefore, the Settling Defendants propose discussing this issue with Town Board representatives informally and also potentially with local realtors, or sending a mailing to local realtors asking that they voluntarily notify Settling Defendants when a house in the SWCA defined above that does not contain a filter is sold.

Another alternative may be to see if local realtors would be receptive to keeping application forms for filters and providing them to prospective purchasers prior to closing.

The Settling Defendants will have a plan in place and submitted for WDNR approval prior to the commencement of the GAC filter installation which plan will include a standard form to send to all affected homes and businesses initially, as well as a program or plan for how to deal with new construction and transfers of property. Given the various issues involved and the necessity for Town Board input, and possibly even realtor input, a final program could not be established by the date this submittal was due.

3.0 GAC SYSTEM INSTALLATION

What about where filters already exist?

3.1 RESIDENCES WHERE FILTERS WILL BE INSTALLED

Dames & Moore will select a qualified point of entry GAC installer based on a bid process. The bid will include installation of the filter systems and any pretreatment devices required, maintenance of the systems, and used-carbon handling and disposal. The selected installer will complete an application form for each filter that will be installed (For an example, see Attachment A). The application should allow the WDNR to verify that all necessary information is provided and expedite approval of the installation. The selected installer will be authorized to proceed with the filter installation after receiving WDNR approval of the application form.

Qualifying residents and businesses will be sent a letter explaining what Dames & Moore will do and what the owner's obligation will be. Dames & Moore will conduct filter system sampling within 15 working days of system installation. This will involve collecting samples from the water supply entering and leaving the point of entry GAC filter and recording the water meter reading and the overall condition of the filter. Samples will be analyzed for VOCs.

3.2 ORGANIC WATER QUALITY

Each residence or business will have private well samples analyzed for the presence of VOCs. The results of this VOC analysis will determine which filter system is installed. The current evaluation of residences in the SWCA is shown in Table 3-1. This table details the date the sample was collected, the resulting trichloroethene and tetrachloroethene concentrations and the recommended filter system (JL-100 or JL-300). This will be updated with the private well sampling scheduled to begin the week ending September 20, 1996.

3.3 INORGANIC WATER QUALITY

Inorganic water quality data will be collected by sampling every home or business within SWCA Sections 13, 14, 15, 22, 23, and 24. These samples will be used to characterize the groundwater and determine if pretreatment of the water is necessary. The analyses required by WDNR-Water Supply include coliform bacteria, total iron and iron bacteria. In addition, every ten homes will also be sampled for hardness, alkalinity and total dissolved solids. Pretreatment requirements will be determined from the data gathered. If the well contains coliform or iron bacteria, additional involvement by WDNR-Water Supply is required.

3.3 MAINTENANCE

The selected installer will conduct carbon change out on a yearly basis or sooner if water usage at the residence/business exceeds the Wisconsin DCOMM approved capacity. The selected installer will be responsible for disposal of the used carbon. Other maintenance tasks will involve responding in a timely manner to requests from GAC users to investigate and repair leaks in pipes and fittings or to address water quality concerns, including concerns about sediment, taste or water clarity.

Dames & Moore will collect annual samples from the supply entering and leaving the GAC filter system for VOC analysis. In addition, annual monitoring of water usage will be conducted to determine if the usage falls within the recommended range for the filter size. Spot checks of at least 10 filter systems will be performed on a quarterly basis to confirm average usage rates. The residences/businesses selected will be those that are likely to have excessive water usage. If it appears that any residence/business may exceed the acceptable capacity prior to the annual carbon change, a representative from Dames & Moore will re-monitor those residences and schedule carbon changeouts as necessary.

APPENDIX E

DAMES & MOORE STANDARD OPERATING PROCEDURES

Dames & Moore Standard Operating Procedures

This appendix contains the Standard Operating Procedures (SOPs) for the anticipated activities of the pre-design investigation. For installation of gas monitoring probes, please refer to SOP #110A entitled "Gas Monitoring Probe Drilling and Construction Procedures" and SOP #210 for Photoionization Detector operation. For the clay cap analysis, please refer to SOP #320A entitled "Soil Sampling Using a JMC Backsaver." For seep sample collection, please refer to SOP #340, entitled "Surface Water Sampling" and SOP #220, #240 and #230 for pH, temperature and conductivity information. For collection of private well samples, please refer to SOP #330A entitled "Private Well Sampling" and SOP #440, entitled "VOA Sample Collection." For gas sample collection and analysis, please refer to SOP #500, 501, and 260, entitled "Air Sampling - Tedlar Bags," "Air Sampling - SUMMA Canister," and "Gas Chromatograph," respectively. Finally, SOP #410 is included for Decontamination procedures, and #470 for information on general landfill gas sampling procedures.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Gas Monitoring Probe Drilling and Construction Procedures
DATE: August 1996
SOP NUMBER: 110A Page 1 of 7

1.0 SCOPE

This operating procedure describes methods for the drilling and installation of gas monitoring probes in unconsolidated deposits in accordance with the requirements of Wisconsin Administrative NR 141, Groundwater Monitoring Well Requirements.

2.0 OBJECTIVES

In order to maintain rigid quality assurance/quality control, a number of factors must be considered while constructing a gas monitoring probe. These factors include:

- Preventing contamination from being carried from contaminated site areas to clean or less contaminated site areas; and
- Preventing surface contamination from entering the well.

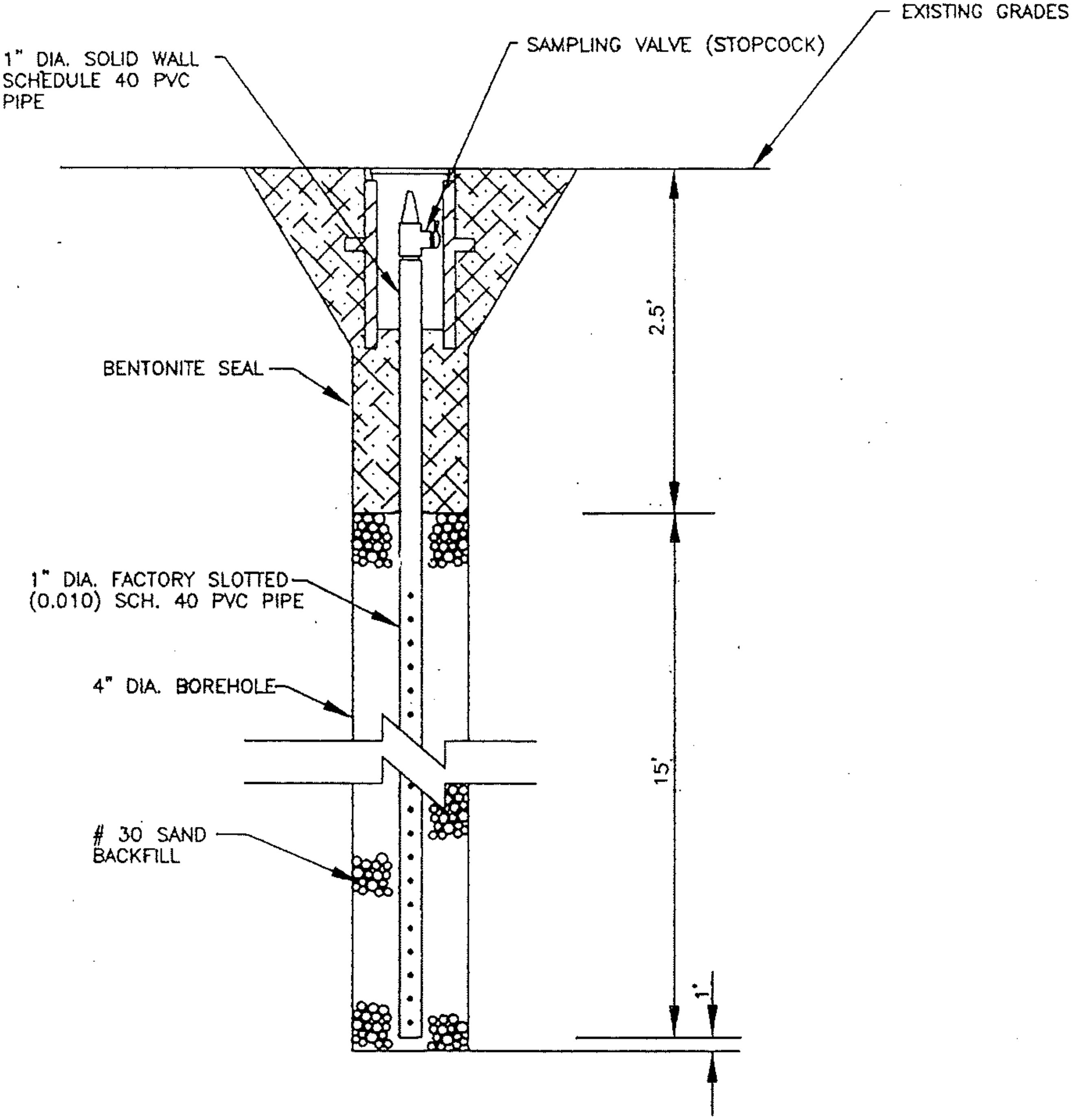
Drilling and gas monitoring probe construction procedures are described below. These procedures apply to probe construction in unconsolidated sediments. Typical monitoring probe installation and construction details in unconsolidated sediment is shown on Figure 110A-1.

3.0 OPERATING PROCEDURE

3.1 Borehole Locations

All boreholes will be horizontally located by measurements to fixed structures or reference points on the site. Stakes will be placed at each location with the correct borehole number marked on each stake. Utility clearance will be coordinated by Dames & Moore. Upon completion of the borings and monitoring probe installations, the horizontal coordinates of each probe will be located on a site grid and the ground surface and top of the PVC probe casing will be surveyed with respect to mean sea level.

Figure 110A-1: Typical Gas Monitoring Probe



3.2 Method of Drilling

Hollow stem augers will be used to advance each borehole. Mud or water rotary techniques may be employed if conditions require.

3.3 Source of Drilling Water

Potable water supply will be obtained on-site or from the local water utility.

3.4 Drilling Fluid

If hollow-stem augers are used, then no drilling fluid is required. Potable water may be used to flush out the augers as needed to collect representative samples with the split-spoon sampler or shelby tube.

If casing is installed in the borehole, potable water will be used as the drilling fluid. The water is circulated down the inside of the drill rods to lubricate the bit as it is advanced and to carry the cuttings up the outside of the rods. The casing is advanced by driving slightly behind the bit, in order to maintain the integrity of the borehole.

If conditions require the use of mud rotary techniques, then potable water and bentonite mud will be used. The bentonite will be sodium-rich montmorillonite-type material such as Volclay or Aqua Gel "Gold Seal," both Wyoming bentonites. A low density, high viscosity mud will be utilized to minimize mud loss to the formation, while maintaining the ability to remove cuttings from the borehole. If drilling fluid is being lost to the formation during drilling, the viscosity of the fluid will be increased by adding more bentonite. If the fluid loss persists, then the borehole will be cased with NW or HW flush joint casing through the zone of fluid loss. The actual mixture of bentonite and water will be determined in the field based on the performance of the mud in each individual borehole.

3.5 Cuttings

Cuttings will be screened for VOCs with a Photoionization Detector (PID); these results will be recorded and the cuttings will be placed in 55-gallon drums or other suitable containers and stored at the site for reclamation or disposal.

3.6 Formation Sampling

Samplings will be collected at the intervals provided in the site-specific work plan. Sampling will be performed using a standard spilt-spoon sampler. Samples for grain size analysis will be selected based on visual observations so as to be representative of the various stratigraphic units. Samples, best covering the spectrum of soils encountered, will be sent to a Dames & Moore geotechnical laboratory for grain size analysis (ASTM Method D-421, D-422 and D-4318) and soil classification. The remaining samples will be archived on-site or in a secured trailer.

The soils will be classified using the Unified Soil Classification System (ASTM Method D-2487-87). A description of the soil and other pertinent information regarding drilling and sampling methods, and geohydrologic data will be recorded on a boring log. Log format may be modified as required.

In areas where volatile compounds have been detected or are suspected, a photoionization detector (PID) will be used to screen samples and the reading will be recorded on the boring log (see SOP 210 for PID operation).

3.7 Diameter of Boring

For one or one and one-half-inch inside diameter probes, the minimum borehole diameter will be 7 ¼-inches, using a 3 ¼-inch I.D. hollow stem auger; or 8 ¼-inches using a 4 ¼-inch I.D. hollow stem auger.

3.8 Documentation Measurements

If groundwater is encountered, the depth to the water level in each boring will be measured just prior to construction of the well in the boring. In addition, the depth of the boring will be measured with a weighted tape to determine final depth.

3.9 Decontamination

The rotary system of the rig, including downhole equipment (drill rods, casing, samplers, bits, and hand tools), the mud tub, and the tremie pipes will be steam cleaned at a decontamination area before initiating drilling, and inspected to ensure the rig is free of leaking oil and grease. This procedure will be repeated between each borehole as needed, and at the conclusion of the drilling program. Prior to use, all downhole tools will be kept from coming in contact with the ground. Prior to being used, the drilling fluid circulation system of the rig will be flushed by circulating potable water through the system. This will be repeated between each well. Decontamination rinse water will be handled in the manner presented in SOP 410.

3.10 Abandoning Boring

Abandoned borings less than 50 feet, will be abandoned with granular bentonite. If the boring is completed more than 20 feet below groundwater or deeper than 30 feet, it will be backfilled to the surface by pressure grouting, using the cement-bentonite grout mixture specified below, using a tremie pipe lowered to the bottom of the boring.

3.11 Lubrication

Lubrication of drilling equipment (rods, sampling tools, casing) may be performed using a minimal amount of vegetable oil only. No synthetic or petroleum based lubricants will be allowed.

3.12 Gas Probe Construction

The screen depth and length is dependent on site geology and will be determined after a review of the site conditions. Monitoring of organic vapors with a PID (SOP 210) and methane with a combustible gas indicator (SOP 470) during gas probe installation will assist in determining the necessary depth and length of screen. Several of the gas probes will be installed as nested probes to distinguish the different migration pathways of landfill gases. A sand filter pack will be placed around the screen as the hollow stem auger or temporary casing is removed. The top of the screen will be a minimum of five feet below the ground surface. A minimum two feet thick bentonite seal will be placed above the sand pack. An appropriate length of riser pipe (casing) will be attached to the screen and will extend about two feet above ground. The well will be completed as described below, under "General Specifications and Procedures" and as shown on Figure 110A-1.

4.0 GENERAL SPECIFICATIONS AND PROCEDURES

4.1 Casing and Well Screen

Minimum one-inch I.D. Schedule 40 or Schedule 80 threaded flush joint, PVC casing and PVC screen will be used. No glue or screws will be used in assembling the well screen and riser casing.

4.2 Sand Pack

The filter pack will be a well sorted, silica based sand or gravel. The sand or gravel used for filter packs will be hard and durable and will have an average specific gravity of not less than 2.50. The sand and gravel will be visibly free of clay, dust and micaceous and organic matter. Not more than 5% of the sand or gravel will be soluble in a 10% hydrochloric acid solution. Thin, flat or elongated pieces of gravel, the maximum dimension of which exceeds 3 times the minimum dimension, may not constitute more than 2% of the material by weight. The filter pack for wells installed in unconsolidted material will be sized to retain at least 50% of the surrounding formation based on a sieve analysis. In formations which are predominantly silt and clay, the filter pack will be a fine sand. In bedrock, the filter pack shall be a medium or coarse sand or gravel. Crushed limestone, dolomite or any material containing clay or any other material that will adversely impact on the performance of the monitoring well may not be used as filter pack.

4.3 Screen Slot Size

The screen slot size will be selected to retain 90% of the filter pack.

4.4 Storage of Casing and Screen

The casing and screen should not be stored directly on the ground. The well casing and screens shall be assembled on racks or on clean polyethylene spread out over level ground.

4.5 Cleaning of Casing and Screen

Casing and screen shall be steam cleaned according to the decontamination procedure presented in SOP 410 before installation in the borehole.

4.6 Bottom Cap

A bottom cap shall be installed below the screen on all probe installations.

4.7 Placement of the Sand Pack

The sand pack will be placed to extend from six inches beneath the bottom of the well to two feet above the top of the well screen. This will be confirmed by measuring down the borehole annular spaced with weighted tape or with a measured small diameter pipe or rod. The sand pack will be poured directly down the annular space.

4.8 Ground Surface Seal

A ground surface seal will be constructed above the sand pack and will extend to the land surface. The ground surface seal will consist of bentonite or concrete. If bentonite is used, the top of the surface seal will terminate 6 inches below the land surface and native soil or topsoil will be placed above the bentonite to prevent drying out. The ground surface seal will be placed around the protective cover, but will not be placed between the protective cover and the well casing. The top of a concrete surface seal, or the soil above a bentonite seal, will be sloped away from the well casing.

4.9 Protective Casing

A seven-foot long section of four-inch to six-inch I.D. steel casing will be placed over the PVC probe casing. The casing will be set into the bentonite-cement grout in the annular space, and should extend above the ground no less than two and one-half feet. If necessary, the finished well will be surrounded by protective posts. The protective casing will have a lock.

4.10 Flush Mounting

In some areas, such as parking lots or roadways, wells may have to be installed flush with the ground surface so that they will not present an obstacle to other activities. In such cases, a flush-mounted protective cap will cover the completed well. A lockable water proof seal will be affixed to each well to prevent rain or other surface water from entering the well. Flush-mounted wells will not be vented and will be constructed according to the details in NR 141.13(3)(b).

5.0 WELL CONSTRUCTION DOCUMENTATION

A detailed diagram of the as-built gas monitoring probe construction specifications will be maintained during installation and development, on WDNR Form 4400-113. A boring log, WDNR Form 4400-112 shall also be completed for each probe.

6.0 WELL LABELING

The complete identification number and elevation of each monitoring well should be painted on or affixed to the protective casing or manhole cover.

7.0 SURVEYING

The elevation of the top of the monitoring probe casing (both PVC and protective steel casing) of each probe will be determined by a surveyor to ±0.01 foot, and the reference point permanently marked on the casing. Elevations will be referenced to mean sea level datum. Probe locations will be measured by surveying, by measuring tape, or by pace and compass, as specified in the project specific work plan.

TITLE: Operation/Calibration of Photoionization Detector
DATE: May 1991
SOP NUMBER: 210

1.0 SCOPE

This procedure describes methods used for the proper operation and maintenance of the photoionization detector (PID). Manufacturer's specifications and recommendations should be followed during operation of the PID.

2.0 OBJECTIVES

The activities covered by this procedure:

- Improve quality control in field methods used in operation.
- Improve the consistency of results.

3.0 OPERATING PROCEDURE

The PID is primarily used for safety and survey monitoring of ambient air. The PID is a non-specific vapor/gas detector. The photoionization detector consists of an ultraviolet (UV) lamp, two electrodes connected to an electrometer, and a small pump which continuously draws air into the ionization chamber. All organic and inorganic vapor/gas compounds having ionization potentials (IP) lower than the energy output of the lamp are ionized, which is detected by the electrometer and indicated on the liquid crystal display.

4.0 EQUIPMENT NEEDED

1. PID (Photovac Microtip);
2. Calibration gas (100 ppm Isobutylene); and
3. Operation manual and accessories.

5.0 OPERATION

1. Personnel responsible for using the PID must first read the user's manual. The user's manual for a Photovac MICROTIP PID will be retained in the instrument's storage case. If a different make or model PID is used, the user's manual must be obtained, read, and taken to the site.

2. All operation of the PID will be in accordance with the user's manual.

3. Preliminary steps such as battery charging, calibration, and maintenance should be conducted in a controlled environment.

4. At the beginning of each day, calibrate the PID making necessary adjustments. Record the calibration information in the field log book and/or on data sheets.

5. Readings should be taken on the lowest possible scale and recorded in the field log book and/or on data sheets.

6. At the end of each day, recheck the calibration and record the information in the field log book and/or on data sheets.

6.0 DOCUMENTATION

Safety and survey monitoring with the PID will be documented in the field log book and/or on data sheets. The following information is to be recorded:

- Project name and number;
- Operator's signature;
- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument response verifications - magic marker or similar test.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE:	pH Meter	
DATE:	February 1994	
SOP NUMBER:	220	Page 1 of 4

1.0 SCOPE

This operating procedure describes the operation, calibration, and maintenance of pH Meter and its accessories for use in the field. Manufacturer's specifications and recommendations should be followed or referred to as and when need arises.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure quality control in field pH measurement.
- Insure uniformity and continuity in operation, calibration, and maintenance of both the equipment and measuring techniques by different qualified field analysts or technicians.

3.0 EQUIPMENT NEEDED

- pH meter[a] and its accompanying electrode or probe.
- Buffer solutions of known pH (4.0, 7.0, and 10.0).
- Plastic or glass beakers or cups (at least 20 mL volume).
- Distilled or deionized water.
- Polyethylene spray bottle.
- Waterproof marking pen or pencil.
- Liquid waste container.
- Thermometer.
- Trash receptacle.
- User's manual for pH meter.

[a] Portable pH meters available with Dames & Moore for use in field include:
- Myron L pDS Meter - Model EP11/pH;
- Orion pH Meter - Model 230A;
- CSI pH/Temperature/Conductivity Tester - Catalog No. 301353;
- Orion pH Meter - Model 407A;
- Hydrolab pH Conductivity; and
- Omega pH and Conductivity Pens.

4.0 PRELIMINARY TO OPERATION

At the start of each field day, the pH meter should be examined for cleanliness, and checked for defects, and any possible need of repair. The checks should include whether the battery and electrode are operable. The meter should also be calibrated at the start of each day, with intermittent calibration checks throughout the day to determine whether recalibration is necessary. The following procedures should be performed at the start of each field day:

- **Battery check** to determine if battery is functional to full scale. Batteries are replaced if found weak.

- **Electrode check** in accordance with the user's manual from the manufacturer. If the electrode check indicates potential problems with the electrode, a different electrode and/or pH meter must be obtained or the electrode must be repaired before going into the field.

- **Meter calibration** in accordance with the user's manual from the manufacturer. Calibration schedule should include daily calibration, and intermittently, when required, during continuous use of the meter. Instrument calibration consists of calibration of the pH meter with pH 7 and pH 10 buffers, and a pH 4 buffer as a check, or with pH 7 and pH 4 buffers, and a pH 10 buffer as a check, depending on the average expected pH values of the samples.

  The calibration for pH is temperature correlated. Please note the **actual** pH of your buffers at the temperature used for calibration. (A chart for this is usually provided on the buffer container.) If the pH meter does not have automatic temperature compensation, you may need to calibrate the 7 buffer to 6.95 or 7.03, or some point in between, depending on the temperature of your buffers. Some pH meters compensate for temperature, but require the user to set a temperature knob on the meter to the measured value. Refer to the user's manual for the pH meter to determine what temperature compensation features the meter has, if any, and follow the meter-specific instructions.

  Calibration should be accomplished through the following steps:

  1. Place the electrode in the pH 7 buffer solution and adjust the meter to read 7.0, or the appropriate value given on the buffer container.

  2. Rinse the electrode with deionized water.

  3. Place the electrode in the pH 4 or pH 10 buffer and adjust the meter slope until the meter reads the appropriate value.

  4. Rinse the electrode with deionized water.

5. Place the electrode in the pH 4 or pH 10 buffer, whichever was not used in Step 3, and read the pH of the check buffer. If the value is not within 0.1 pH unit of the expected value, repeat the calibration procedure. If the meter cannot be successfully calibrated on several successive attempts, another meter should be used or the meter should be repaired prior to use.

6. Record calibration information in the field logbook or on a calibration data sheet (Figure 220-1, or equivalent). Note any problems encountered during calibration.

- Scheduled maintenance will include daily checks by Dames & Moore trained personnel according to procedures provided by the equipment's manufacturer.
- Repairs will be performed by an authorized service representative.

5.0 OPERATING PROCEDURE

1. Turn on power and allow meter to stabilize for about three to five minutes. Caution: Do not leave or use meter in direct sunlight or cold wind.

2. Measure pH of unknown solution in accordance with user's manual. If the pH meter does not have automatic temperature compensation, the temperature of the sample must also be measured and recorded. On some pH meters, it may be necessary to adjust a temperature dial to the temperature of the sample. The meter will then compensate for the sample temperature and report the pH under standard conditions.

3. Record the pH reading and sample temperature in the field logbook or on a data sheet and note whether the pH given is compensated to standard conditions (25°C) or at the temperature of the sample.

4. Obtain one duplicate field measurement for every 20 measurements performed. Initial measurement and duplicate measurement should be within 20 percent.

5. Quality assurance objectives of pH measurement based on EPA Method 150.1, Electronic Measurement of pH (USEPA, 1983)[a] should consist of: Precision (standard deviation): ±0.1 pH unit accuracy determined, based on instrument manufacturer's specific value.

[a] USEPA 1983. Methods for chemical analysis of water and wastes. Environmental Monitoring and Support Laboratory, Office of Research and Development. EPA-600/4-79-020. U.S. Environmental Protection Agency, Cincinnati, Ohio.

Figure 220-1

INSTRUMENT CALIBRATION LOG

INSTRUMENT:			
MANUFACTURER:			
MODEL NUMBER:			
SERIAL NUMBER:			
DAMES & MOORE ASSET NUMBER:			
DATE ACQUIRED OR SERVICED:			
ORIGINAL OR PREVIOUS CALIBRATION DATE:			
CALIBRATED BY:			
NOTES ON ORIGINAL OR PREVIOUS CALIBRATION:			
CALIBRATION SCHEDULE: (circle one)	DAILY	MONTHLY	YEARLY
MAINTENANCE SCHEDULE: (circle one)	DAILY	MONTHLY	YEARLY
CURRENT CALIBRATION RECORD:			
DATE:	TIME:		
CALIBRATION STANDARD(S) USED:			
CONCENTRATION(S):			
PROCEDURE (describe briefly):			
DEFICIENCIES: (if any):			
CALIBRATION PLOTS OR GRAPHS (attach, if any)			

SIGNATURES:

Technician:____________________

Date: __________ Laboratory Manager: ____________________

SYSTEMS OR PERFORMANCE AUDIT:

AUDIT REPORT:

SIGNATURE:

Auditor: ____________________ Date:____________________

QA Manager: ____________________ Date: ____________________

TITLE:	Conductivity Meter	
DATE:	February 1994	
SOP NUMBER:	230	Page 1 of 3

1.0 SCOPE

This procedure describes the operation, calibration, and maintenance of conductivity meters for use in the field sampling activities. Manufacturer's specifications and recommendations for the specific conductivity meter used should also be followed. Project-specific quality assurance objectives may indicate calibration schedules and/or criteria which override those provided in the SOP.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure quality control in field conductivity measurement.
- Insure uniformity and continuity in operation, calibration, and maintenance of both the equipment and measuring techniques by different qualified field analysts or technicians.
- Provide semi-quantitative data for use in determining relative variations in conductivity between two or more water (surface water and/or groundwater) samples.
- Indirectly serve as a means to evaluate the water quality at the time of sampling. It is imperative that temperature compensation is made because conductivity measurements are very sensitive to the temperature of the solution being measured.

3.0 EQUIPMENT NEEDED

- Conductivity meter[a].
- Reference solutions.
- Thermometer.
- Plastic cup or beaker (at least 20 mL volume).
- Distilled water.
- Polyethylene wash bottle.
- Trash receptacle.
- User's manual for conductivity meter.

[a] Portable conductivity meters available with Dames & Moore for use in field include:
- Myron L pDS Meter - Model EP11/pH;
- CSI pH/Temperature/Conductivity Tester - Catalog No. 301353;
- Omega pH and Conductivity Pens; and
- Extech Conductivity and Temperature Meter.

4.0 PRELIMINARY TO OPERATION

1. Examine the conductivity meter for cleanliness, defects, and any possible need of repair. Check the battery and conductivity probe or cell for proper function.

2. Calibrate the conductivity meter in accordance with the user's manual provided by the manufacturer. Calibration of the specific conductivity meter should be made with a standard of approximately the same conductivity as those expected at the site, and should be measured at (or converted to) 25°C. Some conductivity meters automatically compensate for temperature, some compensate after the user adjusts a temperature knob on the meter to the measured temperature, and others have no temperature compensation feature. Refer to the user's manual to determine what temperature compensation features the conductivity meter has and follow the directions. The calibration of the field instruments must be checked every four hours and at the end of the day. If the calibration check is not within ±5% of the expected value, the meter must be recalibrated.

   Record calibration information in the field logbook or on an instrument calibration data sheet (Figure 230-1, or equivalent).

3. Scheduled maintenance will include daily checks by Dames & Moore trained personnel according to procedures provided by the equipment's manufacturer.

4. Repairs will be performed by authorized service representative.

5.0 OPERATING PROCEDURE

1. Measure conductivity and temperature of sample following manufacturer's instructions.

2. Remove probe from sample solution and rinse it thoroughly in deionized waste before proceeding to measure next samples or putting away the equipment.

3. Record data in field logbook or on data sheets.

4. Obtain one duplicate field measurement for every 20 measurements performed. Initial measurement and duplicate measurement should be within 20 percent.

Figure 230-1

INSTRUMENT CALIBRATION LOG

INSTRUMENT:

MANUFACTURER:

MODEL NUMBER:

SERIAL NUMBER:

DAMES & MOORE ASSET NUMBER:

DATE ACQUIRED OR SERVICED:

ORIGINAL OR PREVIOUS CALIBRATION DATE:

CALIBRATED BY:

NOTES ON ORIGINAL OR PREVIOUS CALIBRATION:

CALIBRATION SCHEDULE: (circle one) DAILY MONTHLY YEARLY

MAINTENANCE SCHEDULE: (circle one) DAILY MONTHLY YEARLY

CURRENT CALIBRATION RECORD:

DATE: TIME:

CALIBRATION STANDARD(S) USED:

CONCENTRATION(S):

PROCEDURE (describe briefly):

DEFICIENCIES: (if any):

CALIBRATION PLOTS OR GRAPHS (attach, if any)

SIGNATURES:

Technician: ______________________________

Date: ______________ Laboratory Manager: ______________________________

SYSTEMS OR PERFORMANCE AUDIT:

AUDIT REPORT:

SIGNATURE:

Auditor: ______________________________ Date: ______________

QA Manager: ______________________________ Date: ______________

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Thermometer
DATE: February 1994
SOP NUMBER: 240

1.0 SCOPE

This operating procedure describes the operation, calibration, and maintenance of a thermometer and its accessories for use in the field. Manufacturer's specifications and recommendations for the specific thermometer used should also be followed.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure quality control in field temperature measurement.
- Insure uniformity and continuity in operation, calibration, and maintenance of both the equipment and measuring techniques by different qualified field analysts or technicians.
- Provide an accurate means of compensating pH and conductivity measurements to standard conditions.

3.0 EQUIPMENT NEEDED

- Thermometer[a] with mercury bulb, or probe in modern meters.
- Field log.
- Sample container.
- Rinse Water.

[a] Thermometers available with Dames & Moore for use in the field, such as:
- Temperature Indicator MyCal Model SA-754-B;
- CSI pH/Temperature/Conductivity Tester - Catalog No. 301353; and
- Extech Conductivity and Temperature Meter.

4.0 PRELIMINARY TO OPERATION

1. Check the thermometer for accuracy before each sampling event against an NBS calibrated thermometer.
2. Record the calibration check in the field logbook or on data sheets.

3. For electronic temperature meters, check the battery.

5.0 OPERATING PROCEDURE

5.1 Temperature Meters

1. Turn the meter on.
2. Plug the jacks on the probe into the thermometer.
3. Lower the probe into the sample and record measured temperature in field logbook or on the sampling data sheet.
4. Decontaminate probe when finished.
5. Obtain one duplicate field measurement for every 20 measurements performed. Initial measurement and duplicate measurement should be within 20 percent.

5.2 Bulb Thermometers

1. Lower bulb into sample and allow to equilibrate.
2. Record measured temperature in field logbook or on sampling data sheet.
3. Decontaminate thermometer when finished.
4. Obtain one duplicate field measurement for every 20 measurements performed. Initial measurement and duplicate measurement should be within 20 percent.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Gas Chromatograph
DATE: August 1996
SOP NUMBER: 260 Page 1 of 6

1.0 SCOPE

This operating procedure describes the operation and maintenance of the Photovac 10S gas chromatograph (GC) for use in the field. Manufacturer's specifications and recommendations should be followed or referred to as and when need arises.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure quality control in field GC analyses.
- Insure uniformity and continuity in operation, calibration, and maintenance of both the equipment and measuring techniques by different qualified field analysts or technicians.

3.0 EQUIPMENT NEEDED

- GC and its accompanying accessories.
- Carrier gas.
- Syringe.
- Calibration gas.
- User's manual for GC.

Read all the instructions before using the instrument. Refer to the owner's manual for a list of other trouble shootings and problems, additional maintenance and repair information, as well as for a more detailed description of the equipment and its correct operation.

4.0 PRELIMINARY TO OPERATION

1. Locate the unit in an area with low-traffic and where the temperature remains fairly constant. Peak retention times are inversely proportional to temperature (i.e., retention times get shorter as the environment gets hotter) for which reason the instrument should be located inside a shelter where there could be some degree of temperature control. Avoid exposing the instrument to rain, snow or dust. Never operate the instrument outdoors in a misty or very humid day.

2. The carrier gas recommended is "Air Ultra-Zero" or "Air Zero-Zero"; this must contain less than 0.1 ppm of total hydrocarbon contamination. "Zero" grade air is not recommended.

3. Manual injection of small samples is done using a gas tight syringe (2-inch maximum side port needle).

4. Never turn the instrument "ON" without a flow of carrier gas. It could cause damage to the lamp.

5. If operating the instrument from an external carrier gas cylinder, use a high purity, 2-stage regulator that can reduce the pressure to 40 psi (280 kPa). The connection between the cylinder regulator and the instrument should be very clean and made of Teflon or stainless steel. This transfer line should be ⅛-inch diameter. Never use rubber or soft plastic tubing.

6. If using the automatic mode of operation, follow the next guidelines:

   a) If calibrant is toxic, connect the *PUMP OUT* port to a vented line. Also, purge the calibrant line before connecting.

   b) If using an external pressurized tank to supply the calibration gas, it should be equipped with a reducing regulator to deliver at 5 psi (35 kPa). The delivery line should also be Teflon or stainless steel.

7. Be very careful not to change the settings of any valve, specially of the gas fittings on the instrument top panel (*DETECTOR OUT, AUX OUT*) unless adjustment of the flow is necessary. Refer to the *START-UP AND OPERATION* section for more information.

8. The injection ports are fitted with "Septum" which is used to prevent leakage. The septum is reached by unscrewing the black "Septum Retainers" in the instrument top panel. The septum Teflon side should be down when inserting them into the retainers. The septum must be changed every 25-30 injections. Do not overtighten the retainers because the needle becomes blocked with a core of septum material.

9. Allow 30 minutes for warmup time after the initial switch on of the power before doing any calibration of the instrument.

10. When not using a particular fitting, wrap it in a clean plastic bag or aluminum foil and set aside in a safe place because it could cause contamination of the instrument. The instrument should also be covered with its lid and be closed when not in use.

11. Always disconnect the pressure device when not using the instrument.

12. Concentration range is from 0.001 ppb to 9999 ppm. Below 0.001 ppb, the instrument will print as 0.0000 ppb. Above 9999 ppm will appear as ****.

13. This instrument in particular does not detect compounds with ionization potential greater than eleven (11).

14. Never inject a sample that contains moisture in it.

15. Fill the internal tank of the instrument up to 1200 psi for one day of use. Delivery pressure should not exceed 40 psi.

16. The instrument battery lasts for eight hours. Recharge the battery the same amount of hours it was used.

5.0 MAINTENANCE

1. Routine maintenance of the instrument consists of monitoring the consumable air carrier gas and replacing it periodically.

2. Shut the unit down for 20 minutes after changing carrier gas.

3. Change paper and pens on the printer/plotter as needed.

4. The lamp will also require periodic replacement depending upon the sampling frequency. Refer to the owner's manual for specific details.

5. The septum in the injection ports must be changed every 25-30 injections.

6.0 START-UP AND OPERATION

1. If operating the instrument from an external gas cylinder, connect the cylinder to the *EXTERNAL CARRIER IN* fitting. If operating the instrument from its own internal carrier gas reservoir, check the gauge marked *DELIVERY* located at the rear left on the instrument. This gauge shows the pressure at which the carrier gas is being delivered and should always read 40 psi.

2. Connect the instrument to the main electrical supply or to an external battery providing a maximum of 14 volts.

3. Establish a supply of carrier gas.

4. Use a 2-channel flowmeter (0-50 mL/min) to compare the readings with the Custom Specification Sheet, located in the clear plastic folder in the front of the owner's manual, using the following procedure:

- Connect the left channel to *DETECTOR OUT* and the right channel to the *AUX OUT* needle valve.

- Compare the readings (both flows should be equal and should be between 10-15 mL/min).

- Adjust the readings if necessary:

    - the left channel is adjusted via the RED color coded valve;

    - the right channel is adjusted using the small needle valve attached to the *AUX OUT* port (these valves interact so adjustments need to be iterative);

    - if the readings are not the same, adjust the *AUX OUT* needle valve until they are equal. Turn the valve handle clockwise to lower the right channel and to raise the left channel. Turn it counterclockwise to raise the right and lower the left. Wait 10 seconds or so for the flow to stabilize;

    - once both flows are equal they can be raised or lowered together by using the *RED FLOW* valve at the left on the top panel. Turn it clockwise to lower both flows or counterclockwise to raise both flows to 15 mL/min. Stabilization will take 20 seconds or so;

    - leave the meter in place and continue until the readings are the same.

- Allow the instrument to stabilize for 20 minutes after all adjustments are completed.

5. If using the isothermal GC capillary column oven assembly, connect the power supply to the 10S *EXT DC* top panel receptacle.

    - Open the module to check the oven unit.

    - Check the temperature setting. The corresponding light will be ON if it is selected. Change to the desired temperature by turning the switch.

6. Depress the *ON* key, you should see "LAMP NOT READY PLEASE WAIT". Wait 2-3 minutes until "READY ENTER COMMAND" appears. Let the instrument warmup for 30 minutes.

7. Depress the *TEST* key follow by *ENTER*. A Status Report will be printed. Check all the instrument setup parameters with the Custom Specification Sheet. The headings "Field" and "Power" should be within 20% of the setup, the other numbers should correspond <u>exactly</u> with the Specification Sheet.

8. Press the *LIST* key. Select the library needed and press *ENTER* for a list of all the compounds stored in it. There are four separate libraries available within the computer. These libraries are for storing information entered with the "setup" group and with the *CYCLE*, *EVENT*, *INFO*, and *CAL* keys.

9. Depress the *GAIN* key to set the gain. Press the up arrow key until the gain number correspond with the Specification Sheet and press *ENTER*.

10. Depress the *CYCLE* key. Check the "TIMER DELAY", "ANALYSIS TIME", and "CYCLE TIME" settings with the Specification Sheet. Use the *CLEAR* key and the numerical key pad to make any changes, otherwise press *ENTER*.

11. Depress the *USE* key. Select the library to be used and hit *ENTER*. Type the day number and press *ENTER*. Follow the same procedure for the month, year, hour and minute. Note that the instrument uses a 24-hour clock.

7.0 CALIBRATION AND ANALYSIS

1. If using the automatic mode, connect the supply of calibration gas. The delivery line should be connected to the *CAL IN* port. If calibrant is toxic, connect the *PUMP OUT* port to a vented line. Also, purge the calibrant line before connecting.

   The calibration gas, supplied by Scotty Specialty Gases of Troy, Michigan, will contain the following compounds:

   acetone
   benzene
   chloroethane
   cyclohexane
   cis-1,2 dichloroethene
   ethylbenzene
   4-ethyltoluene
   freon 11
   freon 12
   heptane
   hexane
   methyl chloride
   tetrahydrofuran
   tetrachloroethene
   1,3,5-trimethylbenzene
   1,2,4-trimethylbenzene
   toluene
   trichloroethene
   vinyl chloride
   m-xylene
   o-xylene

2. Press the *INFO* key to enter information to print with each analysis.

3. To see how the results are affected if a parameter is changed after an analysis is done, press the *CAL* key and *ENTER* to relist the information.

4. To store compounds in a library for calibration purposes, press the *STORE* key. Select the peak number corresponding to the chromatogram to be identified and press *ENTER*. Type the name of the compound, the concentration in ppm and its limit value in ppm. The purpose of the limit value is to flag the result by printing in red when this value is exceeded.

5. Use the *EDIT* key if the spelling of a compound or its limit value needs to be changed. Enter the ID number from the library printout. To delete a compound, press the *CLEAR* key and *ENTER* after entering the ID number and pressing *ENTER*.

6. To update retention times due to changes in the instrument temperature, inject a sample of your calibrant gas. After the analysis is done, press the *CAL* key. Type the plotter peak to relist and the ID number in the library. For retention time update only, the concentration in ppm must be zero. For recalibration type the concentration of your standard and hit *ENTER*.

7. The volume of the sample injected is varied by changing the difference between "EVENT 4 OFF" time and "EVENT 5 ON" time. Refer to the owner's manual for additional information and specific instructions. Volumes normally lie in the 200 to 200 μL range.

8. The operating conditions of this instrument depend on the constituents being analyzed for. Oven temperatures range from 30 to 50 degrees Celsius. The temperature most frequently used is 30 degrees Celsius. Flow rates through the column range from 5 μL/s to 20 μL/s. Typical flow rates are 10 μL/s to 15 μL/s.

8. Pump the syringe about 10 times to ensure it is flushed with the mixture, then withdraw a syringe full from the bag or container. Depress the plunger until the desired volume remains in the syringe.

9. Confirmation that the instrument has been properly calibrated is performed by running a standard that contains the constituents the instrument has been standardized for. A single standard is used for this. The results of the analysis of the standard are compared to the known concentrations of the standard. If the two do not agree, the instrument is recalibrated for the constituents that were not a match.

10. To start an analysis, press the *START-STOP* key, select "PROBE IN" and press *ENTER*. Hold the syringe barrel in one hand and guide the needle down the hole in the center of the "Manual Injection Port 1" until resistance as the needle point touches the rubber septum is felt. Get ready with the other hand to push the plunger down. Injection should be made immediately following the two-second buzz.

11. An appropriate standard is run to confirm that the instrument is still properly calibrated after every 10-15 analyses, or as needed. More frequent calibration may be necessary if contamination is high. There are no laboratory blanks used with this instrument.

12. The analyses of this instrument are not validated. The should be used for qualitative analysis.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Soil Sampling via the JMC Backsaver
DATE: August 7, 1996
SOP NUMBER: 320A

1.0 SCOPE

This operating procedure describes the procedures needed for collecting relatively undisturbed soil cores using a JMC Backsaver soil probe. Under ideal circumstances a five foot penetration depth can be obtained in less than one minute.

2.0 OBJECTIVES

The activities covered by this procedure:

- Ensure that safe operation are performed in the field.
- Ensure that the sample collected is both undisturbed and readily identifiable upon retrieval from the subsurface.
- Provides a higher level of certainty in differentiating soil materials and the depth at which they are encountered when compared to soil augering.

3.0 EQUIPMENT NEEDED

- JMC Backsaver equipped with an 18-inch "wet" tube (if soil conditions are moist).
- Pocket measuring tape.
- Cleaner.

4.0 PRELIMINARY OPERATION

As the name implies, the JMC Backsaver is an ergonomically-designed piece of soil sampling equipment that allows one to collect and retrieve soil samples while maintaining an upright standing position. This minimizes the stress on the lower back. Use caution when cleaning out the tip of the soil tube as the edges are extremely sharp.

5.0 OPERATING PROCEDURE

1. Place the fully assembled JMC Backsaver in an upright position that resembles an upper cast "T".

2. To advance the soil sample tube into the soil, place your foot on the foot pedal and press the tube into the ground by raising your foot, then stepping down on the spring-loaded foot pedal in order to place it in the 3:00 position.

3. To retrieve the sample, pull on the T-bar handle (the foot pedal is in the 2:00 position) lifting and keeping the cut away section of the tube facing up so that the soil core does not fall out of the tube.

4. Remove the soil from the tube by rolling the tube so the soil core drops out. If the soil is wet and the soil clings to the tube, then use the soil cleaning device provided.

5. Reinsert the soil tube into the hole advancing it to its previous depth. Gently press the foot pedal into a 2:30 position while simultaneously pulling up on the T-bar handle. This extends the pushrod. The depth of insertion can be determined by measuring the height of the top of the rod from the ground surface and taking the difference between this measurement and the total length of the tube and rod.

6. Again place your foot on the foot pedal and depress it to the 3:00 position and push down to advance the tube.

7. Raise the tube by first pulling on the T-bar handle and then pushing down on the T-bar handle, sliding the casing downward until a comfortable retrieval position is obtained.

8. Repeat steps 4 to 7 as needed until the tube and rod are fully extended or the necessary subsurface soil information is obtained.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Private Well Sampling
DATE: August 1996
SOP NUMBER: 330A Page 1 of 3

1.0 SCOPE

This operating procedure describes steps involved in private well purging and preparation for taking groundwater samples.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure that the groundwater samples taken will be representative of actual groundwater quality.
- Insure quality control and consistency in taking samples.
- Serve as a means to allow traceability of error(s) in sampling and data recording.

3.0 EQUIPMENT NEEDED

- A garden hose for purging well.
- pH meter, conductivity meter, and thermometer.
- A field log.
- Spare batteries for field instruments.

4.0 PRELIMINARY TO OPERATION

1. Review project work plan for site-specific sampling requirements and procedures.
2. The thermometer, pH and conductivity meters should be cleaned, checked for defects, and any possible need for repair.
3. Batteries should be checked in the pH meter (SOP 220) and conductivity meter (SOP 230).

5.0 OPERATING PROCEDURE

1. Connect the garden hose to an outside tap.

2. Purge the water supply for a minimum of 10 minutes.

3. Locate the sampling tap near the pressure tank. This tap should be prior to any treatment device. It is important that an **untreated** groundwater sample be collected.

4. Record the private well residential address, time, and date and all pertinent information and data on groundwater sampling record (Figure 330A.1, or other data sheet or field logbook).

5. Collect private well water sample in appropriate vial for organic or inorganic analyses.

6. Affix labels to each sample bottle recording sample number, well location, date, and time.

7. Record sample information on sampling record or in field log, along with a description of the physical appearance of the sample, including color, odor, and turbidity.

8. Place samples immediately in a shipping container maintained at 4°C.

need unique well #

FIGURE 330A.1

PRIVATE WELL WATER SAMPLING RECORD

PROJECT: ____________________ **SAMPLE OR LOG NO.:** ______________
LOCATION: ____________________ **SAMPLER:** ________________________
DATE (Mo/Dy/Yr): ______________ **TIME: Start** _________ **End** _________
RESIDENTIAL LOCATION: ____

SAMPLE METHOD

Grab Bail Other (describe)

WATER QUALITY OBSERVATIONS

SAMPLING POINT: ______________________________
WATER COLOR/CLARITY: ______________________________
COMMENTS: ______________________________

FIELD TESTING	INITIAL	ADDITIONAL TESTING			
pH					
Conductivity					
Temperature					

NUMBER OF CONTAINERS	ANALYSIS REQUIRED	PRESERVATIVE
____________ ____________ ____________ ____________	____________ ____________ ____________ ____________	____________ ____________ ____________ ____________

SAMPLER'S SIGNATURE: **DATE:** / /

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Surface Water Sampling
DATE: February 1994
SOP NUMBER: 340 Page 1 of 3

1.0 SCOPE

This procedure describes methods used for the collection of water quality samples from rivers, lakes, and ponds.

2.0 OBJECTIVES

The activities covered by this procedure:

- Improve quality control in field methods used to obtain water samples.
- Improve consistency of water sample quality.

3.0 EQUIPMENT NEEDED

- Project-specific sampling plan and site area maps.
- Sample containers and preservatives (provided by lab), labels, insulated containers (e.g., coolers) for sample storage, and an ample supply of ice.
- Field equipment as specified in the sampling program and applicable calibration standards.
- Alpha horizontal type sample collector.
- Boat (for deep rivers, lakes, and ponds) or waders.
- Weighted tape measure, rigid gage, or depth sounder.
- Field-data sheets and/or log book.
- First aid kit.

4.0 PRELIMINARY TO OPERATION

1. Check that pH meter, conductivity meter, and thermometer are functioning and calibrated properly (See Dames & Moore SOPs 220, 230, and 240).

2. Select sample location: The selection of the precise sampling location requires professional judgement and an understanding of the purpose of the study. Sampling locations where mixing is incomplete should be avoided if an average composition is required. Often, areas of poor lateral or vertical mixing can be visually identified. For example, color or turbidity differences may be apparent immediately below the confluence of a tributary and the main river or at wastewater

discharge point. Use of a field conductivity meter is recommended for determining the uniformity of the water composition across the width and depth of the water body.

3. Record a description of the area from which the sample is taken so that others can identify the sampling location. This may be done by detailed description, maps or through the use of stakes, buoys, compass bearings, or other landmarks.

4. Ensure the sampling device has been decontaminated and rinsed with deionized water prior to each sample.

5.0 OPERATING PROCEDURE

5.1 Stream Sampling

1. Unless otherwise indicated in the specific project sampling plan, samples should be collected from the center of the stream, or the deepest flow channel of the stream, at mid-depth. This will insure an average to above average flow of water with minimal settling of solids.

2. In shallow streams which can be traversed safely on foot, the sample bottle can be filled directly with the flowing water unless the sample bottle is preserved. All sample bottles containing preservatives must be filled from a sample transfer container.

3. If the sample is used to collect the sample, the bottle should be lowered into the water while still capped, uncapped under water to allow the sample bottle to fill, and then recapped before removing from the water. The mouth of the sample bottle should be facing into the flow of the water or facing the water surface. Always face upstream when sampling in the water itself, and do not touch the mouth of the bottle with your hands or other potential contaminants. VOA sample collection should be performed in accordance with Section 4 of SOP 440, VOA Sample Collection.

4. In deep streams, a boat will usually be required to be used in order to obtain a representative sample. The depth can be determined using a depth sounder or by physical measurement with a heavily weighted flexible measuring tape or rigid gage.

5. An Alpha horizontal type sampler or a Bacon-Bomb type sampler should be used for collection of samples at a specific depth in the water column. The sampler should be lowered into the water to the appropriate depth, opened to allow for the collection of the sample, closed and removed from the water.

5.2 Lake and Pond Sampling

1. Water in lakes and ponds is generally poorly mixed and thermal stratification is frequently observed. Single samples can only represent the specific spot from which they are taken. Often samples are taken at the inlet and/or outlet of the lake or pond. Sometimes a grid is established if the lake or pond and samples are collected at grid line intersections.

2. An Alpha horizontal type sampler or a Bacon-Bomb type sampler should be used for collection of samples at a specific depth in the water column. The sampler should be lowered into the water to the appropriate depth, opened to allow for the collection of the sample, closed, and removed from the water.

5.3 Sample Handling and Documentation

1. Transfer the sample into appropriate sample bottle(s) directly from the sample container and preserve accordingly. Preservatives may be added to the sample bottles in the field after filling, or the containers can be pre-spiked with the preservative. Check the pH of each sample preserved using the proper range pH paper to ensure that the sample is properly preserved.

2. Secure the container lid tightly and label the sample bottle with appropriate information. Be sure to label the tag carefully and clearly, addressing all the categories or parameters. Complete all chain-of-custody documents, and record in the field logbook.

3. Once a sample is obtained, pH, specific conductivity, and temperature measurements should be made following the appropriate SOPs.

4. All samples should be placed in an appropriate carrying container and iced immediately after collection and should remain iced until delivery to the analytical laboratory.

5. Surface water samples should not be filtered unless specifically requested by the laboratory or required by the work plan.

6. A record of all samples collection must be kept. All sample bottles must be clearly marked using a permanent marker and all completed labels should be wrapped in clear tape to help waterproof. The field record must provide positive sample identification as well as the name of the sample collector, the date, time, and exact location of each sample collection point, and results of all field water quality measurements. Other information such as weather and stream-flow conditions should also be noted. Project-specific data sheets or log books may be used.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Decontamination Procedures
DATE: January 1994
SOP NUMBER: 410 Page 1 of 2

1.0 SCOPE

This operating procedure describes procedures used to decontaminate equipment used during environmental sampling of hazardous waste sites. Project-specific quality assurance objectives, provided in the project work plan and/or quality assurance plan, may override some of the procedure specified in the SOP.

2.0 OBJECTIVES

The activities covered by this procedure:

- Prevent cross-contamination between samples.
- Insure quality control in decontamination of field equipment used in sampling and handling environmental samples.
- Help to maintain a clean working environment for the safety of field personnel.
- Serve as a means to allow traceability of errors in procedures.

3.0 EQUIPMENT NEEDED

- Tap water and distilled water or deionized water.
- Personal safety gear (specified in project Health and Safety Plan).
- Sprayer.
- Five-gallon stainless steel pail and plastic buckets.
- Detergent (Alconox).
- Nylon scrub brush and long handled bottle brush.
- Aluminum foil and paper towels.
- Trash receptacle.

4.0 PROCEDURE

1. Select an area of the site removed from sampling locations. If it can be determined, the area should be downgradient from wells being sampled.

2. Fill a 5-gallon pressurized sprayer or smaller squirt bottle with distilled water.

3. Wash all grit, grime, mud, particulates, etc., from the equipment being decontaminated with tap water and collect in a plastic bucket.

4. Put one gallon of distilled water into a 5-gallon stainless steel pail and add 1-cup of detergent.

5. Wash equipment in the pail using a nylon scrub brush or long handled bottle brush.

6. Rinse all residual detergent from the equipment with the sprayer and collect rinsate.

7. Repeat steps 5 and 6 as necessary.

8. Rinse the equipment thoroughly with organic free distilled water and collect the fluid in a plastic bucket.

9. Dry and then wrap the equipment securely in aluminum foil or polyethylene sheeting.

10. Dispose of soiled materials and fluids in designated disposal containers in accordance with the project-specific plan.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: VOA Sample Collection - 40 mL vials
DATE: January 1994
SOP NUMBER: 440 Page 1 of 2

1.0 SCOPE

This procedure describes the water and surface water sample collection into 40 mL vials.

2.0 OBJECTIVES

The activities covered by this procedure:

- Insure quality control in field sampling of vials for volatile organic analysis (VOA).
- Insure uniformity and continuity in sampling techniques and use of the equipment by different qualified field samplers or technicians.
- Serve as a means to allow traceability of error(s) in sampling.

3.0 EQUIPMENT NEEDED

- Two 40 mL vials.
- Labels.
- Distilled or deionized water.
- Waterproof marking pen or pencil.

4.0 PROCEDURE

Water and surface water sample collection for VOA into 40 mL vials consists of the following steps:

1. Remove cap of vial just prior to sampling.
2. Hold cap in same hand as the bottle.
3. Tilt vial slightly into water and fill slowly to minimize the turbulence and aeration. Bailer bottom emptying device is recommended.

4. Fill vial to overflow insuring that a positive meniscus is formed.

5. Place cap on top of septum and quickly screw it on tightly.

6. Turn vial upside down and tap it several times to insure that there are no air bubbles trapped in liquid or inside of container.

7. If bubbles are noticed, discard the sample and begin over with a new set of vials.

8. Wash outside of vial with distilled or organic free water and wipe clean with a paper towel.

9. Label and mark it with project number, description, sample number, sampler's initials, date and time of sampling, etc., with a waterproof marker.

10. Store in ice-packed sample container and ship with a chain-of-custody record.

This SOP is referenced in SOP 1104 Section 3.12

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Landfill Gas Monitoring
DATE: February 1994
SOP NUMBER: 470 Page 1 of 3

used during gas probe installation

1.0 SCOPE

This operating procedure describes steps involved in landfill gas monitoring. Manufacturer's specifications and recommendations for gas monitoring equipment should be followed.

2.0 OBJECTIVES

The activities covered by this procedure:

- To measure the presence and concentration of landfill gas production.
- Insure quality control and consistency in taking readings.
- Serve as a means to allow traceability of error(s) in data readings.

3.0 EQUIPMENT NEEDED

- Combustible gas indicator capable of obtaining methane gas concentration within two ranges:

  - 0 to 100% LEL (0 to 5% combustible gas by volume)
  - 0 to 100% combustible gas by volume;

- Monitoring field data sheets.

4.0 PRELIMINARY TO OPERATION

1. Review project work plan for site-specific procedures.
2. The combustible gas indicator should be checked for defects, and any possible need for repair.
3. Batteries should be checked in the combustible gas indicator.
4. If the combustible gas indicator does not have an internal calibration, use methane gas to calibrate the combustible gas indicator prior to gas measurements in the field.

5.0 OPERATING PROCEDURE

1. Record the conditions at the site (i.e. weather), time, date and all other pertinent information on a gas monitoring record similar to the attached, or in a field logbook.

2. Obtain barometric pressure and barometric trend (i.e. increasing of decreasing) for the day gas monitoring is preformed.

3. Identify each gas probe (marked on well casing). Turn on the combustible gas meter and allow pump to run in ambient air.

4. Connect the combustible gas indicator to the plastic tubing on the probe. Measure and record the percent of the lower explosive limit. Record maximum reading.

5. Switch meter to percent gas setting and record the maximum reading obtained for percent combustible gas.

6. Disconnect combustible gas meter from tubing. Allow pump to continue to operate in ambient air to purge the meter of excess gas.

LANDFILL GAS MONITORING DATA

DATE:___________
TIME:___________

Mon. Probe Location	% Gas by Volume	% LEL	Comments/Notes

Weather:

Barametric Pressure: Trend:

sop\lfg-mon.470

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Air Sampling - Tedlar Bags
DATE: September 1995
SOP NUMBER: 500 Page 1 of 2

1.0 SCOPE

This operating procedure describes steps involved during the collection of air samples using tedlar bags. Manufacturer's specifications and recommendations for air sampling equipment should be followed.

2.0 OBJECTIVES

The activities covered by this procedure:

- Air sample collection.
- Insure quality control and consistency in obtaining air samples.

3.0 EQUIPMENT NEEDED

- Air sampling pump.
- Tedlar or equivalent sample bags.
- Monitoring field data sheets.

4.0 PRELIMINARY TO OPERATION

1. Review project work plan for site-specific procedures.
2. Fully charge the air sampling pump's batteries.
3. Air sample bags should be labeled for each air sample location.
4. Air sampling locations should be clearly labeled.

5.0 OPERATING PROCEDURE

1. Record the conditions at the site (i.e. weather), time, date and all other pertinent information on a gas monitoring record or field logbook.
2. Identify each air sample location.
3. Connect the air sampling pump to the plastic tubing on the probe or sampling port from which air sample is to be collected. Switch on the air sampling pump and allow pump to purge air for one minute.
4. Open tedlar sample bag to be filled.
5. Connect the air sample pump to the tedlar sample bag and fill sample bag.
6. Disconnect air pump from tedlar sample bag and completely purge tedlar sample bag to remove any impurities from the sample bag.
7. Reconnect air pump to tedlar sample bag and refill bag.
8. Disconnect air pump from the tedlar bag. Close tedlar sample bag and check bag for leaks. If a leak is detected, replace tedlar bag and repeat procedures 3-8.
9. Disconnect air pump from plastic tubing on the probe or sample port. Allow air pump to purge ambient air for thirty seconds before switching air pump off.

DAMES & MOORE
STANDARD OPERATING PROCEDURE

TITLE: Air Sampling - SUMMA Canister
DATE: September 1995
SOP NUMBER: 501

1.0 SCOPE

This operating procedure describes steps involved during the collection of air samples using SUMMA Canisters. Manufacturer's specifications and recommendations for air sampling equipment should be followed.

2.0 OBJECTIVES

The activities covered by this procedure:

- Air sample collection.
- Insure quality control and consistency in obtaining air samples.

3.0 EQUIPMENT NEEDED

- SUMMA Canister.
- Particulate filter (should come with SUMMA Canister).
- 0-30 psi vacuum gauge.
- 1/4-inch flexible tubing.
- Air sampling pump.
- Monitoring data sheets.
- Laboratory canister pressure verification form.
- Chain of custody.

4.0 PRELIMINARY TO OPERATION

1. Review project work plan for site-specific procedures.

2. Check the pre-test vacuum on the SUMMA canister by attaching the pressure gauge to the canister port and opening the canister valve. Verify the canister vacuum is the same as the vacuum recorded on the laboratory canister vacuum verification form.

3. Label SUMMA canister for sample location from which air is to be collected.

4. Air sampling locations should be clearly labeled.

5. Use a new flexible tube and particulate filter for each sample.

5.0 OPERATING PROCEDURE

1. Record the conditions at the site such as weather, time, date and all other pertinent information on a gas monitoring record or field logbook.

2. Identify each air sample location (i.e. sample port, gas probe, flux chamber).

3. Connect air pump to the sample port or gas probe from which air sample is to be collected using flexible tubing. Purge the sample location using the air pump for one (1) minute.

4. Connect the particulate filter to the SUMMA canister port. Use the flexible tubing to connect the particulate filter to the sample port from which the air sample is to be collected. Open the valve on the SUMMA canister. Allow the canister to fill for two (2) minutes.

5. Close the canister valve hand-tight plus 1/4 turn.

6. Check the post-test vacuum on the SUMMA canister by attaching the pressure gauge to the canister port and opening the canister valve. Verify the canister vacuum is zero. Record the vacuum reading on the laboratory canister vacuum verification form.

7. Ship the SUMMA canister along with the particulate filter, chain of custody, and laboratory canister vacuum verification form to the laboratory.

APPENDIX F

LABORATORY STANDARD OPERATING PROCEDURES

Quanterra
Environmental
Services

Controlled Copy
Copy No. 021

SOP No. COI-MS-0003
Revision No. 0
Revision Date: 04/09/96
Page: 1 of 22

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS (VOCS) IN AMBIENT AIR BY GC/MS - SCAN MODE USING EPA METHOD TO-14

(SUPERSEDES: LM-ATL-7001)

Prepared by: Mark Johnson

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1. SCOPE AND APPLICATION

1.1. Analytes: VOCs with molecular weights in the general range of 40-200 g/mol suitable for GC/MS analysis. Target analytes are listed in Table 1.

1.2. Method detection limits (see current MDL study).

1.3. Reporting limits: in the general range of 2-20 ppbv (See Table 1).

1.4. Applicable matrices - air, vapor

1.5. Dynamic range (See Table 1)

1.6. Approximate analytical time

1 min. - cool down of cryotrap
1 min. - flush of inlet system with internal standard
1 min. - collection of 100 ml internal standard on trap
1 min. - flush of inlet system on trap
5 min. - collection of 500 ml of sample/standard on trap
1 min. - flush of trap with HP Helium and GC oven cool down
24 min. - GC run time

When running multiple samples, steps can be overlapped to reduce run time to approximately 25 min.

2. SUMMARY OF METHOD

2.1. An air sample is metered through a mass flow controller and concentrated onto a cryogenically cooled trap. After 100 ml of internal standard and 500 ml (nominal) of the sample has been trapped, a valve is switched and the trap is heated to purge the trap's contents onto the gas chromatography column. The target compounds are analyzed with a mass spectrometer operated in the scan mode.

3. DEFINITIONS

3.1. Standard pressure is defined as 1.0 atmosphere, 14.6 psia, 0" Hg, and 0 psig, assuming Quanterra Air Toxics Lab, City of Industry elevation and average barometric pressure.

3.2. Standard molar volume is defined as 24.5 l/mol at room temperature.

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, which is one of the target compounds. Before using ultra high purity (UHP) Nitrogen (N_2), Hydrocarbon (HC) free air, Internal Standard (I.S.), or a target compound standard mix, each regulator should be purged a minimum of three times with the appropriate gas.

4.2. Contamination may occur in sample or standard results if canisters are not properly cleaned prior to use. Canisters should not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above 0.2 ppbv, or a level previously agreed upon by Quanterra and Client. All other sampling equipment including pumps, flow controllers and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.3. High levels of CO_2 and/or moisture may limit the amount of sample that can be concentrated due to plugging of the trap. High levels of CO_2 may also over-pressurize the instrument's vacuum system requiring delay of scan start time or the reduction of amount of sample trapped. High levels of CO_2 have been shown to shift the retention time of the first several target compounds earlier.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves should be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. At no time should SUMMA canisters be pressurized over 40 psig.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Gas chromatograph - capable of subambient temperature programming for the oven and with the jet separator option (Hewlett Packard 5890).

6.2. Mass-selective detector - equipped with computer and appropriate software (Hewlett Packard 5970B with HP-1000 RTE-A data system).

6.3. Cryogenic trap with temperature control assembly (Nutech 8533 and 3538). See Figure 1.

6.4. Electronic mass flow controller - for maintaining constant sample flow through Nutech concentrators (Unit Instruments)

6.5. Chromatographic grade stainless steel or nickel tubing and stainless steel plumbing fittings.

6.6. Chromatographic column - DB-624 0.53 ID, 30 meter length (J&W Scientific).

6.7. Stainless steel vacuum/pressure gauge capable of measuring from 30" of mercury (Hg) to 40 psig (Span Instruments) or equivalent.

6.8. High precision vacuum gauge - for making daily standards. (Wallace & Tiernan Pennwalt)

6.9. Pressure regulators for carrier gas and standards - 2 stage, stainless steel diaphragm (single stage acceptable for standards).

6.10. SUMMA passivated canisters 6 L or 15 L (Scientific Instrumentation Specialists, Anderson Instruments) or equivalent.

6.11. 7 micron filters (Nupro), or equivalent.

6.12. Vacuum flow regulators capable of flow settings to give required time sample flows (Valin or equivalent).

6.13. Diaphragm type vacuum pump to draw sample through mass flow controller.

7. REAGENTS AND STANDARDS

7.1. 4-Bromofluorobenzene, 25 ng/uL in methanol (for tuning of mass spectrometer)

7.2. High purity air and helium for making dilutions and for carrier gas.

7.3. Pressurized air source for valve actuation.

7.4. Gas calibration stock standards are at a nominal concentration of 1 ppmv (CS_2 is not as stable and so the concentration is 5 ppmv). Standards are prepared in a balance gas of nitrogen and are analytically certified by the supplier (Scott-Marrin, Scott Specialty, or equivalent). To facilitate certification by vendor, the standards were divided into several cylinders. (See current stock cylinder standard certifications)

7.5. Internal standard mix of Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5 at 250 ppbv each in a balance gas of nitrogen (Scott-Marrin or equivalent).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in precleaned and batch analyzed SUMMA passivated canisters. A 7 micron filter should be placed on the inlet of the can to protect the valve from particulates. Canisters should never be pressurized over 40 psig.

8.2. The absolute pressure of the canister should be recorded before and after sample collection. See Section 11.1 Sample preparation.

8.3. Samples must be kept within the temperature range of 20 to 30 °C.

8.4. Tedlar bag samples should be protected from sunlight.

8.5. Canisters samples should be analyzed within 14 days of collection.

8.6. Tedlar Bag samples should be analyzed within 3 days of collection.

9. QUALITY CONTROL

9.1. For every 12 hour shift the mass spectrometer must meet the tuning criteria described in Section 11.2.

9.2. After tuning, a single point check standard must be analyzed. Ninety percent of the target compound response factors must be within ± 30 percent difference of the initial multipoint calibration curve average response factors. If the check standard fails to meet this criterion, the system and standard conditions should be evaluated and the standard reanalyzed. If the reanalysis fails to meet linearity criteria a new five point calibration curve must be run.

9.3. A laboratory control sample (LCS) must be analyzed daily or every 20 samples, whichever is more frequent. This sample will consist of target VOCs prepared in a separate canister from a second source. Five compounds will be used to assess control for the LCS: methylene chloride, 1,1-dichloroethene, trichloroethene, toluene and 1,1-2,2-tetrachloroethane. The percent recovery for the five control compounds must be within a window of 80-115% or a window established using historical lab data. QC sample limits may change once established using historical lab data.

9.4. For each lot of 20 samples analyzed, a duplicate control sample (DCS) must be analyzed. The DCS sample is identical to the LCS in composition and source. The same LCS percent recovery criterion must be met. In addition, the relative percent difference (RPD) for the LCS and DCS must be ≤ 10%. If either of the control samples fail the criteria the system should be checked and the LCS that failed reanalyzed. Samples will not be considered reportable until the DCS criteria are met.

9.5. A method blank must be analyzed every 12 hour shift before samples. The blank results must indicate that there are no target compounds present above the reporting limits (RL). The method blank is prepared by adding zero grade air to a SUMMA canister. Internal standards are added to the trap and the blank is processed exactly as a sample or standard.

9.6. If any of the above criteria are not met, corrective actions must be implemented before analyses can proceed.

9.7. Internal Standards and their associated key ions are noted in Table 2. The internal standard areas are monitored for each 12 hour shift by comparing the areas of the internal standards in each sample with the areas of the internal standards in the daily continuing calibration standard. Sample areas are considered acceptable if they fall between 50% and 150% of the daily standard areas. Any sample outside this criteria should be reanalyzed. Samples failing upon repeat analysis should be documented on the analysis benchsheet and in the report narrative. The internal standard area of Bromochloromethane should always be greater than 50,000 area counts to reliably achieve standard reporting limits.

10. CALIBRATION AND STANDARDIZATION

10.1. A low level static dilution of the stock standard gas mixtures is made in a 6 or 15 liter SUMMA canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 2.00 psi of each of the 4 standard mixtures and 1.00 psi of CS2 are added to the canister (each regulator and the transfer line should be flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the can with HC free air to 30 psig. This will yield a standard with a nominal concentration of 45 ppbv for most compounds. Actual concentrations of each compound as certified should be used in all calculations.

10.2. A high level static dilution of the stock standard gas mixtures is made in a 6 or 15 liter SUMMA canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 8.00 psi of each of the 4 standard mixtures and 2.00 psi of CS2 are added to the canister (each regulator and the transfer line should be flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the can with HC free air to 30 psig. This will yield a standard with a nominal concentration of 179 ppbv for most compounds. Actual concentrations of each compound as certified should be used in all calculations.

10.3. An initial, minimum 5 point, curve is run in the linear working range of the system for each compound. The nominal concentration of the standards are 2.2, 4.5, 11, 45, 144, 287, and 448 ppbv. More than 5 levels are run in order to maximize each compound's linear working range and to verify reporting limits. These concentrations are achieved by varying the amount of standard trapped on the instrument and/or the concentration of the standard being analyzed. A percent relative standard deviation (%RSD) is calculated for each target analyte in that analytes dynamic range as indicated in Table 1 using the calculation in section 14.4. 90% of the target compounds must be less than 30% RSD to accept the curve for analysis. Refer to section 11 for RSD calculation. All compounds should have a response factor greater than 0.05.

10.4. During every 12 hours of operation, after tuning criteria is verified, a one point midrange standard (500 mL of 44 ppbv) is run to verify linearity with the initial calibration. A percent difference is calculated between the response factors (RFs) from the continuing calibration standard and the average RFs from the initial curve for each target analyte using the calculation in section 11. 90% of the target compound response factors must be within 30% difference of the initial multipoint curve average

Response Factor, or a new initial calibration must be run. All compounds should have a response factor greater than 0.05.

10.5. The daily, one point check standard response factors (RFs) are used to quantitate the results of the samples for the 12 hour shift.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. The initial pressure of the sample canister is checked and recorded by attaching a vacuum/pressure gauge to the canister. The gauge should be rinsed before use with zero grade nitrogen by physically holding against the air outlet and flushing for a few seconds. If the pressure is less than approximately 6 psig, pressurize the canister to 10 psig with zero grade nitrogen and record the final pressure. Pressures should be recorded in the sample pressurizing log and on the individual canister field sheets.

11.1.2. If the canister pressure is increased, a dilution factor (DF) is calculated and applied to results:

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = *absolute canister pressure before dilution (initial pressure)*
Y_a = *absolute canister pressure after dilution (final pressure)*

11.2. Initial/Daily GC/MS Tuning

11.2.1. After a successful autotune as per manufacture recommendations each instrument is manually tuned using PFTBA so that m/z 69 is 100%, m/z 131 is approximately 34%, and m/z 213 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day.

11.2.2. At the beginning of each 12 hour shift, prior to any analytical runs, the GC/MS system must be verified that acceptable tune performance criteria are achieved. If any of the key ions fail the abundance criteria listed in Section 11.2.4, the system is considered out of tune and any subsequent sample/standard analysis should be considered unacceptable.

11.2.3. For shift tune verification the cryotrap is not used and should be left at 150C. Alternatively the trap can be cold or cooling down but should be out of line with the column. The GC program (see Table 2) is initiated by using the BAMON commands. The GC programs are named "GCBFB1" and "GCBFB2" for systems A and B respectively. Tune files are typically "MTBFB1" and "MTBFB2." Once the data system has initiated the run and GC oven has stabilized, the remote start light will turn on and the system is ready for injection.

2 uL of a 25 ng/uL 4-Bromofluorobenzene (BFB) standard is injected into the injection port of the concentrator unit and the remote start button is activated.

11.2.4. Once the tuning run is complete (~ 6 minutes), run the program TUNVOA, data file." This is a program that will evaluate the tuning analysis and print out the required information automatically. If the BFB tuning criteria cannot be met on the first injection, retuning the instrument with PFTBA and or system maintenance may be required prior to re-injection of BFB.

Mass	**Ion Abundance Criteria**
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

11.3. One time procedural variations are allowed only if deemed necessary and appropriate by the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo including approval by a Technical Specialist and QA Manager. If it is determined that the quality of the sample data is significantly affected and/or if contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.4. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. ANALYSIS

12.1. The continuing standard and the QA/QC samples are analyzed the same as samples. After the one-point continuing calibration standard is analyzed an evaluated (section 9.2), duplicate laboratory control samples (DCS) are analyzed and evaluated (See sections 9.3 and 9.4).

12.2. Before sample analysis a zero grade nitrogen method blank is analyzed and evaluated (see section 9.5), The method blank consists of a SUMMA canister that is filled with zero grade nitrogen.

12.3. At the beginning of a sample or standard run the sample valve is in the purge position and the trap valve is in the column position. The trap is cooled to <150C. The internal standard canister attached and approximately 100 ml is flushed through (this can be concurrent with the trap cool down). After flushing the trap valve is placed in the sample position and the trap temperature is checked to be at -150C or less.

Trapping is started by switching the sample valve to sample position and activating a timer. When 100 ml has been trapped (1 minutes at 100 ml/min) the sample valve is switched to purge and the internal standard canister is closed and removed.

12.4. The sample/standard canister is then attached and approximately 100 ml is flushed through. Trapping is again started by switching the sample valve to sample and starting a timer. After the appropriate amount of time (5 minutes for 500 ml at 100 ml/min) the sample valve is switched back to purge, the canister is closed and the data system is set up.

12.5. Typically, the "Name" field in the data system set up screen contains the Quanterra sample number and the name of the client. The "Misc." section contains the volume of the sample that was trapped, the client sample ID, the canister number (or a note of "Tedlar bag"), and the canister or Tedlar bag dilution factor (final/initial pressure, if applicable).

12.6. The GC system methods are "GCNEW1: and "GCNEW2" (see Table 3). When the GC reaches ready at the programmed start temperature, and after waiting at least 30 seconds after trapping, the trap valve is switched to column, and then the cryotrap is switched to heat and the run is started simultaneously.

12.7. A canister filled with zero grade air is attached to the sample line in between samples. The line is allowed to flush until it is time for the next sample.

12.8. Trapped sample volumes are adjusted for pressurization to give an undiluted volume of 500 ml.

13. DATA INTERPRETATION

13.1. Qualitative Analyses

13.1.1. Two criteria must be satisfied to verify positive identification. (1) elution of sample component at the same GC relative or absolute retention time as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectra.

13.1.1.1. The sample component relative retention time (RRT) must compare within ± 0.06 RRT units of the RRT of the standard component. As an option, RT must compare within 0.5 minutes of the standard component absolute retention time (RT). For reference, the RT standard must be run within the same 12 hour shift as the sample.

13.1.1.2. (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard reference and sample spectra. (Example: For an ion with an abundance of 50% in the reference spectra, the corresponding sample abundance must be between 30 and 70 percent. Standard reference mass spectra must be obtained on each individual system.

13.1.1.3. If a compound cannot be verified by all of the criteria in the above paragraphs but in the technical judgment of the analytical chemist the identification is correct, then the compound may be reported.

13.1.2. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. normally, compounds will be given general names consisting of major functional groups and number of carbon atoms unless a retention time reference is available. Guidelines for making tentative identification are:

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

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

13.1.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

13.1.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

13.1.2.6. Only peaks having a total ion current greater than 10% of the nearest eluting Internal Standard total ion current will be evaluated for reporting.

13.1.2.7. Semiquantitative results will be calculated for tentatively identified compounds using total ion current areas and assuming a relative response factor of 1.0 of the nearest internal standard.

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

13.2. Quantitative Analysis:

13.2.1. When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. A summary table of primary and secondary ions for target compounds and internal standards is listed in Table 4. The calculation for quantitation of sample is found in section 14.

14. DATA ANALYSIS AND CALCULATIONS

14.1. The HP data system is set up to automatically quantitate the sample results based on a 500 ml sample size. The default result units are in ppbv.

14.2. If a sample size other than 500 ml was used and/or a canister sample was pressurized, the result must be adjusted as shown below:

$$\text{Final result ppbv} = \text{raw result ppbv}\ X\ \frac{500\,mL}{\text{sample volume injected}}\ X\ \frac{\text{final psia}}{\text{initial psia}}$$

14.3. Calculation for Relative Response Factor (RRF):

$$RRF = \frac{\text{Area cpd in Std.}}{\text{Area I.S.}}\ X\ \frac{\text{Conc. I.S.}}{\text{Conc. cpd in Std.}}$$

The area of the primary quantitation ion is used in the calculation.
I.S.: Internal Standard

14.4. Calculation for Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\text{Std. Dev. of RRFs}}{\text{Mean of RRFs}}\ X\,100$$

14.5. Calculation for Percent Difference (%D):

$$\%D = \frac{\text{Average RRF from initial curve RRF cpd.}}{\text{Average RRF from initial curve}}\ X\,100$$

14.6. Calculation for Determining Concentration of Compounds:

$$\text{Conc. Cpd}(ppbv) = \frac{\text{Area cpd in sample}}{\text{Area I.S. in sample}}\ X\ \frac{\text{Conc. I.S.}}{\text{RRF daily Std.}}\ X\ \text{Dil. Factor}(\text{See } 14.2)$$

The area of the primary quantitation ion is used in the calculation.
I.S.: Internal Standard

14.7. Calculation for Percent Recovery (%Rec):

$$\%\,\text{Rec} = \frac{\text{Amount cpd. recovered}}{\text{Amount cpd. spiked}}\ X\,100\%$$

14.8. Calculation for Relative Percent Difference (RPD):

$$RPD = \frac{\text{Value } A - \text{Value } B}{\text{Average of Values}}\ X\,100$$

15. REPORTING

15.1. Standard reporting units are ppbv. If results are to be reported in ng/L or ug/m^3 use the following equation:

$$result\ ppbv\ X \frac{Molecular\ weight\ of\ compound}{24.5} = results\ ng/L\ or\ ug/m^3$$

Note: 24.5 is the molar volume of ideal gas at 25° Centigrade and 1 atm.

15.2. Reporting limits

15.2.1. See Table 1. All reporting limits and MDLs must be derived on GC/MS systems at the Quanterra Air Toxics laboratory and are periodically updated.

15.2.2. "J" values (results below the normal reporting limit but above the MDL) are issued on request only.

15.3. Significant figures

15.3.1. All results should be reported to two significant figures.

15.4. No conversion of the analytical results to standard conditions is made.

16. METHOD PERFORMANCE

17. POLLUTION PREVENTION

18. WASTE MANAGEMENT

19. REFERENCES

19.1. Method Source

19.1.1. "EPA Compendium Method TO-14. The Determination of Volatile Organic Compounds (VOCs) in Ambient Air using SUMMA Passivated Canisters Sampling and Gas Chromatographic Analysis."

19.2. Deviations from Method

19.2.1. Nitrogen is used for dilution/pressurization purposes.

19.2.2. TO-14 recommends the use of a 0.32 mm column coupled directly to the MSD. With the HP system, the MSD can only handle flow of 1 ml/min or less. The 0.32 mm column provides ~ 3 ml/min. Quanterra uses a 0.53 mm column through a jet separator.

19.2.3. TO-14 describes an inlet system that uses a vacuum to pull the sample through the trap. Quanterra optionally uses the pressure of the sample canister to drive the sample through the trap.

19.2.4. TO-14 describes the use of a Nafion dryer to remove excess moisture from air matrices. Quanterra does not use a Nafion dryer since polar compounds may be lost during this removal step.

19.2.5. TO-14 describes the use of SUMMA passivated steel canisters for sampling and analysis. No mention is made of Tedlar sampling bags. Quanterra analyzes samples in Tedlar bags for VOCs using the same procedures described herein. A modification to the method is noted on the final report.

20. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

Table 1 VOC Target Compounds

Compound	R.T. (approx.)	Report Limits (ppbv)	Dynamic Range (ppbv)
Dichlorodifluoromethane (Freon 12)	2.05	2.0	2-450
Chloromethane	2.62	4.0	4-450
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.66	2.0	2-450
Vinyl Chloride	2.89	2.0	2-450
Bromomethane	3.34	2.0	2-450
Chloroethane	3.52	4.0	4-450
Trichlorofluoromethane (11)	3.85	2.0	2-450
1,1-Dichloroethene	4.44	2.0	2-450
Carbon Disulfide	4.54	10.0	10-360
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	4.52	2.0	2-450

Compound	**R.T. (approx.)**	**Report Limits (ppbv)**	**Dynamic Range (ppbv)**
Acetone	4.62	10.0	10-450
Methylene Chloride	5.07	2.0	2-450
trans-1,2-Dichloroethene	5.38	2.0	2-450
1,1-Dichloroethane	5.91	2.0	2-450
Vinyl Acetate	6.13	10.0	10-450
cis-1,2-Dichloroethene	6.70	2.0	2-450
2-Butanone	6.84	10.0	10-450
Chloroform	7.23	2.0	2-450
1,1,1-Trichloroethane	7.33	2.0	2-450
Carbon Tetrachloride	7.56	2.0	2-450
Benzene	7.88	2.0	2-450
1,2-Dichloroethane	7.97	2.0	2-450
Trichloroethene	8.95	2.0	2-450
1,2-Dichloropropane	9.31	2.0	2-450
Bromodichloromethane	9.88	2.0	2-450
cis-1,3-Dichloropropene	10.70	2.0	2-450
4-Methyl-2-pentanone	11.11	10.0	10-450
Toluene	11.21	2.0	2-450
trans-1,3-Dichloropropene	11.82	2.0	2-450
1,1,2-Trichloroethane	12.12	2.0	2-450
Tetrachloroethene	12.19	2.0	2-450
2-Hexanone	12.78	30.0	30-450
Dibromochloromethane	12.80	2.0	2-450
1,2-Dibromoethane	12.90	2.0	2-450
Chlorobenzene	13.98	2.0	2-450
Ethylbenzene	14.33	2.0	2-450
1,4-and 1,3-(p,m)Xylene	14.61	2.0	2-900
1,2-(ortho)Xylene	15.43	2.0	2-450
Styrene	15.49	2.0	2-450
Bromoform	15.78	2.0	2-450
1,1,2,2-Tetrachloroethane	17.18	2.0	2-450
Benzyl Chloride	17.30	10.0	10-450
4-Ethyltoluene	17.55	2.0	2-450
1,3,5-Trimethylbenzene	17.73	2.0	2-450
1,2,4-Trimethylbenzene	18.55	2.0	2-450
1,3-Dichlorobenzene	19.02	2.0	2-450
1,4-Dichlorobenzene	19.26	2.0	2-450
1,2-Dichlorobenzene	19.83	2.0	2-450
1,2,4-Trichlorobenzene	21.32	20.0	20-450

Compound	**R.T. (approx.)**	**Report Limits (ppbv)**	**Dynamic Range (ppbv)**
Hexachlorobutadiene	21.52	2.0	4-450

TABLE 2 BFB GC Method

Method file: GCBFB1(2)

METHOD FILE LIST

Method file:	GCBFB1 GCBFB2	GC Type: 5890 Column: Cap	Run type: SCAN,GC,E1 Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	30.0	100.0	0.0	0.0
Time 1	1.0	3.0	0.0	0.0
Rate	35.0	0.0	0.0	
Temp 2	100.0	0.0	0.0	
Time	15.0	0.0	0.0	

Oven equilibration Time. 0.10 min

Run time:	6.00
Scan Start time	2.50

Scan Parameters:	Mass Range:	34 to 260		
	Multiplier voltage:	varies	Number of A/D samples:	8
	GC Peak threshold:	20000 counts		
	Threshold:	100 counts		

TABLE 3 GC Analytical Method

Method file: GCNEW1(2)

METHOD FILE LIST

Method file:	GCSYS1 GCSYS2	GC Type: 5890 Column: Cap	Run type: SCAN,GC,E1 Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-50.0	10.0	100.0	0.0
Time 1	1.5	0.0	0.0	0.0
Rate	50.0	5.5	40.0	
Temp 2	10.0	100.0	160.0	
Time	0.0	0.0	3.4	

Oven equilibration Time. 0.10 min

Run time:	24.00
Scan Start time	1.60

Scan Parameters:	Mass Range:	34 to 260		
	Multiplier voltage:	varies	Number of A/D samples:	8
	GC Peak threshold:	20000 counts		
	Threshold:	10 counts		

TABLE 4 VOC Key Ions

Compound	**Primary**	**Secondary**		
Bromochloromethane I.S. #1	49	130	128	
Dichlorodifluoromethane (Freon 12)	85	87,	50	
Chloromethane	50	52		
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	85	135	87	
Vinyl Chloride	62	64		
Bromomethane	94	96,	79	
Chloroethane	64	66,	49	
Trichlorofluoromethane (11)	101	103	66	
1,1-Dichloroethene	61	96,	63,	98
Carbon Disulfide	76	78,	44	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	151	101	103	85
Acetone	43	58		
Methylene Chloride	84	49,	86	
trans-1,2-Dichloroethene	96	61,	98,	63
1,1-Dichloroethane	63	65,	83	
Vinyl Acetate	43	44,	86,	42
cis-1,2-Dichloroethene	96	61,	98,	63
2-Butanone	72	57,	43	
Chloroform	83	85,	47	
1,1,1-Trichloroethane	97	99,	61	
Carbon Tetrachloride	117	119	121	82
1,4-Difluorobenzene I.S. #2	114	63,	88	
Benzene	78	50,	52,	77
1,2-Dichloroethane	62	64,	49,	98
Trichloroethene	130	95,	132	97
1,2-Dichloropropane	63	62,	41,	39
Bromodichloromethane	83	85,	129	
cis-1,3-Dichloropropene	75	77,	39	
4-Methyl-2-pentanone	43	58,	57,	85
Toluene	91	65,	92	
Chlorobenzene-d5 I.S. #3	117	52,	54,	82
trans-1,3-Dichloropropene	75	77,	39	
1,1,2-Trichloroethane	97	83,	85,	61
Tetrachloroethene	164	129	131	166
2-Hexanone	58	43	57	
Dibromochloromethane	129	127	208	131
1,2-Dibromoethane	107	109	188	
Chlorobenzene	112	77,	114	

TABLE 4 VOC Key Ions

Compound	Primary	Secondary		
Ethylbenzene	91	106		
1,4-and 1,3-(p,m)Xylene	91	106	105	
1,2-(ortho)Xylene	91	106	105	
Styrene	104	78	103	51
Bromoform	173	171	175	93
1,1,2,2-Tetrachloroethane	83	85,	133	131
Benzyl Chloride	91	126	63	
4-Ethyltoluene	105	120	77	
1,3,5-Trimethylbenzene	105	120	77	
1,2,4-Trimethylbenzene	105	120	77	
1,3-Dichlorobenzene	146	148	111	75
1,4-Dichlorobenzene	146	148	111	75
1,2-Dichlorobenzene	146	148	111	75
1,2,4-Trichlorobenzene	180	182	109	145
Hexachlorobutadiene	225	227	223	

FIGURE 1. Nutech 8533 Flow Diagram

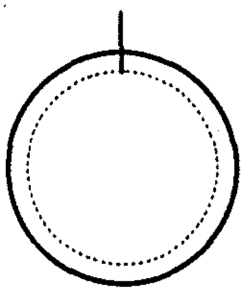
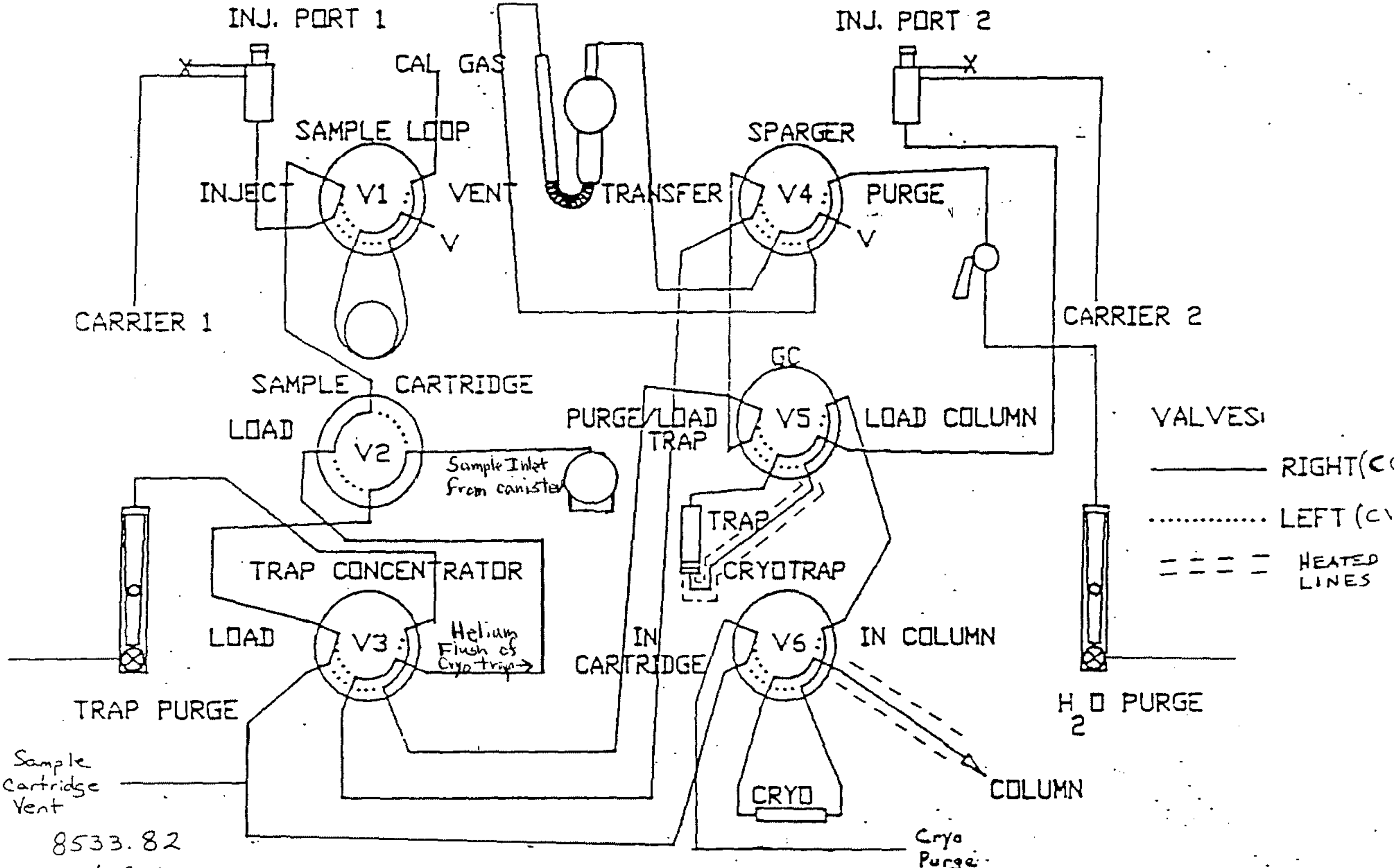


FIGURE I. NuTech 8533 Flow D jram



8533.82
w/ Cryo

VOST ANALYZER FLOW DIAGRAM (FRONT VIEW)

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY

SOP NUMBER: ORG-METH-GC-VOC-1

EFFECTIVE DATE: ______

CONTROLLED COPY NUMBER: ______

Originated By: ______ Date: 8-20-96
NLS Associate

Approved By: ______ Date: ______
Supervisor

Reviewed By: ______ Date: ______
QA Officer

Authorized By: ______ Date: 8/21/96
Laboratory Manager

I. METHOD TITLE: Determination of Volatile Organic Compounds by Gas Chromatography

II. METHOD SCOPE AND APPLICATION

A. This procedure is capable of determining various volatile organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage, groundwater, municipal and industrial wastes soils and sludges.

III. REFERENCES

A. U.S.E.P.A. 1982. "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" EPA Methods 601 & 602.

B. U.S.E.P.A. "Methods for Evaluating Solid Waste Physical Chemical Methods, SW-846", 3rd Edition., EPA Method 8010/8020 & 8021.

IV. METHOD SUMMARY

A. Highly volatile organic compounds with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through a 5 mL aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent material. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components onto a capillary gas chromatography (GC) column. The column is temperature programmed to separate the organic compounds.

B. Tentative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Additional confirmatory information can be gained by comparing the relative response from the two detectors (PID and HECD) to rule out halogenated compounds. Each identified component is measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

V. INTERFERENCES

A. During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) plastic tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in laboratory reagent blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high quantities of VOCs. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross contamination.

VI. SAMPLING

A. Water Samples

1. NLS will supply acid preserved 40 mL vials in duplicate. The vials are to be filled just to overflowing. No air bubbles should be trapped in the sample when the bottle cap is sealed. Store at 4°C. The sample must be analyzed within 14 days from its collection date.

B. Soil Samples

1. NLS will supply 60 mL wide mouth vials with teflon lined caps in duplicate. Also supplied will be 25 mL vials of methanol. As close to 25 grams as possible of soil is to be added to the jar. The 25 mL vial of methanol is to be opened and added immediately to preserve and extract the VOC's from the soil. Samples are to be stored at 4°C. The sample must be analyzed within 21 days and any confirmations must be done within 28 days. Up to 100 uL of methanol can be analyzed in 4.9 mL's of water.

VII. SAFETY

A. General Precautions

1. Some of the target compounds are known carcinogens and many others are suspected carcinogens. As a result, samples and standards should be treated as a potential health hazard and exposure to them should be minimized. See the material safety data sheets for additional information.

2. Personal: Handling samples and loading of the instrument requires eye protection.

VIII. EQUIPMENT AND MATERIALS

A. Sample Container:

1. 40 mL VOC vial with silicone PTFE faced Septa. NLS uses precleaned vials from Baxter Scientific or equivalent.

B. Purge & Trap:

1. Tekmar LSC 2000 or 3000 and ALS 2016 autosampler with 5 mL fitted sparge tubes.

C. Trap:

1. Supelco K - 3000 or equivalent.

D. Purge and Trap Conditions: Three Gas Chromatographic systems are certified for analyzing VOC's.

1. Varian 3400: Subambient cooling equipped with Tracor 703 PID and Tracor 700A HECD detectors.

2. Varian 3300: Subambient cooling equipped with Tracor 703 PID and Tracor 1000 HECD detectors.

3. Varian 3400 CX: Subambient cooling equipped with OI 5230 PID and OI 5220 HECD Detectors.

PURGE AND TRAP CONDITIONS TABLE

PURGE CYCLES	VARIAN 3400	VARIAN 3300	VARIAN 3400 CX
Purge Time:	11 Minutes	11 Minutes	11 Minutes
Flow Rate:	38 mL/Minute	38 mL/Minute	38 mL/Minute
Trap Temperature:	< 30°C	< 30°C	< 35°C
Desorb Temperature:	240°C	240°C	250°C
Bake Temperature:	260°C	260°C	260°C
Bake Time:	20 Minutes	20 Minutes	30 Minutes

4. GAS CHROMATOGRAPHIC CONDITIONS:

CONDITIONS	VARIAN 3400	VARIAN 3300	VARIAN 3400CX
Initial Column Temperature:	20°C	30°C	5°
Initial Hold Time:	5.0 Minutes	5.0 Minutes	2.0 Minutes
Ramp Rate:	4.0°C/Minute	4.0°C/Minute	3°C/Minute
Hold Time:	0 Minutes	0 Minutes	8 Minutes
Second Ramp Rate:	8°C/Minute	10°C/Minute	6°C/Minute
Hold Time:	5 Minutes	8.5 Minutes	1 Minute
Final Temperature:	220°C	210°C	220°C
Third Ramp Rate:	20°C/Minute	------	20°C/Minute
Hold Time:	2 Minutes	------	5 Minutes
Total Time:	53 Minutes	54 Minutes	58 Minutes
Detector Temperature (PID):	225°C	225°C	250°C
Injection Temperature:	125°C	125°C	200°C
Column Flow (He):	5 mL/Minute	5.6 mL/Minute	5.4 mL/Minute
PID Range:	11 or 12 *	11 or 12 *	11 or 12 *
HECD Range:	10	10	N/A
PID Lamp:	10.2 ev	10.2 ev	10.0 ev
GC COlumn:	J&W DB-624	Restek - RTX-502.2	J&W DB-VRX
Column ID:	0.53 mm	0.53 mm	.45 mm
Film Thickness:	3.0 μm	3.0μm	2.55 μm
Column Length:	60 Meters	105 Meters	75 Meters

***NOTE:** PID Attenuation varies upon age of PID Lamp.

5. HECD CONDITIONS:

CONDITIONS	VARIAN 3400	VARIAN 3300	VARIAN 3400CX
Reactor Tube:	Nickel	Nickel	Nickel
Reactor Temperature:	950°C	950°C	950°C
Electrolyte (100%)	N-Propyl ALcohol	N-Proply Alcohol	N-Propyl Alcohol
Reaction Gas Rate (Hydrogen):	35 mL/Minute	35 mL/Minute	100 mL/Minute

6. DATA REDUCTION: NLS is currently employing the HP3365 Series II Chemstation, Version A. 03.33. Using the following acquisition methods:

	Varian 3300	Varian 3400	Varian 3400CX
PID Signal Method	33 PID.MTH	34 PID.MTH	PID.MTH
HECD Signal Method	33 HLISTD.MTH	34 HLISTD.MTH	HALL (IS).MTH

Each data method includes:

a. On line devices.
   i. 35900D, Rev. D.01.00
   ii. 35900 D, Signals 1 and 2
   iii. Automatic Sampler
b. Integration Events.
c. Calibration Parameters.
d. Calibration Tables.
e. Signal Attenuation.
f. Report Specifications.

IX. REAGENTS AND STANDARDS

A. Supelco, Restek, UltraScientific, or equivalent.

1. Recommended Standards:

   a. DWM-580 (200 ug/mL), 60 components.

   b. tert-butyl Methylether (2000 µg/mL, Isopropyl ether (2000 µg/mL), Methylethyl ketone (2000 µg/mL), and Tetrahydrofuran (10,000 µg/mL)..

   NOTE: The final concentration of Tetrahydrofuran will be five times that of all other standards in the curve below. Reagent Purity Specifications.

2. Internal & Surrogate Standards:

Compound	Instrument	PID	HALL	Supplier	Conc.
Fluorobenzene	3300 & 3400	Internal Std.	Not Detected	Restek	10 ppb
1,4 Dichlorobutane	3300 & 3400	Not Detected	Internal Std.	Restek	10 ppb
4.Bromochlorobenzene	3400 CX	Internal Std.	Internal Std.	Supelco	10 ppb
2-Bromochlorobenzene	3300, 3400, 3400 CX	Surrogate	Surrogate	Supelco	10 ppb

a. Internal & Surrogate Standard compounds are added to all standards, spikes, samples and blanks.

b. Calibration curves are generated as necessary. Usually a six point calibration is analyzed. The concentration levels for all target and surrogate compounds are 1 ppb, 2.5, 5.0, 10, 15 & 20 ppb. The IS is always added at 10 ppb.

c. After a curve is generated all compounds must have a correlation coefficient of .990 or better to be acceptable.

d. It is NLS policy to bracket all samples with Quality Control Standards and Spikes. A typical run consists of a calibration check standard (all recoveries must be within 85 - 115 %). A carryover blank, a laboratory blank, 10 samples, a matrix spike, a matrix spike duplicate and finally another calibration check standard.

X. PROCEDURE

A. Extraction/Digestion/Preparation

1. Bring all samples to room temperature. Keep standards in the freezer until just before use.

2. Bring GC to operating conditions. If necessary install new tank of liquid carbon dioxide.

3. Use freshly distilled water. Rinse syringe between samples. Inject 5 mL of sample into Purge & Trap glassware. Add up to 100 ul of the methanol for soils.

4. Program Tekmar ALS to correspond with sample loading list.

5. Press start to begin Tekmar Purge & Trap.

6. If using cryogenic cooling: Press GC CONFIGURE. Press ENTER until: "Turn HARDWARE ON-OFF" appears on screen. Press #4 YES). Continue to press ENTER until COOLANT TO COLUMN ? NO appears on screen. Press #4 (YES). This turns on liquid carbon dioxide to cool GC oven.

7. All stages of the analysis are now functional and the presence of the operator is not necessary.

8. At the completion of the run, look for any possibilities of carryover. Check QC values for any compound found on a clients sample.

9. If QC is within limits, report all hits above MDL to 2 significant figures. See the EPA methods for more specifics on QC.

ANALYTES DETECTABLE BY THIS METHOD
(METHOD VOCs BY EPA 8021)

<u>ANALYTE</u>

Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
Carbon tetrachlordie
Chlorobenzene
Chloroethane
Chloroform
Chloromethane
2-Chlorotoluene
4-Chlorotoluene
Dibromochloromethane
1,2-Dibromo-3-chloropropane
Ethyl Dibromide
Dibromomethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
1,2-Dichloropropane
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
p-Isopropyltoluene
Methylene chloride

ANALYTES DETECTABLE BY THIS METHOD
(Method VOCs BY EPA 8021)

<u>Analyte</u>

Naphthalene
n-Propylbenzene
Styrene/O-xylene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichlorofluoromethane
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
Vinyl Chloride
m/p-xylene
tert-Butylmethyl ether
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene

XI. INSTRUMENT ANALYSIS - Addressed in graphs.

XII. CALCULATIONS

A. General Calculations

1. Computer Generated Numbers

a. Quantitation and retention times are based upon a direct comparison of the areas and retention times of a known standard.

b. Determine the concentration of individual analytes in the sample using the following formula:

i. Water Samples:

$$X \text{ ug/L} \times \frac{5 \text{ mLs}}{Y \text{ mLs}} = \text{Final Conc. (ug/L)}$$

Where: X = Concentration from calibration curve.
Y = mLs purged.

ii. Soil Samples:

$$Xug/L * \frac{1L}{1000\ ml} * \frac{25\ mL}{Ng} * \frac{1000\ g}{Kg} * \frac{1}{\%S} * \frac{5\ mL}{Y\ mLs} = \text{Fincal Conc. (ug/Kg)}$$

Where:
X = Concentration from calibration curve.
N = Grams of sample.
S = % Solid
Y = mLs purged

c. To verify the computer calibration of the curve, hand calculation of a compound from each curve will be performed.

2. Analytical Interpretation

a. Computer printed results:

i. The analyst will visually check the area count on each hit to verify that the hit is legitimate. If the area count doesn't compare to the curve, the hit is to be rejected.

b. Hall/PID confirmations:

i. A compound that appears on both detectors must have a comparable hit on both detectors. If only one detector has a hit the result is rejected.

c. Retention time comparison:

i. The retention time of a compound must be within 0.1 minutes of the actual retention time or the detect will be rejected.

d. Final Results:

$$Xug/L * \frac{1L}{1000\ mL} * \frac{25\ mL}{Ng} * \frac{1000\ g}{Kg} * \frac{1}{\%S} * \frac{5\ mL}{Y\ mLs} = \text{Final Conc. (ug/Kg)}$$

Where:
X = Concentration from calibration curve.
N = Grams of sample
S = % Solid
Y = mLs purged.

B. Significant figures

1. Sample results should be reported to two significant figures except for all Blind and Performance Evaluation studies which require 3 significant figures. The results entered into the template will automatically be rounded to the specified number in the reporting template.

C. Special Adjustments for Samples

1. Sample Size: Typical water sample volume is 5.0 ml. Typical soil sample volume is 100 ul of the methanol preservative.

2. Sample Dilution: Some samples do contain high levels of VOC's. When a sample has a hit above 20 ppb (the instruments calibration limit) it will have to be reanalyzed at a dilution which will bring the result down into the instruments upper half of the calibration range.

XIII. QUALITY CONTROL

A. Standards

1. Standards should be purchased from a reliable supplier and accompanied with a certificate of analysis which verifies the sample concentration for a specific lot number. The certificate of analysis must be kept in a file for a complete record of the standard preparation. Lot numbers and labels should be recorded and attached in the Standard Preparation Logbook. When working standards are made from a stock standard, they must be labeled properly with the date, concentration and description of the mix.

B. Lab Control Standard

1. This is a "second source" standard with a concentration mid-level in the calibration curve and is used to check both the stability of the initial calibration and the accuracy of the individual components. This control standard is used approximately on a Quarterly basis. This check ensures that the calibration is valid at the time of analysis and that the standards are at the level stated by the supplier.

C. Sample QC

1. Accuracy measurements (spike percent recovery)
2. Precision measurements (duplicate - relative percent difference)
3. Surrogate Measurements: Surrogate standards are added to every sample, blank and check standard at a level of 10 ppb.

D. Analytical Limitations

1. Sample Interferences: Matrix affects can be a problem with samples. Contaminant sources also cause problems. Care must be used to keep samples, standards and the analyst himself away from major contaminant sources within the laboratory.
2. Instrument Limitations: Lower quantitative limit is defined by the LOQ. Analytes may be detected at or near the MDL and should be considered the lowest detectable concentration. The upper quantitative limit is defined by the highest calibration standard in the initial calibration.

E. Method Validation:

1. Each analyst performing this analysis must successfully complete the initial demonstration of proficiency requirements stated in the method.

F. MDL study procedure:

1. Each analyte reported should have a Method Detection Limit (MDL) statistically determined according to 40 CFR, Part 136, Appendix B, Rev 1.1. MDLs should be performed at least yearly.

G. QC Failure Corrective Actions:

1. Calibration Check Standards:

    a. Frequency: These standards will be analyzed at the beginning and at the end of every analytical run. There will also be one analyzed after every 10 samples. All samples must be bracketed by calibration check standards (see example run log attached).

    b. Recoveries: Any samples analyzed using method 8021 & 8010-8020 must have compound recoveries of 85 - 115%. Wisconsin LUST type and method 601-602 samples must have compound recoveries of 80 - 120%.

    c. QC out of Limits: If a sample is found to have a compound that is beyond the calibration check standard recovery limits, then that sample is to be repeated unless the integrity of the sample is in question. An example would be that all of the sample vials have been opened and the proposed repeated sample analysis would have to be performed on a vial with headspace. Another example would be performing the proposed repeated analysis past the maximum holding time. In both of the above cases, the sample in question would then be flagged with a qualifier indicating that the calibration check standard was out of QC Limits for those compounds.

    d. Uniform Flag - Calibration check standard recovery for <u>compound name</u> was <u>actual %</u>.

2. Spike & Duplicate Spike Matrix:

    a. Frequency: The matrix spike and duplicate spike will be analyzed every day for each matrix (Excluding methanol). If more than 20 samples are being analyzed then a matrix spike and a duplicate must be analyzed after every 20 samples (see example Run Log attached).

    b. Recoveries: Matrix spike and duplicate spike recovery values must fall between the in-house calculated control limits for that given matrix (see attached QC daily Log Sheets for Soil and Water). This is also true for the relative % difference between spike recoveries and duplicate spike recoveries.

    c. QC out of Limits: If a sample is found to have a compound out of the spike % recovery control limits, then that sample is flagged with a qualifier for poor spike recoveries.

    d. Uniform Flag: Spike recovery for <u>compound name</u> was out of QC limits, but all other spike recoveries and calibration check standards were within QC limits.

3. Laboratory Blanks:

    a. Frequency: A laboratory blank must be analyzed at the beginning of every analytical day (see example Run Log).

    b. Contaminated Blank: If a blank is found to have compound concentrations above the compound's MDL, but lower than the compound's LOQ, any sample containing those compounds must be flagged. If the contaminate level is above the compound's LOQ, then all of the samples with this compound concentration above the

LOQ must be repeated unless the integrity of the sample is in question (see QC out of Limits for Calibration Check Standards). In this case, the sample will be flagged with a qualifier.

   c. Uniform Flag: Compound name was found in the laboratory blank at actual recovery.

4. Sample pH:
   a. Frequency: Every sample and sample repeat will be checked for proper sample pH and recorded on the run log.
   b. pH Requirements: Every sample must have a pH reading of < 2.0. If a sample pH is ≥ 2.0 then the sample must be flagged with a qualifier.
   c. Uniform Flag: Sample pH > 2.0 (WDNR requires sample pH < 2.0).
5. Surrogates:
   a. Frequency: A PID and a HECD compatible surrogate will be added to every sample.
   b. Recoveries: Surrogate recoveries must fall between in-house calculated control limits.
   c. QC out of Limits: If the recovery is found to be higher than the QC control limits, then the matrix of the sample should be examined and if the matrix is contributing to the recovery then that sample should be flagged. If the conclusion is that the internal standard is affecting the surrogate recovery, then the sample should be repeated to get the internal standard in line. If repeating the sample compromises the integrity of the sample, then the sample should be flagged with a qualifier (see QC out of Limits for the Calibration Check Standards for examples).
   d. Uniform Flags:
      i. Surrogate recovery was out of QC limits due to the matrix of the sample.
      ii. Surrogate recovery was out of QC limits due to unidentified hydrocarbons found in the sample.
      iii. The internal standard was contributed to by the sample matrix causing the surrogate recovery to be low.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets and logbook entry

1. Every analytical run must have benchsheets showing all samples analyzed as a batch. There are several ways to print benchsheets. The easiest way to get all VOC Benchsheets is to type 6 in the Benchsheet menu.
2. A logbook is also filled in during every analytical run. The logbook documents all area count recoveries for the internal standard and the surrogate percent recoveries.

Additionally, the logbook records the analysis or run list. There also is an instrument maintenance logbook which requires entries when maintenance or troubleshooting occurs.

B. Units / Significant figures

1. All water final results are rounded to two significant figures and are reported as ug/L. All soil results are rounded to two significant figures and are reported as ug/Kg.

C. Detection limits and Reporting limits

1. All detection limits are determined according to 40 CFR, Part 136, Appendix B, Rev. 1.1.

D. Qualifiers or comments used if data is to be flagged

1. Samples must be flagged with a specific comment if any of the following situations arise and are not corrected through reanalysis or recalibration.

   a. Surrogate recovery out of range.

   b. Holding time exceeded.

   c. Continuing calibration out of range.

   d. Sample not adequately preserved.

   e. Headspace present in sample vials.

E. LIMS Entry

1. The laboratory information system contains all of the data which appears on the final analytical reports. Results are entered into the LIMS system under results entry (RE). Simply enter the appropriate testcode, initials under who, date and time under date and then page down. This will call up all the samples under that testcode which do not have results entered. Under units, enter "see attached". If there are any footnotes, comments or qualifiers enter this information under the notes category. Shift F9 will allow you to see the entire field as you type. Once the correct information is entered save it by entering F4.

2. Now the data must be entered into the templates. To do this go into organic reports. Select enter results. This menu will prompt you for the Template Name, Project Number, Sample Number and Dilution. Enter the information into these fields and enter Page Down. This will allow you to enter in the final numerical result for the appropriate compound. The dilution factor will not adjust the result entered but only the MDL/LOQ on the final report.

F. Client Reports

1. After all the data is entered, return to the previous menu and select option (3) to print results on the final report. To print final results select option (10). Data will be printed on good paper at the main printer in the old building. Check reports to make sure results are correct.

G. Electronic Data Archiving

1. Archiving of data should be performed on a regular basis. Data files are either purged from the system or they are transferred to drive G, (altos ® hard drive) via the network. Since the data files are large it is advisable to contact the LIMS manager to see if there is enough room on drive G before transferring files.

H. Hard Copy Archiving

1. Processed data reports are printed for each sample analyzed. A copy of the final report should be stapled to the processed report. All data compiled from an instrument run should be kept in separate file folder. Information kept in the file folder includes, Continuing Calibration Summary, instrument blanks, and all sample data. The file folder should be labeled with the date of the analytical run. Periodically, data will be moved to the basement archives as the file boxes fill.

I. Instrument Run Logbook

1. The run logbook must be filled out for each analytical run.

J. Standard Preparation Logbook

1. The standard preparation logbook is filled out every time a new standard solution is opened or prepared. If an adhesive label is available it should be attached to the page containing the information about the standard prepared. This label is then traceable to the certificate of analysis for a complete record.

K. Maintenance Logbook

1. The maintenance logbook is a ring binder notebook. All work done on the instrument must be recorded. This information should be kept up to date for an accurate record of the instrument performance over time.

XV. CLEAN UP

A. Lab Work Area

1. Samples should always be returned to the upstairs refrigerator after use. Also, any materials, reagents, supplies or standards used throughout the day should be returned to their proper location. The lab area in general should always be kept clean and free from clutter.

B. Sample Disposal & Standard Disposal

1. Samples are disposed of in accordance with procedures determined in the QA/QC manual. Liquid samples are emptied down the drain and the 40 mL vials are recycled. Standards are emptied from the 5 mL vials into a solvent waste container. The glass vials are discarded into a glass jug that is later disposed. The solvent and standard contents will be removed from the premises by a waste hauler.

C. Equipment / Glassware

1. Equipment should be kept clean and dust free. Occasional cleaning may be needed to ensure equipment is free from dust and other debris. Also, glassware should be cleaned as soon as possible after use.

XVI. MAINTENANCE / TROUBLESHOOTING

A. Preventive maintenance procedures and frequency

1. As needed clean the PID Lamp.
2. As needed check the solvent reservoirs for the HECD detectors.
3. As needed replace the PID Lamp or any problematic HECD part.
4. As needed export files to main system hard drive - see XIV (G).

B. Troubleshooting procedures

XVII. ATTACHMENTS

A. Compound / Analyte list and MDLs

B. Worksheets or forms for recording data

C. Standard Operating Procedure Summary Sheets

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF SEMI-VOLATILES USING GC/MS EPA METHOD 8270B/625

SOP NUMBER: ORG-METH-GC/MS-S/VOL-0

EFFECTIVE DATE: ______

CONTROLLED COPY NUMBER: ______

Originated By: Jim Fisher Date: 8/20/96
NLS Associate

Approved By: ______ Date: ______
Supervisor

Reviewed By: ______ Date: ______
QA Officer

Authorized By: ______ Date: 8/21/96
Laboratory Manager

I. METHOD TITLE: Determination of Semi-Volatiles using GC/MS EPA Method 8270B/625

II. METHOD SCOPE AND APPLICATION

The following SOP is designed to meet the analytical requirements of both the SW-846 Method 8270B, and EPA Method 625.

Method 8270B is used to determine the concentration of Semi-Volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Method 625 is specifically limited to wastewater matrices. Direct injection of a sample may be used in limited applications where the analyte concentration is sufficiently high and the matrix is in a solvent such as Methylene Chloride.

Method 8270B as well as 625 can be used to quantitate most neutral, acidic and basic compounds that are soluble in methylene chloride and capable without derivatization of producing sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphorus esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The information contained in this Standard Operating Procedure is in reference to the GC-MSD system by Hewlett-Packard, model 5972A. The GC is the Hewlett-Packard model 5890, series II with electronic pressure control.

A. Parameters: See attachment I for a complete listing of the compounds which may be determined by method 8270B and 625. The WDNR has given NLS permission to run Semi-volatile compounds not included in the published list as long as MDLs have been determined.

B. NLS Test Codes / Descriptions

1. testcode 15094/8270B Water Analysis
2. testcode 15084/8270B Soil Analysis
3. testcode 15100/625 Analysis
4. testcode 15091/3510B (BNA) Extraction
5. testcode 15092/3510B (Acid) Extraction
6. testcode 15093/3510B (BN) Extraction
7. testcode 15102/3550A Ultrasonic Extraction
8. testcode 15103/3580 Waste Dilution

C. Detection Limits

1. See attachment II for a complete listing of the current MDLs for aqueous and solid samples.

III. REFERENCES

A. EPA and Standard Methods procedure source. EPA Procedure Source: SW-846, Update II & IIA, and Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater 600/4-82-057. July, 1982.

IV. METHOD SUMMARY

A. Gas Chromatography: Chromatography is essentially a physical method of separation in which components to be separated are distributed between two phases, one of them constituting a stationary phase and the other being the mobile phase. Chromatographic separation occurs as a result of repeated sorption-desorption acts during the movement of sample components along the stationary phase.

In Gas Chromatography the sample is volatilized in the injector port and swept onto the stationary phase of the column by the mobile gas phase (Helium). The separation takes place as the analytes travel along the column and interact with the column stationary phase. Analytes emerge at the end of the column at different times and enter the detector where they produce a signal proporitonal to the sample concentration.

The purpose of the injector port is to vaporize the liquid samples and permit the carrier gas to push the vapor in the form of a concentrated plug onto the beginning of the column with a minimum of time lapse. The sample introduction system consists of a heated block containing a glass insert which funnels the vapor onto the column. The liquid samples are injected into the glass insert with a 10 microliter syringe which penetrates the closed system by piercing a pliable synthetic septa which reseals the system after the needle is withdrawn.

The column is housed in a temperature programmable oven which is heated at a rate optimized to produce sufficient peak resolution in the shortest time possible. Heating the oven will speed up the compounds interation with the stationary phase causing it to emerge at the end of the column more quickly than it would at a lower temperature.

The initial oven temperature, the oven temperature ramp rate, final oven temperature and carrier gas flow rate are all variables which influence the separation process.

B. Mass Spectrometry: The mass spectrometer can be divided into the following main parts:

1. Sample Inlet Interface.
2. Ion Source.
3. Mass Filter.
4. Ion Detector.

5. Data Handling System.
6. Vacuum Chamber and Pumping System.

The Inlet system interface is a heated path from the GC oven to the MSD Ion Source. The interface is mounted on the right side of the vacuum manifold. One end of the interface passes through the side of the gas chromatograph and protrudes into the GC oven. The other end of the interface fits into a socket on the Ion Source side in the analyzer. The interface is heated by an electric cartridge heater and should be operated in the range of 250 to 320 degrees centigrade. The set point should be operated slightly higher than the maximum GC oven temperature to avoid condensation of higher-boiling analytes.

The Ion Source is an Electron Impact (EI) source. It has a cylindrical geometry, which ensures proper concentric alignment of the Ion Source elements. The sample enters the Ion Source from the GC interface. Electrons emitted by one of the filaments enter the Ion Source volume, which is at ground potential. A small permanent magnet focuses the electron beam. The electrons ionize and fragment the sample molecules. Positive and negative ions are generated. Positive ions are repelled from the Ion Source volume and pass through several electromagnetic lenses. These lenses concentrate the ions into a tight beam and direct them into the Mass Filter.

The Mass Filter allows selected ions from the Ion Source to reach the detector. When correctly tuned it filters and separates ions based on their mass to charge ratio. Only an ion of one mass can pass through the filter at a given time. Once the ions are inside the Mass Filter they experience the rapidly oscillating RF voltages. Ions which are not mass selected become synchronized with the oscillating RF field and are catastrophically accelerated into the rods.

The quadrupole Mass Filter may be operated in one of two modes. Scan or SIM (Selected Ion Monitoring). In SIM the Mass Filter is set to pass one kind of ion. This provides the greatest sensitivity and is used in quantitative applications. It is used when the analyst has prior to knowledge of what ions to expect. In Scan mode the Mass Filter is set to sequentially operate through a range of masses. It has lower sensitivity because most of the ion strike the rods during the scan.

An Ion Detector is used to collect and count ions or detect their resulting current. Only one mass to charge ion is detected at a given time. When the mass selected ion collides with the detector the resulting signal can be displayed by the data system.

The data system sends information to the Mass Spectrometer electronics. This determines in part how the inlet, ion source, mass filter detector and vacuum system operate. The data system also stores and collects the resultant signal from the ion detector. This information can be displayed real time or stored for later data processing.

The Ion Source, Mass Filter and Detector are in vacuum so that a collision of a given iion with another molecule such as air or water is unlikely. Minimizing collisions ensures that the ions are efficiently mass selected and detected. Common vacuum systems consist of two kinds of pumps. A high vacuum pump and a rough pump. Initially the rough pump pulls out most of the air/water and other gaseous species. The high vacuum pump acts to centrate the rarified high vacuum gasses which are subsequently removed by the rough pump making mass spectral analysis possible.

V. INTERFERENCES

A. Matrix / Chemical Interferences

By nature, GC/MS is designed to limit the misidentification of compounds, however there is a potential for improperly reporting "hits" that show up in the quantitation report. Therefore, all "hits" must be verified through a careful inspection of the spectra, by running it through the NIST library. Some compounds have almost identical spectra and similar elution times. These compounds can be mislabeled if the analysts is not careful in determining which compound is actually present. Some of these compounds pairs are listed as follows: *2,4-dichlorophenol & 2,6-dichlorophenol, 1-chloronaphthalene & 2-chloronaphthalene, 1-naphthalene & 2-naphthalene, 2,4,6-trichlorophenol & 2,4,5-trichlorophenol, Phenanthrene & Antraccene, Benzo(a) anthracene & chrysene, Benzo (b) fluoranthene & Benzo (k) fluoranthene.* These peak pairs can be distinguished using relative retention times if they appear as a single peak, assuming the elution order of the column has been previously determined by injecting the individual analytes.

Raw GC/MS data files from all blanks, samples, and spikes must be evaluated for interference. Determine if the source is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem. Phthalates are common lab contaminants for semi-volatiles. Phthalates and Adipates are plasticizers used to soften plastic and are found virtually everywhere. It is advisable to eliminate all sample contact with plastic being careful not to expose the glassware to any source of Phthalates, such as soap, etc.

VI. SAMPLING

A. Bottle Preparation: Aqueous samples should be collected in two, 1 Liter glass amber containers with Teflon lined caps. Solid samples should be collected in one 6 oz. glass soil jars with a Teflon lined cap. These quantities are sufficient for replicate analysis. Plastic must be avoided to prevent Phthalate contamination.

B. Preservation: Sodium Thiosulfate preservation is required if residual chlorine is present in the sample. Storage at 4 degrees centigrade is necessary for sample transport and storage.

C. Storage:

1. Refrigeration: When samples are not in use, that is during sample log in, sample preparation, etc. they must be kept refrigerated in the central laboratory cooler at 4 degrees centigrade.

D. Holding Times

1. Extraction: Aqueous samples must be extracted within 7 days of sample collection. Soil samples must be extracted within 14 days of sample collection.

2. Analysis: following extraction, both aqueous and solid samples have a 40 day holding time from the time of extraction until analysis.

VII. SAFETY

A. General Precautions: Many of the semi-volatile analytes are either suspected or known carcinogens and must be treated with great care. Analysts should handle these chemicals with Nitrile gloves and with proper ventilation. Skin contact should be avoided as well as inhalation.

1. Laboratory: When extracting samples, protective eye glasses must be worn at all times and methylene chloride vapors must be ventilated properly. Ventilating separatory funnels should be done under the canopy hoods, while KD concentration should be performed under the sash hoods. Analyst should where gloves and a lab coat when performing these operations.

2. Chemicals: Solvents should be stored in the ventilated explosion proof cabinet located in the extraction lab. Additional solvent is kept in the garage. Standards hould be stored in the refrigerator /freezer to minimize degradation. Some pesticides and the internal standard mixes can be kept at room temperature as long as they are kept in a sealed vial and shielded from light.

3. Personal: Wear eye glasses, gloves and lab coat when performing sample extractions. Loading the instrument requires eye protection.

VIII. EQUIPMENT AND MATERIALS

A. Instrumentation: GC-MSD system is by Hewlett-Packard, model 5972. The GC is the Hewlett-Packard model 5890, series II with electronic pressure control. Computer data station is the HP Vectra XM2/100i with the Ultra VGA 1280 monitor. Printer is the HP Laser Jet 4 Plus. The Rough Pump is the Edwards RV3 model. System includes the ChemStation and EnviroQuant Software for instrument control data acquisition and reduction.

B. Extraction/Preparation: PFTE boiling chips, Water Bath with heater control and concentric ring cover.

C. Glassware

1. Specification: 2 Liters separatory funnels with Teflon stopcock, 500 mL KD evaporation flask, 10 ml concentrator tubes, three ball snyder column, 2 mL glass vials, 1 Liter Graduated Cylinder.

2. Preparation: All glassware (except 3 ball Snyder Columns) must be washed in hot soapy water with S/P® Brand Laboratory Detergent Concentrate (Cat C6286-1 Baxter Scientific Products) followed with a final cold water rinse. Glassware is then rinsed with methanol to remove any water. All glassware to drip dry on the glassware racks in the extraction lab.

IX. REAGENTS AND STANDARDS

A. Reagent Purity Specifications

1. Analytical standards should be purchased from a reputable supplier (see list on last page). The certificate of analysis sheets must be kept on file so that all standards have a well documented paper trail.

B. Standards Preparation Directions

1. Initial Calibration Curve Setup: Prepare 5.0 mL of the nominal 80 ug/mL standard with the following Ultra Science Standards. This is for the typical NLS 8270B/625 method.

Inject (uL)	Ultra Mix
200	Mix 1 @ 2000 ng/ul
200	Mix 2 @ 2000 ng/ul
200	Mix 3 @ 2000 ng/ul
200	Mix 4 @ 2000 ng/ul
200	Mix 5 @ 2000 ng/ul
200	Mix 6 @ 2000 ng/ul
200	Mix 9 @ 2000 ng/ul
175	Pyridine @ 4615 ng/ul
100	Surrogates (all) @ 4000 ng/ul

a. Next perform the serial dilution off the 80 ng/uL into 4 additional levels as follows:

Calibration Level ng/uL	Amount of 80 std (uL)	Amount of DCM (uL)
80	200	0
60	150	50
40	100	100
20	50	150
10	25	175
4	10	190

Add 2 uL of Internal standard to each 200 ul volume Solution. Standards are now ready for injection.

2. Continuing Calibration Check Standard is prepared weekly, and is used to validate the initial calibration curve. Prepare 1.0 ml of the solution using the following NSI (second source) mixes. Final standard concentration is 40 ng/uL.

Inject (uL)	NSI Mix
20	Mix 2 @ 200 ng/uL
20	Mix 2 @ 2000 ng/uL
20	Mix 7 @ 2000 ng/uL
20	Mix 8 @ 2000 ng/uL
20	Mix 9 @ 2000 ng/uL
20	Mix 10 @ 2000 ng/uL
20	Mix 11 @ 2000 ng/uL
20	Mix 12 @ 2000 ng/uL
20	Mix 15 @ 2000 ng/uL
810	DCM
10	Surrogates (all @ 4000 ng/uL
10	Internal Standard @ 4000 ng/uL

3. Miscellaneous Standard Preparation:

   a. All 1.0 ml samples receive 10 uL of internal standard. One uL of IS per 100 uL of sample. Final Concentration of IS in solution is 40 ng/uL.

   b. DFTPP Tune Standard is prepared with 50 uL of the tune mix, plus 950 uL of DCM. Final Concentration of DFTPP in solution is 50 ng/uL.

   c. Surrogate Spike Mix is prepared with 1 ml of Acid, and 1 ml of Base Surrogate Mix diluted to 10 ml.

   d. MS/MSD and LCS use 1.0 ml TCL BNA Spike w/o Benzidines.

C. Storage Conditions: Most standards should be stored in the freezer of refrigerator and kept out of the light to prevent photochemical decomposition.

D. Shelf life: Typical shelf life for most stock Semi-volatile standards is one year. With typical usage, solutions will normally be used up within a year.

X. PROCEDURE

A. Extraction Preparation

1. Liquid Samples

   a. For liquid samples measure approximately 1 liter of the sample and transfer it quantitatively to the separatory funnel. Record this volume in the Sample Preparation Logbook. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic fee water to 1 liter. Add one ml of the surrogate standard to all samples, blanks and spikes. The resultant concentration for the base-neutral compounds should be 100 ng/uL while the final concentration for the acid extractable surrogate compounds should be 200 ng/uL. For the sample in each analytical batch selected for the spike (MS & MSD), add one ml of the spiking standard mix.

   b. Check the pH of the sample prior to extraction and adjust the pH according to the following determinative method: Acid extractables require pH < 2, while Basic extractables require pH > 11. The extraction sequence (i.e. Acid extraction first or Base extraction first) may depend upon the analytes of interest since some analytes are lost under strong basic conditions. Acidic conditions are created by adding several drops of Sulfuric Acid Solution (1:1 v/v). Basic conditions are created by adding several drops of 10 N NaOH solution.

   c. Add 60 mls of Methylene Chloride to the separatory funnel. Seal and shake the separatory funnel vigorously for 1 - 2 minutes with periodic venting (under the canopy hood) to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If an emulsion interface between the layers forms and is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. These technqieus may include stirring, filtration through glass wool, centrifugation, or other physical methods. Drain the solvent into a KD boiling flask through 10 cm of anhydrous sodium sulfate.

   d. Repeate the procedure two more times with 60 ml portions of Methylene Chloride.

2. Solid Samples:

3. For solid samples (see SW-846 method 3550A) the procedure is divided into two sections. For low level samples (< 20 mg/Kg) use a large sample size of 30 grams and a more rigorous extraction procedure. For higher concentration samples (> 20 mg/Kg) use a sample size of 2 grams with a less rigorous extraction procedure. The majority of

samples will employ the low concentration technique which will be addressed in this SOP. For adaptations to the high concentration technique consult Method 3550A of SW-846.

4. Weight approximately 30 g of sample into a 400 ml beaker. Record the weight to the nearest 0.1 g. Nonporous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate, may be added. After addition of sodium sulfate the sample should be free flowing. Add 1.0 ml of surrogate solution to all samples, spikes and blanks. For the sample selected for the MS and MSD add 1.0 ml of the spiking solution (see section IX, part B (3d)). Immediately add 100 ml of Methylene Chloride: Acetone to the beaker.

5. Next place the bottom surface of the tip of a 3/4 inch disrupter horn about 1/2 inch below the surface of the solvent but above the sediment layer. Extract ultrasonically for 3 minutes, with output knob at 10 (full power) and with mode switch on Pulse and percent duty knob set at 50% energy. Do not use microtip probes.

6. Decant the extract and filter it through Whatman NO. 41 filter paper (or equivalent) in a Buchner funnel that is attached to a clean 500 ml filtration flask. Repeat the procedure two more times with 100 ml portions of solvent. On the final ultrasonic extraction, pour the entire sample into the Buchner funnel and rinse with the extraction solvent. Sample extract is now ready for concentration.

B. Final sample preparation

1. Concentration Specifications: Add several PFTE boiling chips to the KD flask. Place the KD flask into the water bath at a temperature of 75 to 85 degrees F. Gently boil the solvent so that the balls "chatter" in the snyder chamber without flooding the chamber with solvent. Remove the KD flask when the solvent reaches appoximately 3 mls. Allow the flask to cool and drain back into the concentrator tube.

   a. If further concentration is needed <u>do not</u> use the Nitrogen Blowdown Technique as described in method 3510B. This technique will adersely effect spike recovery for the low boiling analytes. The Micro-Snyder Column technique is an alternate way to further concentrate (1 ml or less) the samples without loss of analytes. This technique involves the use of a small boiling flask (50 ml) connected to the same concentrator tube topped of with the micro-snyder column.

   b. Measure the final volume of solvent in the concentrator tube and record this volume in the <u>Sample Preparation Logbook.</u>

2. Dilution requirements: Sometimes a sample exhibits characteristics (strong petroleum odor & dark color) which suggest a dilution prior to analysis is advisable. If a sample requires dilution, simply remove an aliquot (with a syringe) of the final extract and dilute up to 1 ml with Methylene Chloride in an autosampler vial. Record the dilution factor in the <u>Sample Preparation Logbook</u> and the <u>Instrument</u>

Run Logbook. Store the undiluted extract in the freezer in the event the sample needs to be reanalyzed at a different dilution.

XI. INSTRUMENT ANALYSIS

A. GC/MS operating conditions:

Mass Range:	35 - 500 amu
Scan Time:	1.4 scan/sec
Initial Temperature:	40 degree C, hold 5 minutes
Temperature Program:	Ramp 10 C/min to 290, hold 6 minutes
Final Temperature:	Ramp 20 c/min to 325, hold 5 minutes
Injector Temperature:	250 degrees C
Source Temperature:	280 degrees C
Injector:	Split/Splitless
Sample Volume:	1 uL injection
Carrier Gas:	Helium at 36 cm/sec - EPC flow 1.0 ml/minute
Electron Multiplier:	3 minute solvent delay

The GC oven temperature program is a suggested program and will typically be used for most analysis. The mass spec settings should not be altered.

B. Tuning the MSD

1. The GC/MS must pass the tune specifications listed below by injecting 50 mg of DFTPP (decafluorotriphenylphosphine).

Mass	Ion Abundance Criteria
51	30-60 % of mass 198
68	< 2 % of mass 69
70	< 2 % of mass 69
127	40 - 60% of mass 198
197	< 1 % of mass 198
198	Base Peak, 100 % relative abundance
199	5 - 9% of mass 198
275	10 - 30 % of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40 % of mass 198
443	7 - 23 % of mass 442

2. This criteria must be demonstrated each 12 hour shift. In other words if the tune passes you may proceed with the analysis for up to 12 hours. The last accepted sample must be injected within 12 hours

of the time posted on the DFTPP report. All methods should use the DFTPP.U file as the tune file. If the tune fails repeatedly with a fresh DFTPP standard the target tune relative abundances may have to be changed or the source may be getting dirty and it is time for cleaning.

3. To reset the target tune abundances use the following procedure. Under MSTOP menu go into TUNEMS. Click on TARGET TUNE. Select TUNE. Then select LOAD TUNE VALUES and select the DFTPP.TGT file. Next, select SET TUNE VALUES. Enter in a relative abundance for the 131 Ion, 219 Ion and the 502 Ion. Once these abundances are set select SAVE TUNE TARGETS to DFTPP.TGT. Now select DFTPP TUNE with these new values. The mass spec will go through its tune routine and generate a report. Before exiting TUNE, save the tune values just established from the last DFTPP tune. Now reinject the DFTPP standard under these new settings to see if it will meet the necessary tune specifications. If the tune still does not pass try tuning again with different target tune abundances.

   Some suggested settings for the relative abundance:

ION	% RELATIVE ABUNDANCE
131	24, or 25
219	26, or 27
502	0.8

4. If after several iterations, the DFTPP standard will still not pass it may be time to replace the injection port liner and gold seal or the Ion SOurce may need cleaning. Before cleaning the source, reference HP 5972A MSD Hardware Manual and watch the video "Cleaning the HP 5972A Ion Source".

C. Calibration:

1. Method 8270B requires a five point calibration curve while Method 625 requires only a three point curve. In order to meet the most stringent requirements between the two methods the calibration curve should consist of 5 calibration points with the lowest concentration near the detection limit.

2. There are four compounds called System Performance Check Compounds (SPCCs). These compounds must have a minimum average response factor greater than 0.05. These compounds tend to decrease in sensitivity as the chromatographic system deteriorates or the standard material degrades. If the minimum response factors are not met the system

must be evaluated, and corrective action must take place before sample analysis begins. The four SPCC compounds are:

N-nitroso-di-n-propylmaine
Hexachlorocyclopentadiene
2,4-Dinitro-phenol
4-Nitrophenol

3. Once the SPCC compounds have met specification of the thirteen Calibration Check Compounds (CCCs) must be evaluated. The percent relative standard deviation % RSD of the relative response factor of the five calibration points must be less than 30% for each CCC. The CCC compounds are:

   Acenapthene
   1,4-Dichlorobenzene
   Hexachlorobutadiene
   N-Nitrosodiphenylmaine
   Di-n-octyl phthalate
   Fluoranthene
   Benzo (a) pyrene
   4-Chloro-3-methylphenol
   2,4-Dichlorophenol
   2-Nitrophenol
   Phenol
   Pentachlorophenol
   2,4,6-Trichlorophenol

4. All other compounds should have an RF %RSD less than 15 %. If the %RSD of any compound is 15 % or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD of any compound is greater than 15 %, a first or higher order regression fit of the five calibration points may be used for A/A_{is}) vs concentration. This is a recommended alternative to average response factor calibration.

D. Sample/Standard Presentation to Instrument

1. All sample extracts must be delivered to the 1.8 ml glass autosampler vials and labeled with a complete description. All samples must be injected with 1 uL of the 4000 ng/uL internal standard mix for every 100 uLs of sample. For example, 1.0 ml of sample would receive 10 ul of the 4000 ng/uL internal standard mix for a final sample concentration of 40 ng/uL.

Internal Standard Compounds

Acenaphthene-d10
Chrysene-d12
1,4-Dichlorobenzene-d4
Naphthalene-d8
Perylene-d12
Phenathrene-d10

The resultant sample/extract concentration is 40 ng/uL for the internal standard compounds.

XII. CALCULATIONS

A. General Calculations

1. Response Factor: $RF = (A_s C_{is})/(A_{is} C_s)$

Where:
A_s = Area response for the analyte's Quant Ion
C_{is} = Concentration of the internal standard (ug/L)
A_{is} = Area response for the internal standard Quant Ion
C_s = Concentration of the analyte (ug/L)

2. Final Extract Concentration: $Cex = (A_x C_{is})/(A_{is} RF_{avg})$

Where:
A_s = Area response for the analyte's Quant Ion
C_{is} = Concentration of the internal standard (ug/L)
A_{is} = Area response for the internal standard Quant Ion
RF_{avg} = Average response factor of the initial calibration
C_{ex} = Final concentration in the extract (ug/ml)

3. Final Liquid Concentration: $Cf = (D_1 C_{ex} V_{ex})/Vi$

Where:
C_f = Final concentration in the sample (ug/L)
D_1 = Dilution of the extract (unitless)
C_{ex} = Final concentration in the extract (ug/ml)
V_{ex} = Final volume of extract (ml)
V_i = Initial volume of the sample (L)

4. Final Soil Concentration: $C_f = (D_1 C_{ex} V_{ex})/(M_i S)$

Where:
C_f = Final concentration in the sample (ug/Kg DWB)
D_1 = Dilution of the extract (unitless)
C_{ex} = Final concentration in the extract (ug/ml)
V_{ex} = Final volume of extract (ml)
M_i = Initial Mass of the sample (L)
S = Decimal equivalent of the percent solid content of the soil

B. Significant figures:

1. Sample results should be reported to two significant figures except for all Blind and Performance Evaluation studies which require 3 significant figures. The results entered into the template will automatically be rounded to the specified number in the reporting template.

C. Special Adjustments for Samples:

1. Sample Size: Typical liquid sample volume is 1 Liter. Smaller volumes may be used if necessary but this will elevate the final sample detection limit. Typical soil size is 30 grams, however a smaller size is permissible if desired. Smaller sample size will elevate the final detection limit.

2. Sample Matrix: If the matrix is an oily liquid employ the waste dilution procedure in accordance with EPA 3580.

XIII. QUALITY CONTROL

A. Standards:

1. Standards should be purchased from a reliable supplier (some listed on last page) and accompanied with a certificate of analysis which verifies the sample concentration for a specific lot number. The certificate of analysis must be kept in a file for a complete record of the standard preparation. Lot numbers and labels should be recorded and attached in the Standard Preparation Logbook. When standards are prepared from a stock standard they must be labeled properly with the date, concentration and description of the mix.

B. Lab Control Standard:

1. There are two classifications of laboratory Quality Control Standards. The first classification called a calibration verification standard is designed to ensure accuracy at the instrument level the calibration verification standard is a "second source" standard with a concentration mid-level in the calibration curve and is used to check both the stability of the initial calibration and the accuracy of the individual components. This dual purpose ensures that the calibration is valid at the time of analysis and that the standards are at the level stated by the supplier. Second Source Verification is not required by 8270B/625 and may be left up to analysts discretion.

2. The second classification of laboratory Quality Control Standard is called a method performance verification standard and is used to determine the extraction efficiency of various compounds for a given extraction batch. The method performance verification standard ensures that the process prior to analysis are functioning properly and are producing their intended result. The matrix performance verification standard is called a Matrix Spike (MS) or Matrix Spike Duplicate (MSD) or can also be called a Laboratory Control Standard (LCS).

3. Both the MS and MSD are samples (soil or water) spiked with a known amount of analyte and extracted along with the other samples within the extraction batch. The result of the MS and MSD provide a measure of the accuracy and precison of the extraction batch. The accuracy is expressed as percent recovery and the precision is expressed as percent difference. An LCS is not required by the method but is a good indicator of extraction performance apart from any matrix affects. An LCS is simply laboratory reagent water spike with a known amount of analyte and is the truest measure of the overall performance of the method. An LCS is a very useful tool in troubleshooting the method.

4. An extraction batch is defined as twenty extractions and should always have one Blank, MS and MSD. An LCS is optional. In cases where there is not enough sample available to perform an MS/MSD an LCS should be substituted.

C. Sample QC

1. Accuracy measurements (spike percent recovery):

   a. QC spike acceptance criteria is defined by the upper and lower control limits established by the MS/MSD. Once a minimum of 30 sample points are collected the acceptance range is determined as follows: (p: percent recovery, s_p: standard deviation of percent recovery).

$$\text{Upper Control Limit} = p + 2s_p$$

$$\text{Lower Control Limit} = p - 2s_p$$

2. Precision measurements (duplicate - relative percent difference)

3. Surrogate measurements (percent recovery range)

   a. Surrogate spike acceptance criteria is defined by the upper and lower control limits established by the samples analyzed. Oncce a minimum of 30 sample points are collected the acceptance interval is determined as follows: (p: percent recovery, s_p: standard deviation of percent recovery).

$$\text{Upper Control Limit} = p + 3s_p$$

$$\text{Lower Control Limit} = p - 3s_p$$

D. Analytical Limitations:

1. Sample Interference: Phthalate Esters are the most common interferent and are associated with plastics. Other sources of artifacts may be the reagents, standards and glassware. Use high purity gasses and reagents and be careful to rinse glassware properly to avoid sample contamination. Also it is important to use good laboratory technique when preparing standards or samples in order to minimize introduction of unwanted artifacts. Syringes should be triple rinsed after exposure to standards or sample concentrates.

2. Instrument Limitations: Lower quantitative limit is defined by the LOQ. Analytes may be detected at or near the MDL and should be considered the lowest detectable concentration. The upper quantitative limit is defined by the highest calibration standard in the initial calibration. Estimated sample concentrations may be determined above the highest point on the curbe but should only be used to gauge the necessary dilution for the re-analysis.

   Qualitative limitations are defined by the temperature range of Semi-Volatile compounds, the calibration curve and the NIST library.

E. Method Validation: Each analyst performing this analysis must successfully complete the initial demonstration of proficiency requirement stated in the method. This involves the extraction and analysis of 4 reagent water samples spiked with a known amount of analyte. The final numbers should be compared to the accuracy and precision tables in the method to determine if the analyst has successfully meth the minimum performance criteria. In addition the laboratory QA officer will issue Blind Performance Evaluation samples to test the analysts proficiency. These Blind studies usually coincide with State and Federal Performance Evaluation studies conducted periodically for various monitoring programs.

F. MDL study procedure: Each analyte reported should have a Method Detection Limit (MDL) statistically computed according to 40 CFR, Part 136, Apdx B, Rev 1.1. MDLs should be performed at least yearly or whenever significant changes are made to the instrument impacting MSD sensitivity.

XIV. RECORDS AND REPORTING DATA

A. Benchsheet entries: See attachment VI for an example of the benchsheet used. Benchsheets may serve as a work order but are not necessarily filled out. The Benchsheet should accompany all the raw data in the file folder.

B. Units / Significant figures

1. Liquid samples are reported as ug/L at 2 significant figures.

2. Solid samples are reported as ug/Kg DWB at 2 significant figures.

C. Detection limits and Reporting limits: All detection limits are determined according to 40 CFR, Part 136, Apdx B, Rev 1.1. If the MDL is not determined by this procedure for a given compound a conservative estimate may be used in the interim. This estimate should be noted on the final report. For additional references on MDL determination see <u>Analytical Detection Limit Guidance, WDNR OTS, April 1995 draft</u>. See attachment II for a complete listing of individual MDLs and LOQs.

D. Qualifiers or comments used if data is to be flagged:

1. Samples must be flagged with a specific comment if any of the following situations arise and are not corrected through a sample rerun, re-extraction or recalibration.

   a. Contaminated Extraction Blank.

b. Surrogate Recovery out of range.

c. Holding time exceeded.

d. MS/MSD spike recovery exceedance.

e. Continuing Calibration out of range.

f. CCC & SPCC compounds outside tolerance.

E. LIMS Entry

1. The laboratory information system contains all of the data which appears on the final analytical reports. Results are entered into the LIMS system under results entry (RE). Simply enter the appropriate test code, initials under who, date and time under date and then page down. This will call up all the samples under that test code which do not have results entered. Under units, enter "see attached". If there are any footnotes, comments or qualifiers enter this information under the notes categroy. Shift F9 will allow you to see the entire field as you type. Once the correct information is entered save it by entering F4.

2. Now the data must be entered into the templates. To do this go into organic reports. Select enter results. This menu will prompt you for the Template Name, Project Number, Sample Number and Dilution. Enter the information into these fields and enter Page Down. This will allow you to enter in the final numerical result for the appropriate compound. The dilution factor will not adjust the result entered but only the MDL/LOQ on the final report.

F. Client Reports:

1. After all the data is entered, return to the previous menu and select option (3) to print results on the final report. If printing soil results select (3s). To print final results select option (10).

G. Data archiving or filing:

1. Archiving of HP-MSD should be performed on a regular basis. If the hard drive fills up while acquiring data, "bad things" happen to the software because it is not properly shut down. If this happens it may be necessary to reload all of the software. Make certain that there is enough space for data acquisition. Data files are either purged from the system or they are transferred to drive W, (ALTOS® hard drive) via the network. Since the HP-MSD data files are large it is advisable to contact the LIMS manager to see if there is enough room on drive W before transferring files.

2. Data files are transferred using **FILE MANAGER** utility located in the main window. To transfer files from c:\hpchem\1\data to w:\archive\svgc, simply highlight the data directory to be moved and strike the **F8** key. This will prompt you for the destination, which is: W:\archive\svgc. Once the files have been transferred, confirm it by checking the destination directory. If the transfer was successful, then return to drive C; and delete the highlighted files using the **DELETE** key.

H. Hard Copy Archiving: Processed data reports are printed for each sample analyzed. A copy of the final report should be stapled to the processed report. All data compiled from an instrument run should be kept in separate file folder. Information kept in the file folder includes, the DFTPP Tune report, Continuing Calibration Summary, MS & MSD reports (if analyzed), extraction and instrument blanks, and all sample data. The file folder should be labeled with the date of the analytical run and stored in the vertical file cabinet located in the laboratories central office area. Periodically, date will be moved to the basement archives as the file cabinet fills up.

I. Instrument Run Logbook: See attachment III for an example of the instrument run logbook. The run logbooks must be filled out for each analytical run. If only DFTPP is run for that day, it is not necessary to record it in the logbook since an electronic record is available.

J. Sample Preparation Logbook: See attachment V for an example of the Standard Preparation Logbook. The standard preparation logbook is filled out every time a new standard solution is opened or prepared. If an adhesive label is available it should be attached to the page container the information about the standard prepared. This label is then traceable to the certificate of analysis for a complete record.

K. Maintenance Logbook: The maintenance logbook is a ring binder notebook with 5 divisions: INT STD contains internal standard information, MAINTENANCE contains the record of all maintenance activities, INTEGRATION contains specific peak integration instructions, FILES contains all of the miscellaneous file names and their descriptions, 502 ION contains a running record of the 502 abundance and EM voltages for various tunes. This information should be kept up to date for an accurate record of the instrument performance overtime.

XV. CLEAN UP

A. Lab Work Area: Samples should always be returned to the walk-in cooler after use. Also, any materials, reagents, supplies or standards used throughout the day should be returned to their proper location. The lab area in general should always be kept clean and free from clutter.

B. Sample Disposal & Standard Disposal: Samples are disposed of in accordance with procedures determined in the QA/QC manual. Soil samples are left in their glass jars and directly discarded into the trash. Liquid samples are emptied down the drain and the glass amber containers are placed in a storage area in the old lab. Glass amber bottles are not reused. Standards and sample extracts are emptied from the 2 ml vials into a solvent waste jug designated for GC/MS Semi-Volatile samples. The glass vials are discarded into a glass jug that is later disposed. The solvent and standard contents will be removed from the premises by a waste hauler.

C. Equipment / Glassware: Equipment should be kept clean and dust free. Occasional cleaning may be needed to ensure equipment is free from dust and other debris. Also, glassware should be cleaned as soon as possible after use. If immediate cleaning is not possible, all used glassware should be left to soak in a tub of hot soapy water until it can be cleaned.

XVI. MAINTENANCE / TROUBLESHOOTING

A. Preventive maintenance procedures and frequency

1. Check daily the vacuum manifold pressure - Should read about 4.5 x 10^{-5} TORR.
2. Check weekly the Rough Pump Oil Level.
3. Check weekly the pressure of the Carrier Gas (He).
4. Check monthly and refill the CAL gas vial as necessary.
5. Replace every six months the Rough Pump Oil.
6. Replace every six months the Rough Pump Trap Pellets.
7. Check yearly and replace if necessary the Diffusion Pump oil.
8. As needed clean the Ion Source.
9. As needed check the oxygen Carrier Gas Trap and replace.
10. As needed replace the Filament and Multiplier.
11. As needed (100 injections) replace the injector septa.
12. As needed replace injection port glass insert & O-ring, gold seal & washer.
13. As needed export files to main system hard drive - see XIV (G).

B. Troubleshooting procedures: See Chapter 3 of the Hewlett-Packard 5972A Hardware Manual.

XVII. ATTACHMENTS

A. Attachment I: Semi-Volatile parameter list.

B. Attachment II: Method detection limits and Limits of quantitation.

C. Attachment III: Instrument Run Logbook.

D. Attachment IV: Sample Preparation Logbook.

E. Attachment V: Standard Preparation Logbook.

F. Attachment VI: Benchsheets.

XVIII. MISCELLANEOUS INFORMATION

A. Service Phone Numbers:

1. For questions on part numbers & pricing for HP supplies call (916) 783-0804.
2. For technical questions as to what the parts do and what parts are necessary for HP systems call (800) 424-9759.
3. General HP number (800) 227-9770 Purchases, etc.
4. For Technical Support/Service Calls contact LINC Quantum Analytics (800) 458-9641 or AIS (Analytical Instrumentation Service, Inc.) (708) 739-4135, ask for Greg Bedenk.

B. Special HP Part Numbers

1. Injector Inserts: 5062-3587, these are precleaned and deactivated.
2. On-column Needles: 5182-0832, these needles attach to the syringe barrel and are used for automatic on-column injections. A 0.530 mm precolumn must be joined to the 0.25 mm ID capillary column.
3. Ferrule for Injector: 5181-3323, these 10% graphite, 90% Vespel®, and are for 0.25 mm column ID, comes as a package of 10.
4. Ferrule for Detector Inlet: 5062-3508, these are 15% graphite, 85% Vespel®, and are for 0.25 mm Column ID, comes as a package of 10.
5. Gold Plated Seal for Injector: 18740 - 20885.
6. Washer for Injector (12/pk): 5061-5869.

C. Standard Supplier Telephone Numbers:

1. Ultra Scientific:	(800) 338-1754
2. NSI:	(800) 234-7837
3. ChemService:	(800) 452-9994
4. Restek:	(800) 356-1688

D. Equipment Model and Serial Numbers: Installation & Warranty Start Date: 08/08/95.

Model #	Serial Number	Description
5972A	3501A02657	Mass Spec
5890E	3336A59178	Gas Chromatograph
D3233A	3318A66831	Vectra Xm2 4/100i
D2807A	JP50807564	Ultra VGA 1280 Monitor
C2037A	JPGL072537	LaserJet 4 Plus
G1512A	3529A02405	ALS Controller
18596M	3522A38797	ALS Tray
18593B	3529A43280	ALS Tower
59864A	342010	ION Gauge Controller
RV3	03594	Rough Pump
G1033A	3235A23472	Environquant Software
G1034C	N/A	MS Software

E. Miscellaneous Installation Instructions:

1. Split/Splitless Injector Column Installation: Before slipping the column nut and ferrule over the column, force the column to serve as a holder and positioner for installation. Next, slide column nut and ferrule (HP 5181-3323, 10% graphite, 90% Vespel®) onto the RESTEK Rtx-5 MS, 0.025 mm ID column (with fused guard column). Cut the column cleanly with 4 to 6 mm extending past the ferrule. Insert the column nut into the injector inlet fitting and hand tighten being careful not to move the position of the column. Tighten with a wrench.

2. Y Vu-Union Connector: The end of the column is connected to the Y Vu-Union® Connector (restek cat.% 20432, glass insert cat. # 20433). This Y configuration allows for the SDWA column from injector B to be joined to the A injector column which is then connected to the MSD inlet. The ferrules used for this connector must be graphite (HP 5080 - 8853). Installation is as follows:

    a. Slide the knurled nut and the ferrule onto the column ends. Install the ferrule so that the tapered end faces outwards toward the nut.

    b. Cut the column end so that the ends are cut squarely. This will ensure a gas-tight connection.

    c. Push the column ends into the glass insert taper. Leave a 1/16 inch gap between the column end and the tapered region before the ferrules are preseated.

    d. To preseat the ferrules, finger-tighten the knurled nut until the column does not slide when gentle pressure is applied. The ferrules are now preseated and properly conformed to fit the connector cavity.

e. Loosen the knurled nuts and gently push each column end into the glass insert taper until it makes contact. Tighten each knurled nut (being careful not to overtighten) until they are tight against the ferrule. The presence of a uniform brown ring circling the entire column at the tapered region indicates a proper connection.

f. Use the "S" hook to hang the Y Vu-Union® Connector from the cage support in a manner which will not place strain against the fused silica tubing.

Attachment I

Parameter Number of compounds	CAS Number 254	8270A 250	Apdx IX 127	625 81	525.1 43	CLP SVOA 64	CAL STD 127	NLS LIST 100	TCLP 18	ULTRA MIX # 127	NSI Mix 127	NSI LCS 69
1,2,4,5-tetrachlorobenzene	95-94-3	*	*				*	*		5 (H-1737)	2	
1,2,4-trichlorobenzene	120-82-1	*	*	*		*	*	*		3 (H-1520)	8	*
dichlorobenzene	95-50-1	*	*	*		*	*	*		1 (H-0371)	8	*
1,2-diphenylhydrazine	122-66-7	*	*				*			9 (H-1644)		
1,3,5-trinitrobenzene	99-35-4	*	*				*			12 (H-0298)	2	
1,3-dichlorobenzene	541-73-1	*	*	*		*	*	*		1 (H-0371)	8	*
1,4-dichlorobenzene	106-46-7	*	*	*		*	*	*		1 (H-0371)	8	*
1,4-naphthoquinone	130-15-4	*	*				*			10 (H-1806)	5	
1-chloronaphthalene	90-13-1	*	*				*	*		5 (H-1737)	2	
1-naphthylamine	134-32-7	*	*				*	*		5 (H-1737)	15	
2,3,4,6-tetrachlorophenol	58-90-2	*	*				*	*		5 (H-1737)	7	
2,4,5-trichlorophenol	95-95-4	*	*			*	*	*	*	5 (H-1737)	10	*
2,4,6-trichlorophenol	88-06-2	*	*	*		*	*	*	*	5 (H-1737)	10	*
2,4-dichlorophenol	120-83-2	*	*	*			*	*		3 (H-1520)	10	*
2,4-dimethylphenol	105-67-9	*	*	*		*	*	*		3 (H-1520)	10	*
2,4-dinitrophenol	51-28-5	*	*	*		*	*	*		5 (H-1737)	10	*
2,4-dinitrotoluene	121-14-2	*	*	*		*	*	*	*	5 (H-1737)	8	*
2,6,-dichlorophenol	87-65-0	*	*			*	*	*		3 (H-1520)	7	*
2,6-dinitrotoluene	606-20-2	*	*	*		*	*	*		5 (H-1737)	8	
2-acetylaminofluorene	53-96-3	*	*				*			10 (H-1806)	2	
2-chloronaphthalene	91-58-7	*	*	*		*	*	*		5 (H-1737)	8	*
2-chlorophenol	95-57-8	*	*	*		*	*	*		1 (H-0371)	10	*
2-methyl-4,6-dinitrophenol	534-52-1		*	*			*	*		6 (H-1639)	10	*
2-methylnaphthalene	91-57-6	*	*			*	*	*		3 (H-1520)	8	*
2-methylphenol	95-48-7	*	*			*	*	*	*	1 (H-0371)	10	*
2-naphthylamine	91-59-8	*	*				*	*		5 (H-1737)	15	
2-nitroaniline	88-74-4	*	*			*	*	*		5 (H-1737)	11	*
2-nitrophenol	88-75-5	*	*	*		*	*	*		3 (H-1520)	10	*
2-picoline	109-06-8	*	*				*			1 (H-0371)	5	
3,3'-dichlorobenzidine	91-94-1	*	*	*		*	*	*		2 (H-0325)	6,13	
3,3'-dimethylbenzidine	119-93-7	*	*				*			8 (H-1733)	13	
3-methylcholanthrene	56-49-5	*	*				*			4 (J-0408)	1	
3-nitroaniline	99-09-2	*	*			*	*	*		5 (H-1737)	11	*
4 methylphenol	106-44-5	*	*			*	*	*	*	1 (H-0371)	10	*
ninobiphenyl	92-67-1	*	*				*	*		6 (H-1639)	2	
4-bromophenyl phenyl ether	101-55-3	*	*	*		*	*	*		6 (H-1639)	12	*
4-chloro-3-methylphenol	59-50-7	*	*	*		*	*	*		3 (H-1520)	10	*
4-chloroaniline	106-47-8	*	*			*	*	*		3 (H-1520)	11	*

Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
4-chlorophenyl phenyl ether	7005-72-3	*	*	*		*	*	*		5 (H-1737)	12	*
4-nitroaniline	100-01-6	*	*			*	*	*		5 (H-1737)	11	*
4-nitrophenol	100-02-7	*	*	*		*	*	*		5 (H-1737)	10	*
-nitroquinoline-1-oxide	56-57-5	*	*				*			8 (H-1733)	5	
-nitro-o-toluidine	99-55-8	*	*				*			8 (H-1733)	2	
7,12-dimethylbenzo[a]anthracene	57-97-6	*	*				*			4 (J-0408)	1	
a,a-dimethylphenethylamine	122-09-8	*	*				*			3 (H-1520)		
acenaphthene	83-32-9	*	*	*		*	*	*		5 (H-1737)	9	*
acenaphthylene	208-96-8	*	*	*	*	*	*	*		5 (H-1737)	9	*
acetophenone	98-86-2	*	*				*			3 (H-1520)	5	
aniline	62-53-3	*	*				*	*		1 (H-0371)	11	*
anthracene	120-12-7	*	*	*	*	*	*	*		6 (H-1639)	9	*
aramite (total)	140-57-8	*	*				*			7 (G-0415)	0	
benzidine	92-87-5	*	*	*			*	*		2 (H-0325)	6,13	
benzoic acid	65-85-0	*	*				*	*		3 (H-1520)	7	*
benzo[a]pyrene	50-32-8	*	*	*	*	*	*	*		4 (J-0408)	9	*
benzo[b]fluoranthene	205-99-2	*	*	*	*	*	*	*		4 (J-0408)	9	*
benzo[ghi]perylene	191-24-2	*	*	*	*	*	*	*		4 (J-0408)	9	*
benzo[k]fluoranthene	207-08-9	*	*	*	*	*	*	*		4 (J-0408)	9	*
benzyl alchohol	100-53-3	*	*				*	*		1 (H-0371)	8	*
benz[a]anthracene	56-55-3	*	*	*	*	*	*	*		2 (H-0325)	9	*
bis(2-chloroethoxy)methane	111-91-1	*	*	*		*	*	*		3 (H-1520)	12	*
bis(2-chloroethyl) ether	111-44-4	*	*	*		*	*	*		1 (H-0371)	12	*
bis(2-chloroisopropyl) ether	108-60-1	*	*	*		*	*	*		1 (H-0371)	12	*
bis(2-ethylhexyl)phthalate	117-81-7	*	*	*	*	*	*	*		2 (H-0325)	12	*
butyl benzyl phthalate	85-68-7	*	*	*	*	*	*	*		2 (H-0325)	12	*
chlorobenzilate	510-15-6	*	*				*			7 (G-0415)	7	
chrysene	218-01-9	*	*	*	*	*	*	*		2 (H-0325)	9	*
di-n-butyl phthalate	84-74-2	*	*	*	*	*	*	*		6 (H-1639)	12	*
di-n-octyl phthalate	117-84-0	*	*	*		*	*	*		4 (J-0408)	12	*
diallate (total)	2303-16-4	*	*				*			7 (G-0415)	5	
dibenzofuran	132-64-9	*	*			*	*	*		5 (H-1737)	8	*
dibenzo[a,h]anthracene	53-70-3	*	*	*	*	*	*	*		4 (J-0408)	9	*
dibenzo[a,j]acridine	224-42-0	*	*				*			4 (J-0408)		
diethyl phthalate	84-66-2	*	*	*	*	*	*	*		5 (H-1737)	12	*
dimethoate	60-51-5	*	*				*			7 (G-0415)	4	
imethyl phthalate	131-11-3	*	*	*	*	*	*	*		5 (H-1737)	12	*
dinoseb	88-85-7	*	*				*			7 (G-0415)	7	
diphenylamine	122-39-4	*	*				*	*		9 (H-1644)	15	
disulfoton	298-04-4	*	*				*			7 (G-0415)	4	

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Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
ethyl methanesulfonate	99-35-4	*	*				*			12 (H-0298)	3	
famphur	52-85-7	*	*				*			7 (G-0415)	4	
fluoranthene	206-44-0	*	*	*		*	*	*		6 (H-1639)	9	*
uorene	86-73-7	*	*	*	*	*	*	*		5 (H-1737)	9	*
exachlorobenzene	118-74-1	*	*	*	*	*	*	*	*	6 (H-1639)	8	*
hexachlorobutadiene	87-68-3	*	*	*		*	*	*	*	3 (H-1520)	8	*
hexachlorocyclopentadiene	77-47-4	*	*	*	*	*	*	*		5 (H-1737)	8	*
hexachloroethane	67-72-1	*	*	*		*	*	*	*	1 (H-0371)	8	*
hexachlorophene	70-30 4	*	*				*			10 (H-1806)	2,14	
hexachloropropene	1888-71-7	*	*				*			10 (H-1806)	2	
indeno[1,2,3-cd] pyrene	193-39-5	*	*	*	*	*	*	*		4 (J-0408)	9	*
isodrin	465-73-6	*	*				*			10 (H-1806)	2	
isophorone	78-59-1	*	*	*		*	*	*		3 (H-1520)	8	*
isosafrole	120-58-1	*	*				*			10 (H-1806)	5	
kepone	143-50-0	*	*				*			7 (G-0415)	2	
m-dinitrobenzene	99-65-0	*	*				*			10 (H-1806)	2	
methapyrilene	91-80-5	*	*				*			10 (H-1806)	5	
methyl methanesulfonate	62-50-0	*	*				*			12 (H-0298)	3	
N-nitorsodi-n-propylamine	621-64-7	*	*	*		*	*	*		1 (H-0371)	12	*
N-nitrosodi-n-butylamine	924-16-3	*	*				*	*		3 (H-1520)	15	
N-nitrosodiethylamine	55-18-5	*	*				*			8 (H-1733)	15	
N-nitrosodimethylamine	62-75-9	*	*	*			*	*		1 (H-0371)	12	*
N-nitrosodiphenylamine	86-30-6	*	*	*		*	*	*		9 (H-1644)	12	*
N-nitrosomethylethylamine	10595-95-6	*	*				*			8 (H-1733)	15	
N-nitrosomorpholine	59-89-2	*	*				*			8 (H-1733)	15	
N-nitrosopiperidine	100-75-4	*	*				*	*		3 (H-1520)	15	
N-nitrosopyrrolidine	930-55-2	*	*				*			8 (H-1733)	15	
naphthalene	91-20-3	*	*	*		*	*	*		3 (H-1520)	9	*
nitrobenzene	98-95-3	*	*	*		*	*	*	*	3 (H-1520)	8	*
O,O,O-triethyl phosphororthioate	126-68-1	*	*				*			10 (H-1806)	4	
o-toluidine	95-53-4	*	*				*			8 (H-1733)	2	
p-(dimethylamino)azobenzene	60-11-7	*	*				*	*		2 (H-0325)	15	
parathion (methyl)	298-00-0	*	*				*			7 (G-0415)	4	
parathion (ethyl)	56-38-2	*	*				*			7 (G-0415)	4	
pentachlorobenzene	608-93-5	*	*				*	*		5 (H-1737)	2	
pentachloronitrobenzene	82-68-8	*	*				*	*		9 (H-1644)	2	
entachlorophenol	87-86-5	*	*	*	*	*	*	*	*	6 (H-1639)	10	*
phenacetin	62-44-2	*	*				*			9 (H-1644)	5	
phenanthrene	85-01-8	*	*	*	*	*	*	*		6 (H-1639)	9	*
phenol	108-95-2	*	*	*		*	*	*		1 (H-0371)	10	*

Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
phorate	298-02-2	*	*				*			7 (G-0415)	4	
pronamide	23950-58-5	*	*				*			9 (H-1644)	5	
pyrene	129-00-0	*	*	*	*	*	*	*		2 (H-0325)	9	*
ole	94-59-7	*	*				*			10 (H-1806)	5	
thionazin	297-97-2	*	*				*			7 (G-0415)	4	
2,4-D	94-75-7		*				*			7 (G-0415)		
p-phenylenediamine	160-50-3		*				*			8 (H-1733)	15	
silvex (2,4,5-TP)	93-72-1		*				*			7 (G-0415)		
sulfotepp	3689-24-5		*				*			7 (G-0415)	4	
1-Acetyl-2-thiourea	591-8-2	*										
Alachlor	15972-60-8				*							
Aldrin	309-00-2	*		*	*							
2-Aminoanthraquinone	117-79-3	*										
Aminoazobenzene	60-9-3	*										
Anilazine	101-05-3	*										
o-Anisidine	90-04-0	*										
Aroclor-1016	12674-11-2	*		*								
Aroclor-1221	11104-28-2	*		*								
Aroclor-1232	11141-16-5	*		*								
Aroclor-1242	53469-21-9	*		*								
Aroclor-1248	12672-29-6	*		*								
Aroclor-1254	11097-69-1	*		*								
Aroclor-1260	11096-82-5	*		*								
Atrazine	1912-24-9				*							
Azinphos methyl	86-50-0	*										
Barban	101-27-9	*										
p-Benzoquinone	106-51-4	*										
a-BHC	319-84-6	*		*								
b-BHC	319-85-7	*		*								
d-BHC	319-86-8	*		*								
g-BHC (Lindane)	58-89-9	*		*	*				*			
Bromoxynil	1689-84-5	*										
Captafol	2425-06-1	*										
Captan	133-06-2	*										
Carbaryl	63-25-2	*									11	*
Carbazole	86-74-8					*						
bofuran	1563-66-2	*										
Carbophenothion	786-19-6	*										
a-Chlordane	5103-71-9				*							
Chlordane	57-74-9	*		*					*			

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Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
g-Chlordane	5103-74-2				*							
trans-Nonachlor-Chlordane	39765-80-5				*							
Chlorfenvinphos	470-90-6	*										
Chloro-2methylaniline	95-79-4	*										
2-chlorobiphenyl	2051-60-7				*							
3-(chloromethyl) pyridine hydrochloride	6959-48-4	*										
Coumphos	56-72-4	*										
p-Cresidine	120-71-8	*										
Crotoxyphos	7700-17-6	*										
2-Cyclohexyl-4,6-dinitrophenol	131-89-5	*										
4,4'-DDD	72-54-8	*		*				!				
4,4'-DDE	72-55-9	*		*				!				
4,4'-DDT	50-29-3	*		*				!				
Demeton-o	298-03-3	*										
Demeton-s	126-75-0	*										
2,4-Diaminotoluene	95-80-7	*										
Dibenzo(a,e)pyrene	192-65-4	*										
Dichlone	117-80-6	*										
2,3-Dichlorobiphenyl	16605-91-7				*							
Dichlorovos	62-73-7	*										
Dicrotophos	141-66-2	*										
Dieldrin	60-57-1	*		*				!				
Diethyl Sulfate	64-67-5	*										
Diethylstilbesterol	56-53-1	*										
3,3'-Dimethoxybenzidine	119-90-4	*										
4,6-Dinitro-2-methylphenol	121-14-2	*				*						
1,4-Dintrobenzene	100-25-4	*										
1,2-Dinitrobenzene	528-29-0	*										
Dinocap	6119-92-2	*										
5,5-Diphenylhydantoin	57-41-0	*										
Di(2-ethylhexyl)adipate	103-23-1				*							
Endosulfan I	959-98-8	*		*				!				
Endosulfan II	33213-65-9	*		*				!				
Endosulfan Sulfate	1031-07-8	*		*	*			!				
Endrin	72-20-8	*		*				!	*			
Endrin Aldehyde	7421-93-4	*		*				!				
ndrin Ketone	53494-70-5	*										
EPN	2104-64-5	*										
Ethion	563-12-2	*										
Ethyl carbamate	51-79--6	*										

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Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
Fensulfothion	115-90-2	*										
Fenthion	55-38-9	*										
Fluchloralin	33245-39-5	*										
tachlor	76--44--8	*		*	*			!	*			
Heptachlor Epoxide	1024-57-3	*		*	*			!				
2,2',3,3',4,4',6-Heptachlorobiphenyl	52663-71-5				*							
2,2',4,4',5,6-Hexachlorobiphenyl	60145-22-4				*							
Hexamethyl Phosphoramide	680-31-9	*										
Hydroquinone	123-31-9	*										
Leptophos	21609-90-5	*										
Maleic anhydride	108-31-6	*										
Malathion	121-75-5	*										
Mevinphos	7786-34-7	*										
Mestranol	72-33-3	*										
Methoxychlor	72-43-5	*			*			!	*			
4,4'-Methylenebis(2-chloroanline)	101-14-4	*										
3-Methylphenol	108-39-4	*						*	*			
Mexacarbate	315-18-4	*										
Mirex	2385-85-5	*										
Monocrotophos	6923-22-4	*										
Naled	300-76-5	*										
Nicotine	54-11-5	*										
5-Nitroacenaphthene	602-87--9	*										
4-Nitrobiphenol	92-93-3	*										
Nitrofen	1839-75-5	*										
5-Nitro-o-anisidine	99-59-2	*										
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8				*							
Octamethylpyrophosphoramide	152-16-9	*										
4,4'-Oxydianiline	101-80-4	*										
2,2',3',4,6-Pentachlorobiphenyl	60233-25-2				*							
Phenobarbital	50-06-6	*										
1,4-Phenylenediamine	106-50-3	*										
Phosalone	2310-17-0	*										
Phosmet	723-11-6	*										
Phosphamidon	13171-21-6	*										
Phthalic Anhydride	85-44-9	*										
eronyl Sulfoxide	120-62-7	*										
Propylthiouracil	51-52-5	*										
Pyridine	110-86-1	*						*	*		2	*
Resorcinol	108-46-3	*										

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Parameter	CAS Number	8270A	Apdx IX	625	525.1	CLP SVOA	CAL STD	NLS LIST	TCLP	ULTRA MIX #	NSI Mix	NSI LCS
Simazine	122-34-9				*							
Strychnine	57-24-9											
Sulfallate	95-06--7	*										
Terbufos	13071-79-9	*										
2[illegible]4,4'-Tetrachlorobiphenyl	2437-79-9				*							
Tetrachlovinphos	961-11-5	*										
Tetraethyl pyrophosphate	107-49--3	*										
Thiophenol (Benzenethiol)	108--98-5	*										
Toluene Diisocyante	584 84--9	*										
Toxaphene	8001-35-2	*		*	*				*			
2,4,5-Trichlorobiphenyl	15862-07-4				*							
Trifluralin	1582-09-8	*										
Trimethyl phosphate	512-56-1	*										
2,4,5-Trimethyaniline	137-17-7	*										
Tri-p-tolyl phosphate (h)	78-32-0	*										
Tris(2,3-dibromopropyl) phosphate	126-72-7	*										

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INSTRUMENT TEMPLATE REPORT

Template Description: Semi-Volatile Organic Compounds by EPA 8270B
Template Name: NLS8270   Rounded: Y 2   Date Printed: 01/23/96   Page: 1

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NAME	S#	PEAK DESCRIPTION	MDL	LOQ	UNIT	SD	PCODE	METHD	R#
NLS8270	1	Pyridine	3.1	10	ug/L	2			1
NLS8270	2	N-nitrosodimethylamine	0.47	1.6	ug/L	2			2
NLS8270	3	Aniline	0.60	2.0	ug/L	2			3
NLS8270	4	Phenol	0.45	1.5	ug/L	2			4
NLS8270	5	Bis(2-chloroethyl)ether	0.67	2.2	ug/L	2			5
NLS8270	6	2-Chlorophenol	0.65	2.2	ug/L	2			6
NLS8270	7	1,3-Dichlorobenzene	0.41	1.4	ug/L	2			7
NLS8270	8	1,4-Dichlorobenzene	0.48	1.6	ug/L	2			8
NLS8270	9	1,2-Dichlorobenzene	0.45	1.5	ug/L	2			9
NLS8270	10	Benzyl Alcohol	1.2	4.1	ug/L	2			10
NLS8270	11	Bis(2-chloroisopropyl)ether	0.71	2.4	ug/L	2			11
NLS8270	12	2-Methylphenol	0.76	2.5	ug/L	2			12
NLS8270	13	Hexachloroethane	0.43	1.4	ug/L	2			13
NLS8270	14	N-nitroso-di-n-propylamine	0.67	2.2	ug/L	2			14
NLS8270	15	3-Methylphenol	0.52	1.8	ug/L	2			15
NLS8270	16	4-Methylphenol	1.3	4.3	ug/L	2			16
NLS8270	17	Benzoic Acid	2.8	9.3	ug/L	2			17
NLS8270	18	N-nitrosopiperidine	0.75	2.5	ug/L	2			18
NLS8270	19	N-nitrosodi-n-butylamine	0.75	2.5	ug/L	2			19
NLS8270	20	2,4-Dichlorophenol	0.77	2.6	ug/L	2			20
NLS8270	21	Nitrobenzene	0.71	2.4	ug/L	2			21
NLS8270	22	Isophorone	0.72	2.4	ug/L	2			22
NLS8270	23	2-Nitrophenol	0.64	2.1	ug/L	2			23
NLS8270	24	2,4-Dimethylphenol	0.51	1.6	ug/L	2			24
NLS8270	25	Bis(2-chloroethoxy)methane	0.80	2.7	ug/L	2			25
NLS8270	26	2,6-Dichlorophenol	0.76	2.5	ug/L	2			26
NLS8270	27	1,2,4-Trichlorobenzene	0.61	2.0	ug/L	2			27
NLS8270	28	Naphthalene	0.88	2.8	ug/L	2			28
NLS8270	29	4-Chloroaniline	0.75	2.5	ug/L	2			29
NLS8270	30	Hexachlorobutadiene	0.54	1.8	ug/L	2			30
NLS8270	31	4-chloro-3-methylphenol	0.70	2.3	ug/L	2			31
NLS8270	32	2-Methylnaphthalene	0.68	2.3	ug/L	2			32
NLS8270	33	Hexachlorocyclopentadiene	1.2	4.0	ug/L	2			33
NLS8270	34	1,2,4,5-Tetrachlorobenzene	0.69	2.3	ug/L	2			34
NLS8270	35	1-Chloronaphthalene	0.59	2.0	ug/L	2			35
NLS8270	36	4-Nitrophenol	0.43	1.4	ug/L	2			36
NLS8270	37	Pentachlorobenzene	0.63	2.1	ug/L	2			37
NLS8270	38	1-Naphthylamine	0.43	1.4	ug/L	2			38
NLS8270	39	2-Naphthylamine	0.65	2.2	ug/L	2			39
NLS8270	40	2,3,4,6-Tetrachlorophenol	0.58	1.9	ug/L	2			40
NLS8270	41	2,4,6-Trichlorophenol	0.86	2.9	ug/L	2			41
NLS8270	42	2,4,5-Trichlorophenol	0.74	2.5	ug/L	2			42
NLS8270	43	2-Chloronaphthalene	0.78	2.6	ug/L	2			43
NLS8270	44	2-Nitroaniline	0.59	2.0	ug/L	2			44
NLS8270	45	Acenaphthylene	0.71	2.3	ug/L	2			45
NLS8270	46	Dimethylphthalate	0.77	2.6	ug/L	2			46
NLS8270	47	2,6-Dinitrotoluene	0.74	2.5	ug/L	2			47
NLS8270	48	Acenaphthene	0.71	2.3	ug/L	2			48
NLS8270	49	3-Nitroaniline	0.75	2.5	ug/L	2			49
NLS8270	50	2,4-Dinitrophenol	0.51	1.7	ug/L	2			50
NLS8270	51	Dibenzofuran	0.70	2.3	ug/L	2			51

Attachment II

INSTRUMENT TEMPLATE REPORT

Template Description: Semi-Volatile Organic Compounds by EPA 8270B

Template Name: NLS8270 Rounded: Y 2 Date Printed: 01/23/96 Page: 2

****

NAME	S#	PEAK DESCRIPTION	MDL	LOQ	UNIT	SD	PCODE	METHD	R#
NLS8270	52	2,4-Dinitrotoluene	0.71	2.3	ug/L	2			52
NLS8270	53	Fluorene	0.61	1.9	ug/L	2			53
NLS8270	54	4-Chlorophenyl-phenyl ether	0.65	2.2	ug/L	2			54
NLS8270	55	Diethylphthalate	0.81	2.7	ug/L	2			55
NLS8270	56	4-Nitroaniline	0.69	2.3	ug/L	2			56
NLS8270	57	4,6-Dinitro-2-methylphenol	0.59	2.0	ug/L	2	38779		57
NLS8270	58	4-Aminobiphenyl	0.67	2.3	ug/L	2			58
NLS8270	59	Diphenylamine	0.70	2.3	ug/L	2			59
NLS8270	60	Pentachloronitrobenzene	0.62	2.1	ug/L	2			60
NLS8270	61	N-nitrosodiphenylamine	0.70	2.3	ug/L	2			61
NLS8270	62	4-Bromophenyl-phenyl ether	0.57	1.9	ug/L	2			62
NLS8270	63	Hexachlorobenzene	0.60	2.0	ug/L	2			63
NLS8270	64	Pentachlorophenol	0.65	2.2	ug/L	2			64
NLS8270	65	Phenanthrene	0.63	2.0	ug/L	2			65
NLS8270	66	Anthracene	0.60	1.9	ug/L	2			66
NLS8270	67	Di-n-butylphthalate	0.74	2.5	ug/L	2			67
NLS8270	68	Fluoranthene	0.60	1.9	ug/L	2			68
NLS8270	69	Pyrene	0.59	1.9	ug/L	2			69
NLS8270	70	Benzidine	0.31	1.0	ug/L	2			70
NLS8270	71	p-(Dimethylamino)azobenzene	0.53	1.7	ug/L	2			71
NLS8270	72	Butylbenzylphthalate	0.43	1.4	ug/L	2			72
NLS8270	73	3,3'-Dichlorobenzidine	0.90	3.1	ug/L	2			73
NLS8270	74	Benzo(a)anthracene	0.57	1.8	ug/L	2			74
NLS8270	75	Chrysene	0.68	2.2	ug/L	2			75
NLS8270	76	bis(2-Ethylhexyl)phthalate	0.82	2.6	ug/L	2			76
NLS8270	77	Di-n-octylphthalate	0.43	1.4	ug/L	2			77
NLS8270	78	Benzo(b)fluoranthene	1.9	6.3	ug/L	2			78
NLS8270	79	Benzo(k)fluoranthene	0.60	1.9	ug/L	2			79
NLS8270	80	Benzo(a)pyrene	0.64	2.0	ug/L	2			80
NLS8270	81	Indeno(1,2,3-cd)pyrene	0.78	2.5	ug/L	2			81
NLS8270	82	Dibenzo(a,h)anthracene	0.70	2.2	ug/L	2			82
NLS8270	83	Benzo(g,h,i)perylene	0.78	2.5	ug/L	2			83

Attachment II

NORTHERN LAKE SERVICE
GC/MS - SEMI-VOLATILES INSTRUMENT RUN LOG
HP 5890 GC / HP 5972 MSD

BOOK: ____________
PAGE: ____________

Date:	Sequence:	Analyst:

Method: →	☐ 8270/625 Analysis; ☐ 525 Analysis	Date of Calibration:

ICAL Reference:	CCAL Reference:

ISTD STD Reference:	Surrogate STD Reference:	DFTPP Reference:

HP File	Sample Number	Dilution Factor	Initial Vol/Mass	Final Volume	Matrix Type*	Comments
DFTPP						
DAT001						
DAT002						
DAT003						
DAT004						
DAT005						
DAT006						
DAT007						
DAT008						
DAT009						
DAT010						
DAT011						
DAT012						
DAT013						
DAT014						
DAT015						
DAT016						
DAT017						
DAT018						
DAT019						
DAT020						

Additional Comments:

* Matrix Type: S = Soil, GW = Groundwater, WW = Wastewater, DW = Drinking Water

Org-26 (8/95)

Attachment III

NORTHERN LAKE SERVICE
GC/MS - SEMI-VOLATILES
SAMPLE PREPARATION LOGBOOK

BOOK: ____________
PAGE: ____________

Date:	* Extraction Method:	Surrogate Mix Reference:
Analyst:	Extraction Batch:	Spike Mix Reference:

NLS Sample Identification	Initial Mass/Volume	Final Volume	Surrogate Volume	Spike Volume	Comments
Blk					
MS					
MSD					

* Methods: 3510B: Liq/Liq, 3550A: Sonication, 3580: Waste Dilution, 3535: SPE

KEY		
MS = Matrix Spike	LCS = Laboratory Control Spike	A = Acid
MSD = Matrix Spike Duplicate	BN = Base / Neutral	ABN = Acid / Base / Neutral

Org-28 (9/95)

Attachment IV

NORTHERN LA[illegible] SERVICE, INC.
GC/MS SEMI-VOLATILES
STANDARDS PREPARATION LOGBOOK

BOOK: ____________
PAGE: ____________

Attachment V

Date	Vendor	Mix #	Lot #	Stock Conc.	Dilution/Prep	Final Conc.	Mix Ref. #	Analy

ICAL Reference:	Continuing Calibration Reference:	Matrix Spike Reference:

Comments:

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15094 -- Semivolatile GC/MS by 8270B            PAGE: 1

PRINTED: 01/22/96 17:21:57      DATE & TIME ANALYZED ______________________

DEFAULT UNITS ug/L
======================================================================================

97998 -- 19273___________________________________________  __________  __________
Northern Lake Service - Bottle Checks -- MAX: -- 1000 Amb Bin A 1A --  3 d-old 3 d-hse

97999 -- 19273___________________________________________  __________  __________
Northern Lake Service - Bottle Checks -- MAX: -- 1000 Amb Bin B 2A --  3 d-old 3 d-hse

98000 -- 19273___________________________________________  __________  __________
Northern Lake Service - Bottle Checks -- MAX: -- 1000 Amb Bin B 3A --  3 d-old 3 d-hse
```

Attachment VI

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF METALS BY ICP AND TRACE ICP

SOP NUMBER: INO-METH-ICP-METALS-0

EFFECTIVE DATE: ____________

CONTROLLED COPY NUMBER: ____________

Originated By: ____________ Date: ____________
NLS Associate

Approved By: ____________ Date: ____________
Supervisor

Reviewed By: ____________ Date: ____________
QA Officer

Authorized By: ____________ Date: ____________
Laboratory Manager

I. METHOD TITLE: Determination of Metals by ICP and Trace ICP

II. METHOD SCOPE AND APPLICATION

A. Inductively coupled plasma-atomic emission spectroscopy (ICP) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

B. Elements for which Method 6010 is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

Table 1 - See Attachment A

III. REFERENCES

A. Winge, R.K.: Peterson, V.J.; Fassel, V.A. Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines (final report, March 1977-February 1978); EPA-600/4-79-017, Environmental Research Laboratory, Athens, GA, March 1979; Ames Laboratory: Ames IA.

B. Test Methods: Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1982; EPA-600/4-82-057.

C. Patel, B.K.; Raab, G.A.; et al. Report on a Single Laboratory Evaluation of Inductively Coupled Optical Emission Method 6010; EPA Contract No. 68-03-3050, December 1984.

D. Sampling and Analysis Methods for Hazardous Waste Combustion; U.S. Environmental Protection Agency; Air and Energy Engineering Research Laboratory, Office of Research and Development: Research Triangle Park, NC, 1986; Prepared by Arthur D. Little, Inc.

E. Bowmand, P.W.J.M. Line Coincidence Tables for Inductively Coupled Plasma Atomic Emission Spectrometry, 2nd ed.; Pergamon: 1984.

F. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

G. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

H. Methods for Chemical Analysis of Water and Wastes. EPA March 1983, Method 200.7.

I. Standard Methods for the Examination of Water and Wastewater, 18th Ed. 1992.

J. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846 1986.

IV. METHOD SUMMARY

A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3005 - 3050). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

B. Method 6010 describes the simultaneous, or sequential, multielemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

V. INTERFERENCES

A. Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Users of all ICP instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Recommended wavelengths are listed in Table 1 and potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

B. Element-specific interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing, height, argon flow rate, etc. The user should be aware of the possibility of interferences other than those specified in Table 2 and that analysts should be aware of these interferences when conducting analyses.

C. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Changing the nebulizer and removing salt buildup at the tip of the torch sample injector can be used as an additional measure to control salt buildup. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

D. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

Table 2 - See Attachment B

VI. SAMPLING

A. Bottle Preparation

B. Preservation - Samples must be preserved by addition of Nitric Acid to lower pH to <2.

C. Holding Times - Holding time of six months (180 days) for preserved samples.

VII. SAFETY

A. Acids and metal salts in solution may be toxic/corrosive. Gloves, labcoats and safety glasses should be worn when handling these materials. Fume hoods are available and should be used when the situation requires. All personnel should be aware of the location of showers and eyewashes.

VIII. EQUIPMENT AND MATERIALS

A. Digestion/Extraction/Preparation Equipment

1. Inductively coupled argon plasma emission spectrometer:
   a. Computer-controlled emission spectrometer with background correction.
   b. Radio frequency generator compliant with FCC regulations.
   c. Argon gas supply - Welding grade or better.
2. Operating conditions:
   a. The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
3. Class A volumetric flasks
4. Eppendorf mechanical pipettes.

IX. REAGENTS AND STANDARDS

A. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

1. Hydrochloric acid (conc), HCl.
2. Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriate beaker.

3. Nitric acid (conc), HNO_3.
4. Nitric acid (1:10, HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriate beaker.

B. Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

C. Standard stock solutions are purchased.

D. Working standard preparation information is located in the standard preparation logbook for each instrument (Trace) (Atomscan 25).

X. PROCEDURE

A. Extraction/Digestion/Preparation

1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched. Solubilization and digestion procedures are presented in Digestion SOP's.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. Set up the instrument with proper operating parameters as established.

B. Stabilization

1. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration.

C. Calibration

1. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. Flush the system with the calibration blank between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve should consist of a blank and three standards.

D. Sample/Standard Presentation to Instrument

1. Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5% (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

2. Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. Analyze the second source check standard and calibration blank before and after samples are run and every 10 samples throughout. The blank must be ± LOQ for each element and the second source standard must be ± 10% of true value.

   a. Interelement correction factor checks.

      i. Trace

         (i.) A solution containing interferents and analytes should be run at the beginning (after calibration) and the end of the run. The measured concentrations should be within ± 20% of the true value.

      ii. Atomscan 25

         (i.) An interferent solution should be prepared and analyzed at the beginning and end of the run. The analyte concentration (not interferent) displayed should be ± LOQ. If not calculate new IEC's.

XII. CALCULATIONS

A. General Calculations

1. Samples above known range must be diluted. After dilution the resulting concentration must be multiplied by the dilution factor.

   Example: 47 mg/L X 50 = 2350 mg/L

   The sample was over range and was diluted 50 times. The MDL & LOQ must also be multiplied by the dilution factor (50).

B. Calculations for Solid Samples

1. $C = \frac{r \times d \times v}{w \times s}$

   Where: c = concentration in mg/Kg.
   r = sample result of digestate (ug/ml).
   d = dilution factor.
   v = final volume of digestate (mL).
   w = weight of sample (g).
   s = % solid of the sample (50% = 0.50).

C. Hardness (mg $CaCO_3$/L) = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

XIII. QUALITY CONTROL

A. Standards - Standard information can be found in standard preparation logbooks.

B. Sample QC

1. Spike recovery must be within NLS control limits.

2. Precision measurements must be within NLS control limits.

3. Failure of accuracy and precision measurements should refer to QC failure corrective action document.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets function as run logs and data sheets.

B. Units - mg/L for Atomscan, ug/L for Trace ICP with some exceptions (Al, Ca, Fe, Mg, Na, K mg/L)

C. Detection limits are determined yearly and can be located on a list of detection limits or on benchsheets.

D. Analytical data including benchsheets and instrument printout should be kept on file by dat~~a~~e analyzed.

XV. CLEAN UP

A. Lab Work Area - Work areas should be cleaned after use.

B. Sample Disposal - Samples to be disposed of can be rinsed down the drain with copious amounts of cold water.

C. Containers can be rinsed and recycled.

XVI. MAINTENANCE / TROUBLESHOOTING

A. Preventive maintenance procedures ~~and frequency~~

1. Daily
    a. Peristaltic pump tubing should be replaced.
    b. Waste carboy should be emptied as needed.
    c. Sample uptake lines and nebulizer should be free of plugs.
    d. Argon must have sufficient pressure and volume to complete the day's analysis including warmup.
    e. Instrument communications board and nebulizer pressure set switch should be reset.
    f. Computer should be rebooted.
    g. Profile Trace ICP; Peak Search Atomscan25 before beginning calibration.

B. Troubleshooting procedures - See operators manual.

XVII. ATTACHMENTS

A. Table 1 Recommended Wavelengths and Estimated Instrumental Detection Limits

B. Table 2 Analyte Concentration Equivalents Arising From Interference at the 100-mg/L Level

Table 1 Recommended Wavelengths and Estimated Instrumental Detection Limits

Detection Element	Trace ICP Wavelength[a] (nm)	Detection Limit	AtomScan 25 Wavelength
Aluminum	308.215 (273.3)	*	**
Antimony	306.833	*	**
Arsenic	189.0	*	**
Barium	493.4	*	**
Beryllium	313.04	*	**
Boron	249.6	*	**
Cadmium	226.502	*	**
Calcium	317.933	*	**
Chromium	267.716	*	**
Cobalt	228.616	*	**
Copper	324.754	*	**
Iron	259.940 (271.4)	*	**
Lead	220.353	*	**
Lithium	N/A	*	670.8
Magnesium	279.079	*	**
Manganese	257.610	*	**
Molybdenum	N/A	*	202.0
Nickel	231.604	*	**
Potassium	766.491	*	**
Selenium	196.026	*	**
Silver	328.068	*	**
Sodium	588.995 (330.2)	*	**
Strontium		*	407.77
Thallium	190.864	*	**
Tin	N/A	*	189.9
Titanium	N/A	*	**
Vanadium	292.402	*	**
Zinc	213.856	*	**

* See Current List - Updated Yearly
**See Current Method

Table 2 Analyte Concentration Equivalents Arising From Interference at the 100-mg/L Level

		Interferent^{a,b}									
Analyte	Wavelength (nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	----
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	----
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	213.856	--	0.05	--	0.005	--	--	--	--	0.02	--

		Interferent[a,b]									
Analyte	Wavelength (nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Zinc	213.86	--	--	--	0.14	--	--	--	0.29	--	--

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Mg - 1000 mg/L
Ca - 1000 mg/L	Mn - 200 mg/L
Cr - 200 mg/L	Ti - 200 mg/L
Cu - 200 mg/L	V - 200 mg/L
Fe - 1000 mg/L	

[b]The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

NORTHERN LAKE SERVICE, INC.

TITLE:

Determination of Metals by Atomic Absorption - Flame or Furnace

SOP NUMBER: INO-METH-AA-FCE-0

EFFECTIVE DATE: JAN 2 1996

CONTROLLED COPY NUMBER: ________

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TEST: Determination of Metals by Atomic Absorption - Flame or Furnace.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

INSTRUMENTATION:

Varian AA-1475 Atomic Absorption Spectrophotometer.
Varian GTA-95 Graphite Tube Atomizer.
Perkin Elmer 4100ZL Zeeman Atomic Absorption Spectrophotometer.

SCOPE AND APPLICATION:

This method can be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, waste water, and waste material. Dissolved metals are determined from filtered and acid preserved samples. Total metals are determined after appropriate digestion procedures have been performed. Drinking waters free of particulate matter may be analyzed directly. Domestic and industrial wastes require processing to solubilize suspended matter. Sludges, sediments and other solid samples can be analyzed after appropriate treatment.

Detection limits, sensitivity and optimum ranges will vary with various models of atomic absorption spectrophotometers. Table 1 and Table 2 contain the detection limits for the various metals analyzed on the Varian AA-1475 and the Perkin Elmer 4100ZL. Detection limits by direct aspiration may be extended through concentration of the sample and/or through solvent extraction techniques. Lower concentrations can also be determined through the furnace technique. When using the furnace technique, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may interfere with the metals analysis. In order to provide valid data with furnace techniques, the analyst must examine each matrix for possible interference effects. If interference does occur in a particular matrix, the sample should be analyzed accordingly either through dilution, matrix modification, or the method of standard addition.

In samples where direct aspiration does not provide adequate sensitivity, the furnace technique may be used. Other specialized procedures are also available such as gaseous hydride method for arsenic and selenium, cold vapor method for mercury, and the chelation-extraction procedure for specific metals. Approved colorimetric methods for many elements are also available.

SUMMARY OF METHOD:

The following is a summary of the direct aspiration atomic absorption spectroscopy method and the furnace atomic absorption spectroscopy method. Lower detection limits can be determined with the furnace technique.

Direct Aspiration

In direct aspiration atomic absorption spectroscopy, a sample is aspirated into a flame. The heat of the flame causes the sample to atomize. A light beam from a hollow cathode lamp with a cathode made of the element of interest is directed through the flame into a monochromator. A detector in the monochromator measures the amount of light absorbed by the atomized sample. The amount of absorption depends on the presence of free unexcited ground state atoms in the flame. The light produced by the cathode lamp has a wavelength characteristic of the metal being analyzed. An increase in the element of interest in the flame causes more light to be absorbed. The detector monitors this absorption and a determination of the amount of element in a sample can be derived.

Furnace Technique

When using the furnace technique, a known amount of sample is placed in a graphite tube in the furnace. The graphite tube is then heated, causing the sample to evaporate, char, and finally atomize. Since a greater percentage of the atoms are atomized in the tube than in the flame, the detection of very low concentrations of elements is possible. The principle is very similiar to flame technique, except a furnace is used to atomize the sample instead of a flame. Light of a specific wavelength is directed through the vapor of the atomized sample. The intensity of the transmitted light decreases as the amount of ground state element increases. A monochromator isolates the wavelength of interest and transmits this to the photosensitive detector. The detector monitors the amount of absorption and a determination of the amount of element in the sample can be derived.

General Sample Characteristics

The atomic absorption method is generally limited to the analysis of metals in solution or solubilized through some form of processing. Wastewater and industrial effluents usually require some form of digestion because of the complexity and variability of the matrix. A digestion is required when the breakdown of organic material is necessary.

DEFINITIONS:

Optimum Concentration Range - A range below which scale expansion must be used and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating conditions.

Sensitivity - The concentration in milligrams of metal per liter that produces an absorption of 1%.

Detection Limit - Detection limits can be expressed as either instrument detection limits or method detection limits. Instrument detection limit is defined as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength. Method detection limit is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing the analyte.

Dissolved Metals - Those constituents (metals) which will pass through a 0.45 um membrane filter.

Suspended Metals - Those constituents (metals) which are retained by a 0.45 um membrane filter.

Total Metals - The concentration of metals determined on an unfiltered sample following vigorous digestion if necessary, or the sum of the concentrations of metals in both the dissolved and suspended forms. Any samples containing visible particulates or solid material will require a digestion. Total metals, including Lead and Copper, in drinking water is performed on an unfiltered and undigested sample if the sample turbidity is less than 1 NTU.

Total Recoverable Metals - The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference sheet for material handling is available for all chemicals used in this method. These sheets are available to all analysts.

INTERFERENCES IN FLAME ATOMIZATION:

The most common type of interference is chemical interference. This occurs when there is a lack of absorption of atoms bound in molecular combination in the flame. This can occur when the flame is not sufficiently hot enough to dissociate the molecules. The addition of lanthanum into the sample matrix will overcome the phosphate interference in the Magnesium, Calcium, and Barium determinations. The addition of Calcium will eliminate the Silica interference with Manganese. Chemical interferences may also be eliminated by separating the metal from the interfering material. While complexing agents are used to increase the sensitivity of the analysis, they may also be used to eliminate interferences.

INTERFERENCES IN FLAMELESS ATOMIZATION:

Although the use of flameless atomization greatly reduces oxide formation, it is still subject to chemical and matrix interferences. Gases produced in the furnace during analysis may have molecular absorption bands that overlap the analytical wavelength. The use of a background correction or an alternate wavelength should eliminate this problem.

Interferences from a smoke producing sample matrix can be reduced by extending the charring time at a higher temperature, or using an ashing cycle in the presence of air. Samples with high concentrations of organic material should be oxidized by a digestion procedure prior to analysis. Nitric acid is preferred for the digestion procedure. If the addition of other acids is required, use minimal amounts. This particularly applies to Hydrochloric acid.

EQUIPMENT:

The following is the equipment used to analyze water and waste samples for trace elements:

1. Varian AA-1475 Atomic Absorption Spectrophotometer.

2. Varian GTA-95 Graphite Tube Atomizer.

3. Perkin Elmer 4100ZL Zeeman Atomic Absorption Spectrometer.

3.5 Electrodeless Discharge Lamp (EDL) II Power Supply System.

4. Hollow Cathode Lamps (element specific).

4.5 Electrodeless Discharge Lamp (EDL) - Element specific.

5. Strip Chart Recorder, used primarily during Hg analysis.

6. Glassware.

REAGENTS AND STANDARDS:

The following is the required chemicals for atomic absorption analysis:

1. Distilled and deionized water.

2. Nitric Acid, concentrated.

3. Nitric Acid, (1:1) Add 500 mL of concentrated HNO_3 to 500 mL laboratory grade, distilled water.

4. Hydrochloric Acid, (1:1) Add 500 mL of concentrated HCL to 500 mL deionized, distilled water (for flame use only).

5. Stock standard metal solutions used for calibration and sample spiking.

6. Fuel and oxidant (for flame use only).

SAMPLE HANDLING AND PRESERVATION:

All laboratory glassware used in this procedure must be thoroughly washed with detergent and tap water; rinsed with (1:1) nitric acid; and final rinsed three times with distilled and deionized water. Chromic acid may be used to remove organic deposits from glassware, but extreme care must go into the final rinse in order to remove all traces of Chromium.

Before collection of the sample, a decision must be made as to what types of data is desired. For the determination of dissolved parameters, the sample must be filtered throught a 0.45 um membrane filter as soon as practical after collection and acidified to a pH <2.0 with (1:1) Nitric acid. For the determination of total or total recoverable parameters, the sample must be acidified to a pH <2.0 with (1:1) Nitric acid as soon as possible after the time of collection. For determinations of suspended parameters, a measured volume of nonpreserved sample must be filtered through a 0.45 um membrane filter as soon as possible after collection. The filter should then be transferred to a suitable container for storage. Drinking water samples containing suspended and setteable material should be prepared as a total recoverable parameter.

SAMPLE PREPARATION :

For the determination of dissolved parameters, the filtered, preserved sample may often be analyzed as received. Digestion is usually not required for a filter sample. The acid matrix and concentration of the samples and calibration standards must be the same. If a precipitate has formed upon acidification of the sample, the precipitate must be redissolved before analysis through the addition of more acid or by heat. If the precipitate can not be redissolved, the sample must be digested for total recoverable parameters.

Samples requiring total parameters for ground water, waste water, soils, and sludges must be digested prior to analysis. Follow the digestion procedures given on page DIG - 1.

PROCEDURE:

Instrument parameters specific to each element analyzed by atomic absorption are given in individual operating procedures in this manual. Refer to these procedures for instrument setups. The general start-up and analysis procedures for samples run on the Varian AA-1475 and the Perkin Elmer 4100ZL are given below.

I) Start-up procedure for Varian AA-1475 / GTA-95 Graphite Furnace.

1. Turn on exhaust fume hood (Hood is normally left on).

2. Turn argon carrier/purge gas on at tank requlator.

3. Turn printer on.

4. Turn power on to AA-1475 and GTA-95.

5. Select program to run on furnace.

   A. Pick 1 of 8 preset programs on instrument panel.
   B. Manually program element specific parameters.
   C. Element specific parameters begin on page AAM - 1.

6. Select proper Hollow Cathode Lamp and insert into turret.

7. Adjust lamp current to proper setting.

8. Allow lamp to warm up and stabilize for 15 - 45 minutes, depending upon the element of interest and choice of source lamp.

9. Check graphite tube appearance.

   A. Clean furnace head and quartz window if necessary.
   B. Insert pyrolytic platform or partition tube if replacement is necessary.

10. Select and optimize for element specific wavelength.

11. Optimize furnace workhead for maximum light throughput.

12. Optimize lamp for maximum light throughput using axial

    adjustment on lamp turret and recheck workhead alignment.

13. Change water in reagent blank/autosampler flush reservoir.

14. Purge air bubbles from autosampler syringe.

15. Insert appropriate modifiers, blanks, and standards into autosampler. Use the loading order list given below.

    A. Verify auto sampler positions and sample pickup, adjust if necessary.
    B. Verify sample injection into graphite tube. Adjust depth if necessary. Adjust autosampler if necessary.

16. When verification of autosampler is complete, reset the autosampler and restart the program with the appropriate samples inserted in the autosampler. Use the loading list given below.

17. Press run to initiate program.

18. Furnace will run a standard curve for the element of choice, followed by a verification standard and a blank.

19. If the results obtained for the calibration data and the from the analysis of the blank are in control, continue to analyze samples loaded according to loading list given below.

20. Follow QC requirements given at the end of this SOP.

II) Shutdown procedure for Varian AA-1475 and GTA-95.

1. Verify completion of last cycle.
2. Push STOP button on AA-1475.
3. Turn down lamp current if applicable.
4. Turn off GTA-95.
5. Turn off AA-1475.
6. Turn off Printer.
7. Turn off Argon gas at regulator.
8. Fume hood is normally left on.

III) Start-up procedure for Perkin Elmer 4100ZL.

1. Turn Argon gas on at tank regulator.
2. Turn printer on.
3. Turn computer on and start furnace software by executing AA-INSTRUMENT file.
4. Turn 4100ZL on.
5. Prepare fume extraction unit.
   - A. Empty scrubber water container and refill with lab grad,e deionized water (if required).
   - B. Remove scrubber filter paper and replace with new filter (if required).
5. Prepare distilled, deionized water reservoir and waste containers.
   - A. Fill water reservoir.
   - B. Drain waste reservoir and replace.
6. The EDL II source is now active.
7. Turn on the voltage supply and adjust to the proper voltage.
8. Allow 45 minutes for the EDL II's / HC's to warm up and stabilize.
9. Select the method to load into the furnace.
10. Pull down the <Align Lamps> menu and select the element to analyze. Select the <AGC/AIC> icon to get the intensity reading for the element. Align lamp for maximum intensity.
11. Select <Wavelength Scan> to verify the peak of interest.
12. Exit the <Align Lamps> window.

13. Prepare standards, blanks, modifiers, and verification standard and insert them in the proper locations in the autosampler. Use the loading list given below for sequence.

14. Select the <AS-70> window, and then select <Sampler Standby> and verify the sipper probe is entering the graphite tube properly. Use the X-Y Axis adjustments if needed.

15. Select <Flush Sampler> and remove impurities from the sipper.

16. Select <Calibrate> to initiate the run of the calibration curve. Visibly inspect and ensure proper sample deposition. Run the standard curve and observe peak shapes and correlation coefficients.

17. If the proper peak shapes are observed and the correlation coefficient is observed, calculate characteristic mass to verif the proper sensitivity.

18. Select the <AS-70> window and enter the positions required for the analysis.

19. Select <Run Samples> to initiate the run.

20. Use the loading list given below.

21. Verification standards recovery limits, QC limits, spikes recovery limits, and duplicate spike limits, are all preprogrammed in the method. They will be run automatically. If any deviations from the limits are encountered, the instrument automatically executes the corrective action required by the method.

22. Follow QC requirements given at the end of this SOP.

IV) Shutdown Procedure for Perkin Elmer 4100ZL.

1. Select the <AS-70> window, verify the current run has been completed, or select terminate run from the menu.

2. Turn down the voltage to the EDL II's.

3. Exit furnace software program.

4. Turn 4100ZL off.

5. Turn printer off.

6. Turn computer off

7. Turn off Argon gas supply at regulator.

IV) Sample Loading List Varian AA-1475 / GTA-95 Graphite Furnace.

1. The following is the loading list for the autosampler. These positions are relative to initial calibration and blank reagent samples.

Position Number	Sample Type
1	Check Standard
2	Blank
3	Sample 1
4	Sample 1 spiked
5	Sample 1 duplicate spike
6 - 10	Samples 2 through 6
11	Check Standard
12	Blank
13	Sample 7
14	Sample 7 spiked
15	Sample 7 duplicate spike
16 - 20	Samples 8 through 12

2. The same pattern holds true for positions 21 through 40. A blank and a check standard will be analyzed before every group of six samples.

V) Sample Loading List For Perkin-Elmer

1. The following is the loading list for the autosampler. These positions are relative to initial calibration and blank reagent samples.

Position Number	Sample Type
Check Std.	Check Standard
Blank	Blank
1a	Sample A
1b	Sample A plus spike
2a	Sample A duplicate
2b	Sample A duplicate plus spike
3-11	Samples
Check Std.	Check Standard
Blank	Blank
12a	Sample B
12b	Sample B plus spike
13a	Sample B duplicate
13b	Sample B duplicate plus spike
14-22	Samples
23a	Sample C
23b	Sample C plus spike
24a	Sample C duplicate
24b	Sample C duplicate plus spike
25-33	Samples

CALCULATIONS:

Direct determination of liquid samples can be derived from calibration curve linear regression analysis or from the direct readout of the instrument. If dilution of the sample was required:

ug/L metal in sample = $A\,[(C + B) / C]$

where A = ug/L of metal in the diluted aliquot from curve,
B = mL of deionized water used for dilution,
C = mL of sample aliquot.

If sample contains particulates:

ug/l metal in sample = A (V / C)

where A = ug/L of metal in the sample from curve,
V = final volume of the processed sample,
C = mL of sample processed.

For dry solid samples (reported as mg/kg dry weight basis):

mg metal / kg sample = (A x V) / D

where A = mg/L of metal in processed sample from curve,
V = final volume of the processed sample in mL,
D = weight of dry sample in grams.

For wet solid samples (reported as mg/kg dry weight basis):

mg metal / kg sample = (A x V) / (W x P)

where A = mg/L of metal in processed sample from curve,
V = final volume of the processed sample in mL,
W = weight of wet sample in grams,
P = percent solids as a decimal.

QUALITY CONTROL:

Instrument are standardized prior to each analytical run. All sample sets are bracketed by verification standards and blank check samples before and after the run. If verification standards and/or blanks are found to be out of control, possible sources of error will be checked and the samples will be reanalyzed using a new standard curve and all appropriate check standards and blanks. Spike recovery data must conform to established control limits that are established by the Northern Lake Service - Quality Control Database. If spike recovery data is found to be out of control, dilution of the sample and /or the method of standard additions will be employed to bring the sample into recovery limits or to qualify the data.

All quality control results from an analytical run must be entered into the Northern Lake Service Quality Control database. Control limits are recalculated every three to six months. Only those parameters with a minimum of thirty data points will be recalculated.

INSTRUMENT MAINTENANCE:

Procedures for general maintenance on the Varian AA-1475, the Varian GTA-95, and the Perkin Elmer 4100ZL are given in the owners manual for the instrument. Refer to these manuals for maintenance procedures.

INDIVIDUAL FURNACE ANALYSIS TECHNIQUES

1. ANTIMONY
2. ARSENIC
3. BERYLLIUM
4. CADMIUM
5. CHROMIUM
6. COPPER
8. LEAD
9. SELENIUM
10. SILVER
11. THALLIUM

TEST: Antimony by atomic absorption furnace technique.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 20 - 300 ug/l

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: The calibration standards should be matrix matched to the samples analyzed.

12.5 ug/L 25.0 ug/L 50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 60 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1050 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2000 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 217.6 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

3. If Chloride presents a matrix problem, add an excess of 5 mg of ammonium nitrate to the furnace and ash using a ramp accessory or with incremental steps until the recommended ashing temperature is reached.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Arsenic by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: The calibration standards should be matrix matched to the samples analyzed.

12.5 ug/L
25.0 ug/L
50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 60 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 193.7 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame or Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Beryllium by atomic absorption furnace technique.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1 - 10 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

1.0 ug/l	2.0 ug/l	3.0 ug/l

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 50 seconds at 250 degrees Celsius.

2. Ashing time and temperature: 35 seconds at 1000 degrees Celsius.

3. Atomizing time and temperature: 6 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 234.9 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen should not be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Cadmium by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

0.5 - 3 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

1.0 ug/L
2.0 ug/L
3.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 45 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 700 degrees Celsius.

3. Atomizing time and temperature: 5 seconds at 1800 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 228.8 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Chromium by atomic absorption furnace technique.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 5 - 100 ug/L

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standard should be matrix matched to samples being analyzed.

5.0 ug/L 10.0 ug/L 20.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 40 seconds at 130 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2500 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 357.9 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required for samples high in dissolved solids.

2. Hydrogen peroxide added to the acidified solution will convert all Chromium to the trivalent state.

3. Nitrogen should not be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Copper by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1.0 - 60 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

5.0 ug/l   10.0 ug/l   20.0 ug/l

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 30 seconds at 125 degrees Celsius.

2. Ashing time and temperature: 30 seconds at 900 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2700 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 324.7 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.

2. Nitrogen may be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Lead by atomic absorption furnace.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 1.0 - 60 ug/L

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

5.0 ug/L 10.0 ug/L 20.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 30 seconds at 125 degrees Celsius.

2. Ashing time and temperature: 30 seconds at 500 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2700 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 283.3 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

3. To suppress sulfate interference, lanthanum can be added to both samples and standards.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Selenium by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

12.5 ug/L
25.0 ug/L
50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 70 seconds at 250 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 196.0 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operationg Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Silver by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1.0 - 25 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

2.5 ug/L 5.0 ug/L 10.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 35 seconds at 240 degrees Celsius.
2. Ashing time and temperature: 36 seconds at 600 degrees Celsius.
3. Atomizing time and temperature: 4 seconds at 2000 degrees Celsius.
4. Purge gas atmosphere: Argon.
5. Wavelength: 328.1 nm.
6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.
2. Nitrogen may be used as a purge gas.
3. Avoid the use of halide acids.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Thallium by atomic absorption furnace.

REFERENCE

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standard should be matrix matched to samples being analyzed.

10.0 ug/L
20.0 ug/L
30.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 55 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 550 degrees Celsius.

3. Atomizing time and temperature: 5 seconds at 2200 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 276.8 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Total Dissolved Solids - 180°C

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March 1983.

METHOD: EPA Method 160.1 (Gravimetric, dried at 180°C) Filterable Residue

SCOPE AND APPLICATION:

A well mixed sample is filtered through a 0.45 um membrane filter (Wisconsin Groundwater Program) or through a glass fiber filter (all other programs and dried to a constant weight at 180°C.

This procedure is applicable to drinking water, surface, and saline waters, domestic and industrial waste.

INTERFERENCES:

Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying desiccation and rapid weighing.

Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

Sample Preservation: Cool to 4°C. Analyze within 7 days from date of collection.

Sample Size: 50 mL (minimum)

Equipment: 100 mL crucibles, Class A pipet (50 or 100 mL), Mettler analytical balance.

PROCEDURE:

If volatile residue is to be determined, heat clean dish at 550°C + 50°C for one hour and desiccate. If only filterable residue is to be determined heat dish at 180°C for one hour and desiccate at least one hour. Store in desiccator and weigh on Mettler immediately before use. Make sure that the Mettler balance has been standardized using the 100 mg "Class S" weight. Do not touch the crucibles with bare hands, handle them with tongs. Place 50 mL of sample in weighed crucible and place in oven. Evaporate over night at 103-105°C. Remove dish and place immediately in a heated 180°C oven for one hour, then transfer to a desiccator for at least one hour. Weigh again and calculate the weight difference. Repeat cycle of drying, cooling, desiccating, and weighing process until a constant weight is obtained, or until weight loss is <4% of previous weight or 0.5 mg, whichever is less.

CALCULATIONS:

$$\text{mg/L TDS} = \frac{(\text{Wt. of Crucible \& dried sample in grams}) - (\text{Tare Wt. of Crucible in grams})}{\text{Sample size in mLs.}} * 1,000,000$$

DETECTION LIMIT: Approximately 2 mg/L for a 50 mL sample size.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control guidelines given in the quality control manual. Analysis 1/10 samples in duplicate.

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF ALKALINITY BY TITRATION

SOP NUMBER: INO-METH-TITR-ALK-O

EFFECTIVE DATE: JAN 2 2 1996

CONTROLLED COPY NUMBER:

Originated By: Nathaniel W. [illegible] Date: 12/14/95
NLS Associate

Approved By: Thomas R Priebe Date: 1/12/96
Supervisor

Reviewed By: W J Mosek Jr Date: 1/15/96
QA Officer

Authorized By: Date: 1/7/96
Laboratory Manager

I. METHOD TITLE: Determination of Alkalinity By Titration

II. METHOD SCOPE AND APPLICATION

A. Parameter(s)

1. Bicarbonate as HCO_3
2. Total as $CaCO_3$
3. Total unfiltered as $CaCO_3$
4. Hydroxide as $CaCO_3$
5. Carbonate as $CaCO_3$

B. NLS Test Codes / Descriptions

1. 180 bicarbonate
2. 200 total
3. 201 total unfiltered
4. 204 hydroxide
5. 205 carbonate

C. Detection Limit(s)

1. Aqueous
2. Solid
    a. MDL calculated from water
    b. LOQ calculated from water

III. REFERENCES

A. EPA 310.2 Titrimetric Alkalinity (pH 4.5)

B. Standard Method 18th edition 1992 Method 2320 Alkalinity

IV. METHOD SUMMARY

A. Alkalinity is a measure of a water's/soil's/waste's acid neutralizing capacity. It is the total of all titratable bases. The value may differ greatly with the end-point pH used. Alkalinity is a measure of a physical property of water/soil/waste and can be interpreted in terms of specific substances only when the chemical makeup of the sample is known.

B. Alkalinity is important in uses and treatments of natural waters, soils and wastes. The alkalinity of surface waters is primarily a function of carbonate, bicarbonate, hydroxide and phenolphthalein content. It is an indicator of the concentrations of these. The value may also include borates, phosphates, silicates or other bases if present. Alkalinity in excess of alkaline earth metal concentrations is important in determining a water's suitability for irrigation uses. Alkalinity is used in the interpretation and control of water and wastewater treatment processes. Raw domestic wastewater has an alkalinity less than, or only slightly greater than the water supply. Properly functioning anaerobic digestors have typical supernatant alkalinities in the 2000 to 4000 mg calcium carbonate ($CaCO_3$)/L.

C. Hydroxyl ions present in a sample as a result of dissociation or hydrolysis of solutes react with additions of standard acid. Alkalinity depends on the end-point pH used. For samples with low alkalinity values (< 20 mg/L $CaCO_3$) use an extrapolation technique based on the near proportionality of concentration of hydrogen ions to excess of titrant beyond the equivalence point. The amount of standardized acid needed to reduce the pH exactly. 0.30 pH s.u. is carefully measured. The change in pH corresponds to an exact doubling of the hydrogen ion concentration, a simple calculation can be made to the end point.

V. INTERFERENCES

A. Soaps, oily matter, suspended solids or precipitation may coat the glass pH electrode and cause a slow response. Allow additional time between titrant additions to let electrode come to equilibrium or clean it occasionally. Do not filter, dilute, concentrate or alter sample at all, in an attempt to compensate for sample matrix.

VI. SAMPLING

A. Bottle Preparation

1. Prepare bottles according to established lab procedure

B. Preservation

1. Non preserved samples

C. Storage

1. Refrigeration - before analysis

2. Ambient - during analysis

D. Holding Times

1. Analysis

a. 14 days from date of collection.

VII. SAFETY

A. Follow all laboratory safety guidelines established.

B. Consult safety officer/committee if in doubt.

VIII. EQUIPMENT AND MATERIALS

A. Reagents listed below in IX.

B. 10 mL Buret graduated in 0.05 mL increments

C. pH meter calibrated to manufacturers specifications.

D. pipets - Class A glass

1. 5 mL
2. 10 mL
3. 25 mL
4. 50 mL

E. 1 mL Eppendorf pipetter

F. Erlenmeyer Flasks - 250 mL size

IX. REAGENTS AND STANDARDS

A. Na_2CO_3 0.05N

1. Dry 3 to 5 grams primary standard Na_2CO_3 at 250°C for 4 hours and cool in desiccator. Weight 2.5 ± 0.2 grams and dilute in 1 liter volumetric flask. Do not keep longer than 1 week.

2. NLS uses a commercially prepared Na_2CO_3 solution 0.1N for standardization and spiking. The reagent is more stable and has a longer shelf life than IX.A.1.

3. Standard sulfuric acid 0.02N
    a. NLS dilutes off of a commercial stock of sulfuric acid 0.2N prepared by JT Baker or equivalent.
        i. 100 mLs of the 0.2N is diluted to 1L with reagent water.

4. Mixed bromcresol green - methyl red indicator solution
    a. Aqueous solution - not used by NLS
        i. 100 mg bromcresol green sodium salt
        ii. 20 mg methyl red sodium salt
        iii. Dissolve in 100 mL of deionized water
    b. Alcoholic solution - used by NLS
        i. 100 mg bromcresol green sodium salt
        ii. 20 mg methyl red sodium salt
        iii. Dissolve in 100 mLs of 95% ethyl alcohol or isopropyl alcohol

B. Storage Conditions

1. Reagents stored at room temperature

C. Shelf life

1. Follow dates on reagent bottles

X. PROCEDURE

A. Alkalinity as mg/L $CaCO_3$

1. Acid Standardization and verification analysis
    a. 1 mL of 0.1N Na_2CO_3 diluted to 50 mL is equal to 100 ppm
        i. Titrated value will equal 5 mL of 0.02N H_2SO_4 (100 ppm)
        ii. Must be ± 0.2 mL when titrated.

iii. If not try again.

iv. Remix titrant if still not in range.

b. Prepare the verification standard in accordance with manufacturers instructions and titrate to pH end-point of 4.5 ± 0.2 s.u.

c. Calculate results of verification and compare with manufacturers' certified value.

2. Sample Titration

a. Determine the correct dilution factor from the back data

i. See attached table.

b. Pipet the appropriate volume of sample and deionized water (if necessary).

c. Add three drops of mixed indicator

d. Insert pH probe into sample, wait for it to stabilize. Record pH as start pH.

e. Titrate dropwise (slowly) letting pH meter stabilize frequently.

f. When 4.50 ± 0.2 s.u. is reached a noticeable color change should occur.

g. Record end pH.

3. Calculate as follows:

$$\frac{A \times N \times 50{,}000}{\text{mL of sample}} = \text{alkalinity, mg } CaCO_3/L$$

Where: A = mL of acid/titrant used
N = normality of acid/titrant used
See note under general calculations

4. An abbreviated calculation for 50 mL sample size is as follows:

$$A \times 20 \times D = \text{Alkalinity, mg } CaCO_3/L$$

Where: A = mL of acid/titrant used
D = dilution factor if any

B. Alkalinity as Carbonate mg/L CO_3^{2-}

1. It is present when phenolphthalein alkalinity is not zero but is less than total alkalinity

C. Alkalinity as Hydroxide mg/L OH^-

1. It is present if phenolphthalein alkalinity is more than half the total alkalinity.

D. Alkalinity as Bicarbonate mg/L HCO_3^-

1. It is present if phenolphthalein alkalinity is less than half the total alkalinity.

2. Calculate bicarbonate alkalinity as follows:

$A \times 24.3843 \times D$ = Bicarbonate alkalinity, mg/L HCO_3

E. Alkalinity as Phenolphthalein

1. pH endpoint is 8.3 s.u.
2. if pH is < 8.3 s.u. phenolphthalein alkalinity is not present.

F. Alkalinity relationships (See Table A)

1. If phenolphthalein alkalinity is not present:
   a. carbonate alkalinity is not present
   b. hydroxide alkalinity is not present

Table A Alkalinity Relationships

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity As $CaCO_3$	Bicarbonate Concentration As $CACO_3$
P = 0	0	0	T
P < 1/2 T	0	2P	T - 2P
P = 1/2 T	0	2P	0
P > 1/2 T	2P - T	2(T - P)	0
P = T	T	0	0
Key: P - phenolphthalein alkalinity			
T - total alkalinity			

XI. CALCULATIONS

A. General Calculations

1. See X.A.3 and X.A.4 for calculations

Note: The 50,000 value in this calculation is a function of the molecular weight of $CaCO_3$. The 1,000 part of this number is a conversion factor to go from (L) on one side of the equation to mL on the other side. The 50 represents the $CaCO_3/H_2SO_4$ reaction. $CaCO_3$ has a molecular weight of 100. It takes two moles of H_2SO_4 to react with 1 mole of $CaCO_3$. The value of 50 is a simplification of this reaction. The calculation is further abbreviated by multiplying 50 by 1000 to get 50,000. NLS further abbreviates the calculation as seen in X.A.4.

B. Significant figures - no decimal points in reported value

C. Special Adjustments for Samples
   1. Sample Size
      a. 50 mLs
   2. Sample Dilution
      a. see table for dilution data

XII. QUALITY CONTROL

A. Duplicate 1 in 10 samples

B. Spike 1 in 10 samples (can be the same sample)

C. Calculate spike as follows:

$$\frac{\text{(1/2 of first mLs of titrant - spiked mLs of titrant)}}{5} \times 100 \text{ to determine \% of recovery}$$

D. If spiking a sample using a dilution calculate as follows:

1. $$\text{First Value} = \frac{\text{(first mLs of titrant x first dilution factor)}}{\text{second dilution factor}}$$

2. $$\frac{\text{second mLs titrated - first value}}{5} \times 100 \text{ to determine \% of recovery}$$

XIII. RECORDS AND REPORTING DATA

A. Report as mg/L $CaCO_3$

B. Enter dilution factor (where applicable) in proper column in computer.

C. Qualifiers on Data
   1. If titrated past holdtime of 14 days.
   2. Any other unusual occurence or problem.

XIV. CLEAN UP

A. Lab Work Area - done in metals preparation area

B. Equipment / Glassware - follow manufacturer's/laboratory procedure

XV. MAINTENANCE / TROUBLESHOOTING

A. Preventive maintenance procedures and frequency
   1. Clean pH probe.
   2. Replace buffers monthly.

B. Troubleshooting procedures

XVI. ATTACHMENTS

Table to Determine the correct dilution factor from historical data.

Previous Data	Dilution Factor	Sample Size	Deionized Water	Total Sample
0 - 200	(-)	50 mL	0 mL	50 mLs
200 - 400	X2	25 mL	25 mL	50 mLs
400 - 1000	X5	10 mL	40 mL	50 mLs
800 - 2000	X10	5 mL	45 mL	50 mLs
1500 - 5000	X25	2 mL	48 mL	50 mLs
4000 -	X50	1 mL	49 mL	50 mLs

301.1

Total Coliform/E. coli MMO-MUG (Colilert) Presence/Absence Test

1. Description and Application

This procedure provides simultaneous detection of total coliforms and E. coli from all types of potable water samples.

2. Safety

All samples and cultures contain potentially harmful pathogenic organisms. Care must be taken not to contaminate either the work area or oneself. All spills must be decontaminated with Wescodyne solution for 5 minutes prior to cleanup. All personnel must wash hands thoroughly with disinfectant hand soap prior to all breaks and at the end of each shift.

3. Equipment and reagents

A. 35° Walk-in incubator
B. Colilert P/A reagent
C. Test vials - Clear plastic, sterile, calibrated at 100 ml.
D. 4 or 6 watt long wavelength (366 nm) ultraviolet
E. Racks
F. Colilert Color Comparator

4. Procedure

A. Thoroughly mix sample container by shaking 25 times.
B. Pour 100 ml of sample into test vial labeled with sample number. It is acceptable to use the sample container as the test vial by pouring off excess sample to the 100 ml calibration. Test vials should be placed in racks in numerical order (the same order as data sheets).
C. Aseptically empty contents of Colilert reagent vial into bottle containing sample. It is acceptable to add Colilert reagent to test vial prior to adding sample.
D. Close cap tightly and shake test vial to dissolve most of the reagent.
E. Rusty colored samples are marked with a "*" on bottle cap.
F. Place racks in 35° walk-in incubator, so that the 1st sample in the sequence (usually the lowest sample number) is at the back of the shelf and all the way to the right in the area

1130

IRON BACTERIA

1. Description and Application

This technique gives an approximation of the iron bacteria concentration using a microscopic technique. The presence of iron bacteria is generally associated with discoloration, odors and slime growth in drinking water.

2. Procedure

A. Pass an appropriate volume of sample through a .45 micron pore size 25 mm filter using a funnel with a useful filtration diameter of 16 mm. The proper volume is determined by the amount of debris present and the concentration of organisms. Typical volumes range from 0.1 ml to 100 ml.
B. Thoroughly dry the filter in a 35°C incubator for at least 4 hours.
C. Place the dried filter on a slide on top of 2 drops of immersion oil. Place an additional 2 drops of oil on top of the filter and add a coverslip. This step should produce a clear filter. (Wet filters do not clear well).
D. Using a calibrated microscope, view 15 fields at 430X and score the number of fields containing each species of Iron Bacteria. Ideally, organisms will be present in some but not all of the 15 fields examined.
E. Using the table in the Lueschow article (see Reference), calculate the theoretical density of Iron Bacteria species per ml. (15 fields = 1 ml with 100 mls filtered.)

3. Reference

Lueschow, L.A. and K.M. Mackenthun. "Detection and Enumeration of Iron Bacteria in Municipal Water Supplies." J.A.W.W.A. Vol. 54, No. 6, 1962. P. 751.

ESS
April, 1995

WSLH WISCONSIN STATE LABORATORY OF HYGIENE

Collecting a water sample

Complete the request form as fully as possible, writing only on the front of the form. Please read the information on the back of the request form to help you choose which test or tests you want done. Write your name as it appears in the address box on the side of the sample bottle.

Sample collection for drinking water

1. Locate a sample tap near the well, preferably not a swing, leaky or outside faucet. Remove any screens or aerators.
2. Sterilize metal taps by heating with a flame (butane lighter, propane torch, alcohol lamp, etc.). Eliminate this step for plastic taps.
3. Let the water run for several minutes. Do not change the flow rate, shut the faucet off, or wipe or wash the faucet.
4. Do not open the bottle until ready to collect sample. Take care not to touch the top of the bottle or the inside of the cap. Fill bottle to within 1/2 inch of the top.

Chlorinated water samples

Samples collected from continuously chlorinated water, such as city water supplies and swimming pools, must be collected in a special bottle containing a chlorine neutralizing substance. These special bottles are not appropriate for wells that have been temporarily chlorinated. Temporarily chlorinated wells should be pumped until they are free of chlorine prior to sampling.

Sample collection for swimming pools

Sample the pool during a period of average use. Using a sweeping motion, collect a sample from a depth of 18 inches. Be careful not to rinse the chlorine neutralizing substance from the bottle.

For more information

For further information, call (608) 262-1210 or write:
Water Microbiology Unit
State Laboratory of Hygiene
465 Henry Mall
Madison, WI 53706

See mailing instructions on reverse side

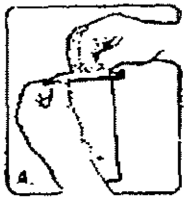
10/92 Hyg. 690

Mailing samples to the laboratory

Because old samples give inaccurate results, the laboratory should receive samples within 48 hours after collection. To ensure this, take the sample to the post office in time to have it processed before the last daily mail dispatch to Madison when it will not be delayed over a weekend or holiday.

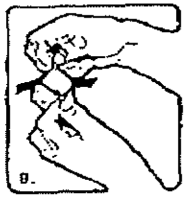
Be sure the request form is filled out and the sample bottle is placed in the plastic whirl-pak bag before placing it in the styrofoam container (See instructions below). Please use the color-coded return mailing address label provided.

Important: Post office requires sample bottle to be placed in a sealed plastic bag prior to mailing.



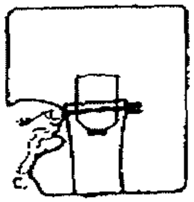
A.

Tear off top at scored line



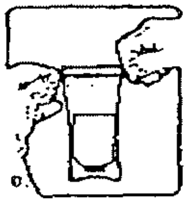
B.

Pull tabs outward to open bag. Sometimes a pull on the bottom is helpful.



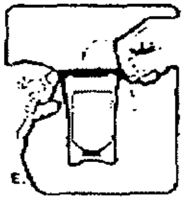
C.

Place sample bottle in Whirl-Pak bag.



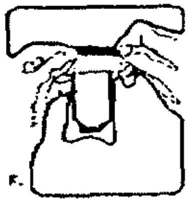
D.

Pull wire ends to close bag. Squeeze out excess air.



E.

Whirl bag 3 complete revolutions.



F.

Turn tape wire inward on opposite face of fold.

Secure shipping container with two rubber bands as pictured below.