WARZYN

Final Quality Assurance Project Plan (QAPP) 13076.12

Remedial Investigation/ Feasibility Study Wausau NPL Site Wausau, Wisconsin

Prepared for:

United States Environmental Protection Agency
Region V
Chicago, Illinois

Prepared by:

Warzyn Engineering Inc.
Madison, Wisconsin

September 1987

WARZYN

Engineers & Scientists
Environmental Services
Waste Management
Water Resources
Site Development
Special Structures
Geotechnical Analysis

September 22, 1987
13076.12

Ms. Margaret M. Guerriero
Wausau Project Officer
Environmental Protection Agency
5-HR-11
230 S. Dearborn Street
Chicago, IL 60604

Re: Revision of Quality Assurance Project Plan (QAPP)

Dear Ms. Guerriero:

Enclosed are three copies of the Quality Assurance Project Plans (2nd revision) for the Wausau NPL site RI/FS. Revisions were incorporated based on comments provided by the Quality Assurance Offices (QAO) on September 16, 1987 (received September 21). Where appropriate, these changes are designatd by the revision number and date of revision. Table 2 and Appendix D have been modified to reflect fast turnaround VOC analyses of existing wells by the CLP through SAS.

Please contact us if you have questions or comments on the revised QAPP.

Sincerely,

WARZYN ENGINEERING INC.

Dennis L. Iverson

Dennis L. Iverson, P.E.
Project Manager

CSR/jpl/KJQ
[jpl-600-21b]

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WARZYN

Remedial Investigation/ Feasibility Study Wausau NPL Site Wausau, Wisconsin

September 1987

QUALITY ASSURANCE PROJECT PLAN (QAPP) for

WAUSAU NPL SITE

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

Prepared by:

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Approvals:	Date:
Warzyn Site Project Manager	9/22/87
Warzyn Quality Assurance Officer	9/22/87
EPA Remedial Project Manager	9/23/87
EPA Region V Quality Assurance Officer	

SECTION 1.0 - INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, functional activities and specific Quality Assurance (QA) and Quality Control (QC) activities associated with the Remedial Investigation/Feasibility Study (RI/FS) at the Wausau NPL Site in Wausau, Wisconsin. The QAPP is designed to achieve the specific data quality goals of the RI/FS.

The United States Environmental Protection Agency (U.S. EPA) requires contractors performing Remedial Investigations (RIs) to participate in centrally managed QA programs. This requirement applies to environmental monitoring and measurement efforts funded by the U.S. EPA.

The contractor generating data has the responsibility to implement minimum procedures so that precision, accuracy, completeness, and representativeness of data collected are known and documented. This responsibility is uniformly met through preparation of a written, site-specific quality management plan, or QAPP.

This QAPP has been prepared using the following guidance documents:

- U.S. EPA December 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80
- U.S. EPA Region V, December 1985, Preparation of Federal-Lead Remedial Investigation Quality Assurance Program Plans for Region V, Draft

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3.0 - PROJECT DESCRIPTION

This RI/FS consists of an assessment of volatile organic compound (VOC) contamination in groundwater in the vicinity of City of Wausau Municipal Wells. The objectives of the Remedial Investigation (RI) are:

- Identification of contaminant sources
- Determination of the nature and extent of impacts
- Determination of probable fate of contaminants through time
- Assessment of the danger to public health associated with the contamination
- Development of remedial action alternatives to address varying levels of response
- Evaluation of response actions utilizing environmental, public health, economic, institutional, and technical reliability criteria

A variety of field activities have been proposed for this purpose. The RI will be conducted in such a manner as to gather data necessary to support the Feasibility Study (FS). Tasks and subtasks are directed toward accomplishing project objectives.

3.1 - Background

The City of Wausau is located in Marathon County along the Wisconsin River in the north-central region of Wisconsin. The City provides drinking water for approximately 33,000 residents primarily from 5 groundwater production wells (see Figure 1). Well CW-8, located north of Well CW-3 is used infrequently because of high iron concentration. Three of these wells, CW3, CW4 and CW6, are contaminated with volatile organic compounds (VOCs). The City, with the support of Wisconsin Department of Natural Resources (WDNR), U.S. EPA and several local industries have made several attempts to mitigate the problem and locate contaminant sources. Thus far, the results are inconclusive.

In Mid-1983, Wausau drinking water exceeded recommended drinking water standards for VOCs. In 1984, the City of Wausau and WDNR contacted the U.S. EPA for emergency assistance. The U.S. EPA Emergency Response Group installed temporary granular activated carbon (GAC) adsorption units on Production Well CW6 and a VOC stripper at Production Well CW4 in August, 1984. A second stripper has been used at Production Wells CW3 and CW6. The temporary GAC units were shut down on October 29, 1984. Recently, the City has been blending treated water with well water from uncontaminated supply sources to reduce VOC concentrations in the water distribution system.

Prior to treatment (before July 1984), the water supply contained trichloroethene TCE ranging from detectable levels to 80 ug/L. Shortly after discovery of the contamination, but probably before blending had reduced levels of VOCs, lower levels of tetrachloroethene (PCE) and dichloroethene (DCE) were found. After installation of the water treatment systems, the water distribution system had relatively low levels of VOCs.

Wausau Chemical Company is located between Production Wells CW3 and CW4 (see Figure 3) and has had documented releases of PCE. High concentrations of VOCs (e.g., PCE/TCE/DCE of 20,000/8,800/8,800 ug/L at Well WC6A in April 1985) have been detected in the groundwater beneath this facility. However, Wausau Chemical Company is presently operating an extraction and treatment system. The results of these measures are not available at this time.

3.2 - Topography and Hydrology

The surface topography in the Wausau area is controlled by the underlying Pre-Cambrian bedrock topography, glacial deposition and post-glacial erosion. The glacial terrain consists of relatively low rolling hills marked with topographic highs formed by the bedrock. The Wisconsin River has created a major valley and flood plain by erosion into the glacial deposits. Valley slopes are very steep along the river in the west study area and are lower in the vicinity of the east study area.

The Wisconsin River is controlled by a dam in downtown Wausau, which is south (downstream) of the study area. Therefore, the river stage within the study area is relatively controlled. A stream gauging station on the river is operated by the Wisconsin Valley Improvement Company and will provide river stage records needed in the modeling and analysis of groundwater flow. The U.S.G.S. maintains a stream gauge at the City of Merrill, located approximately 20 miles upstream. Records from both the Wausau and Merrill stations will be used in establishing historical stage levels.

3.3 - Hydrogeology

The aquifer within the Wausau area consists of glacial outwash deposits. Figure 2 is a cross-section of the east study area, which illustrates representative study area soil types. The outwash deposits appear laterally extensive and consist of fine-to-coarse sand. Existing studies report only minor silty sand units within the aquifer. The aquifer thickness is controlled by the topography on the Pre-Cambrian igneous and metamorphic bedrock. Although the bedrock is used as an aquifer for domestic supplies in some areas, it has a very low hydraulic conductivity relative to the sand aquifer.

The bedrock topography has been characterized through seismic surveys on both sides of the river (Weston, 1985) and through wells which have extended to bedrock. These sources indicate a major bedrock valley that roughly parallels the present Wisconsin River (see Weston, 1985 bedrock maps). The bedrock slopes to the east at a relatively uniform, moderate slope (36 feet/mile) in the vicinity of Production Wells CW3 and CW4. The bedrock surface near Production Wells CW6, CW7 and CW9 slopes upward to the northwest, away from the river. The slope is cut by two deep east-west trending tributary valleys that are approximately 100 feet deep.

Under normal conditions, the Wisconsin River is a regional discharge area for groundwater flow, which occurs primarily in the sand aquifer (Lippelt and Hennings, 1981). Although river stage records are not presented with the

groundwater levels shown in Figures 3 and 4, the groundwater levels in the vicinity of municipal production wells are below the river stage indicating that the City production wells are inducing infiltration from the river. Fluctuations in river stage apparently have a strong effect on the rate of recharge from the river, which in turn, affects groundwater flow directions and the amount of dilution of VOCs at certain well locations.

Figure 2 presents the vertical distribution of observed groundwater heads and inferred groundwater flow directions in the east study area. The vertical flow net was developed from groundwater levels recorded by Roy F. Weston Inc. (1985). The divide located between Production Wells CW3 and CW4, shown on the water table map, was very close to Well CW4 at the time of the water level monitoring. The small cone of depression shown at Well CW4 was probably due to the infrequent pumpage at Well CW4 (see Figure 6). The divide between Wells CW3 and CW4 extended below the Wausau Chemical Co. site. Downward vertical flow occurred at Monitoring Well Nest WC3, whereas upward flow occurred in the shallow portion of the aquifer at Monitoring Well Nest WC5. Present groundwater flow conditions may have changed substantially due to the installation and operation of groundwater extraction wells at the Wausau Chemical Company facility.

Groundwater recharge from the vicinity of Monitoring Well Nest WC3 appears to flow deep within the aquifer and discharge to Production Wells CW4 and CW3. Recharge that occurs west of Monitoring Well Nest WC3 appears to remain relatively shallow, but appears to discharge to Well CW3 and/or the Wergin well. Recharge at the Wausau Chemical Company site may or may not discharge to the Wisconsin River, depending on river stages and pumping conditions.

Recent hydrogeologic investigations (RMT/Geraghty & Miller Inc., 1987) indicate Production Wells CW6, CW7 and CW9 exert considerable influence on groundwater flow conditions on the west side of the Wisconsin river (Figure 4). Water levels recorded during sustained production well pumpage indicate a large asymmetric cone(s) of depression that is several thousand

feet in diameter. The orientation of the cone of depressions appears to be strongly controlled by the relative pumping rates of the three production wells and the proximity of induced recharge areas (Wisconsin River and Bos Creek). Comparison of groundwater levels at Monitoring Well Nest R1D/R1S, R2D/R2S, and R3S/R3D indicates strong vertically downward gradients (±0.007 ft/ft to 0.025 ft/ft) (recharge conditions) relative to the horizontal gradient (±0.01 ft/ft) These vertically downward gradients may be the result of induced recharge caused by production well pumpage, by a small impoundment area on Bos Creek or by a combination of these factors.

3.4 - Groundwater Quality Based on Prior Investigations

East Study Area

The inferred vertical distribution of contaminants between Production Wells CW3 and CW4 is shown in Figure 5. The contaminant plumes shown on this cross-section are based on the inferred flow directions shown in Figure 2 and the VOC concentrations shown on the cross section. The contaminants present and the flow directions between Monitoring Well WC5A and the wells surrounding the Wergin well indicate a probable continuous plume below some installed wells.

The plume observed at the groundwater surface in Monitoring Well WC6A and at depth at Well WC3A contains all three of the primary VOC's (PCE/TCE/DCE) identified at Production Wells CW3 and CW4. The flow directions between Well WC3A and Production Well CW3 indicate a probable continuous plume below the installed monitoring wells.

Figure 6 presents a comparison of the total VOC concentrations observed at selected wells on east side of the river and their pumping history. Observations that can be made from these comparisons are:

- VOCs were observed at Well CW4 only after it was placed into continuous use for several months.
- The VOCs at the Wergin well decreased substantially after Well CW3 pumpage was shifted to Well CW4 for several months in 1982.

- The VOCs at Monitoring Well Nest MW10 in the vicinity of Marathon Box appear to be strongly related to the pumping rate at Production Well CW4.

These observations indicate that Production Well CW4, even when pumped intermittently, has an affect on VOC concentrations at Well CW3 and the Wergin Well. Based on this information, it appears the source(s) is (are) influenced by pumpage by both Production Wells CW3 and CW4, and are probably located between the two wells. The correlation of concentration at Monitoring Well Nest MW10 with pumpage at CW4 indicates that as the cone of depression from Well CW4 developed during 1984 and then again in 1986, the flow direction changed and Monitoring Well Nest MW10 intercepted contaminant flow, probably from the north. Prior to the continuous pumpage at Well CW4, this contaminant plume may have been migrating toward Production Well CW3 and the Wergin Well.

West Study Area

Prior to the RMT investigation of Marathon Electric, several monitoring wells were installed in the west study area with little success in identifying the contaminant plume affecting Production Well CW6. The four monitoring wells installed near Production Well CW6 (Wells MW5, MW6, MW7, MW8) were all placed above the screened interval of Pumping Well CW6. Based on observations from the east study area (see cross-section in Figures 2 and 5), the flow to a pumping well which is pumping from the base of the aquifer creates a downward vertical gradient at some distance away from the pumping well. Therefore, unless the source is immediately adjacent to the pumping well, contaminants are not expected to be present above the screened interval of the pumping well.

The RMT/Geraghty and Miller (1987) investigation of the west study area (see Figure 7) indicates that groundwater flow in the vicinity of the former City of Wausau Landfill is toward the southeast and the river. Groundwater flow in that area does not indicate an influence from the municipal wells to the north. The water quality generated for that study indicated a TCE concentration of between 280 to 1,058 ug/L at Well C2S. This well may indicate the southern extent of a plume on the perimeter of the cone of depression for Well CW6.

3.5 Project Objectives and Use of Data

The RI will be performed to gather and assess information needed to accomplish the following objectives:

- Identification of contaminant sources;
- Characterization of on-site physical features and facilties that could affect contamination, migration and remediation;
- Determination of the nature and extent of impacts;
- Determination of probable fate of contaminants through time;
- Assessment of the dangers to public health asocaited with the contamination;
- Development of remedial action alternatives to address varying levels of response; and
- Support future enforcement action under CERCLA.

The following tasks have been formulated to achieve these objectives:

Task 4 - Site Investigation

Subtask 2.1.3 - Preparation for Site Investigation

In preparation for the field gas chromatography (GC) analysis of soil gas and water quality samples, a short method development and detection limit study will be conducted in the lab. This study will establish the most efficient and effective methods (i.e. detection limits, temperature programs, column type and standard preparation) to detect the primary compounds of interest; PCE, TCE and DCE. The data from the soil gas and water samples analyzed by on-site GC are intended to be used only as a guide for well locations and screen placements (see Appendix F for methodology). The data will be reported, but considered as estimated and tentatively identified due to the lack of second column confirmation. Sample documentation will be limited to the sampler's and field analyst's notebooks.

Subtask 2.1.4 - Existing Well Sampling

Selected existing monitoring wells (51) and production wells (5) will be sampled for water quality. The purpose for this sampling and analysis is to provide information on existing site conditions at the beginning of the study. Although there is a substantial amount of existing data, the changes in pumping rates of the City wells may have affected the plume distribution since the last sampling. These data are intended to be used to assess existing conditions in order to optimize field investigation activities.

Pending approval by the Central Regional Laboratory (CRL), VOC analyses will be performed (see Warzyn Standard Operating Procedure, Appendix G) by Warzyn using gas chromatography in order to provide fast turnaround (approximately 7 days). If dilution of samples is required, Warzyn will report results for both original and diluted sample analyses. The fast turnaround will make data available for developing the field investigation strategies including: soil gas investigation locations and monitoring well locations and depths. The fast turnaround VOC analysis data is essential to subsequent soil gas investigations which must be conducted before ambient soil temperatures become too low to volatilize existing organic compounds. If Warzyn cannot meet CRL requirements, the fast turnaround VOC analyses (through SAS, see Appendix D) will be submitted for contract laboratory bid.

In addition, samples will be analyzed by CLP (SAS) for the following water quality parameters (See Appendix D for Methodology):

- Calcium*
- Magnesium*
- Alkalinity
- Sulfate
- Chloride
- Iron*
- Nitrate+Nitrite-Nitrogen
- Ammonia-nitrogen
- Total Kjeldahl nitrogen
- Total Organic Carbon
- Sodium*
- Potassium*

* Samples for these parameters will be field filtered prior to preservation.

Field measurements for pH, specific conductance and temperature will also be performed on these samples. (See Appendix C1-C4)

The above indicator parameters, here and in other subtasks, will fill the following data needs. TOC will be used as a non-specific indicator of organic carbon concentration. It will be used with results of VOC analyses to indicate the extent to which dissolved organics have been identified and as an indicator of total carbon that may require treatment. Nitrogen data will be used for comparison to water utility records (nitrate-nitrite) to obtain an indication of potential reducing conditions or possible anaerobic biological activity (ammonia-N versus oxidized N species), and to provide an indication of the abandoned landfill as a contaminant source (ammonia-N, TKN). The metals are commonly occurring cations, to be used as general water quality indicators in evaluation of the groundwater geochemistry. Data for all but potassium can be compared to water utility records. Calcium, magnesium and iron in particular are relevant to existing water treatment plant processes (Fe, Mn removal and softening) and consideration of alternative source water quality. Chloride and sulfate are commonly occurring anions, to be used in conjunction with alkalinity as general water quality indicators. Comparison of major cation and anion concentrations to conductivity data will be useful in assessing data consistency and possibly indicating whether additional major ionic species may remain unidentified.

Subtask 2.1.5 - Soil Gas Survey

A soil gas survey will be conducted in order to help evaluate areas as potential VOC source areas and to attempt to map the distribution of a plume that may be present at the water table surface in the immediate area of potential VOC sources. Results of on-site GC analyses of the soil gas samples for VOCs (see Appendix F) will be used only as a guide in the selection of well locations. Data reported will be considered estimated and tentatively identified.

Soil gas sampling will be conducted at an estimated 10 facility locations. Seven of the soil gas investigation areas are shown in Figures 7 and 8. The remaining 3 locations are anticipated new potential sources which may be identified through an industrial survey of the area. Table 1 lists sites to be surveyed and tentative numbers of samples.

Soil gas sampling will consist of driving a probe to a depth of approximately 3 ft, purging the sampling probe and tubes and collecting a sample in a glass gas sampling container. The sample will be returned to the on-site GC for analysis. Sample blanks will be collected between each facility being surveyed. Results of the soil gas analyses will be plotted and used to evaluate each facility as a potential VOC source, to locate additional soil gas sampling sites on the property if needed, and to identify areas where direct groundwater sampling is required. Soil gas sample locations may also be dependent on the results of water quality analyses of samples collected during drilling operators.

Subtask 2.1.6 - Well Drilling

Thirty-nine (39) wells, 14 at new locations and 8 wells nested adjacent to existing wells are planned. An additional 15 wells are planned to be installed based on information obtained in the field. Locations of and rationale for these wells are presented in Figures 7 and 8.

Split spoon sampling will be performed at the deepest well in each nest on a 5 ft interval to a depth of 25 ft and a 10 ft interval to the bottom of the hole or at changes in soil types. Samples will be logged by a geologist or geotechnical engineer present at the drilling rig. Each of the deep wells will be logged using a natural gamma ray logging tool, a Mount Sopris 1000C unit. The gamma ray log will provide information on the clay content of the formations penetrated and will be used in selecting the vertical position of the well screen (See Appendix E for operating instructions).

Thirty (30) soil samples will be analyzed for grain size distribution and 15 for organic carbon content (see Appendix D for methods). These data will be used to classify the soils, and estimate hydraulic conductivity and potential adsorption capacities of the soils. [Thirty soil samples will be submitted to the CLP for organic target compound analyses.] These data will be used to characterize contamination in the unsaturated zone at potential contaminant source areas.

Approximately, one hundred six (106) water quality samples will be collected during drilling operations. Sample locations will be selected to help identify the plume distribution prior to setting of well screens. These samples will be analyzed for VOCs using the on-site GC. Results will be used to help identify the vertical and horizontal plume distribution, so that the well screen can be placed within the plume. The location of additional wells may also be determined based on an improved knowledge of plume distribution. The results of the on-site VOC analyses are to be used primarily for making field decisions.

Twenty (20) of the above mentioned groundwater samples will be submitted to CLP SAS for VOC analysis following the specified procedures (See SAS request in Appendix D). The requested method is consistent with that for analyses of groundwater samples collected in Subtask 2.1.8. The rationale for using the SAS methodology is to obtain lower detection limits than available through CLP RAS.

Subtask 2.1.7 - Surface Water and Sediment Investigation

Six surface water and six sediment samples will be collected from the Wisconsin River and Bos Creek to supplement the results of a prior WDNR survey. The locations (see Figures 7 and 8) will be in the Wisconsin River, one upstream and one downstream of the Wausau Chemical Co. site, one upstream and one downstream of the City of Wausau landfill, and two in Bos Creek. These samples will be used to determine if volatiles have been released at each respective location. A sediment sample will be collected at each surface water sampling location. Sediment samples will be analysed for VOC's using CLP RAS.

Surface water samples will be submitted to CLP for VOC analysis following specified procedures (See SAS request in Appendix D.) The rationale for using the SAS methodology is to obtain the lower detection limits than those available through CLP RAS. In addition, surface water samples will be analyzed for the following water quality parameters: (see Appendix D for methodology).

- Calcium*
- Magnesium*
- Alkalinity
- Sulfate
- Chloride
- Iron*
- Nitrate + Nitrite - Nitrogen
- Ammonia - Nitrogen
- Total Kjeldahl nitrogen
- Total Organic Carbon
- Sodium*
- Potassium*

* Samples for these parameters will be field filtered prior to preservation.

Filtration of samples for metals analysis will allow comparability of results with groundwater results. In addition, surface water samples will be analyzed for pH, specific conductance and temperature in the field (See Appendices C1-C4 for methodology).

Surface water and sediment data are intended to be used in characterizing the nature and extent of contaminants and in evaluating potential remedial actions.

Subtask 2.1.8 - Groundwater Sampling and Aquifer Testing

One hundred seventeen (117) monitoring wells and 5 production wells will be sampled and analyzed for groundwater quality during this task. Analytical results will form the basis for the RI analysis, including source identification, extent of contamination, determination of the mass of contaminants present, and the evaluation of remedial action alternatives.

Groundwater samples will be analyzed for VOCs and the following water quality parameters by CLP SAS. (See Appendix D for methodology).

- Calcium*
- Magnesium*
- Alkalinity
- Sulfate
- Chloride
- Iron*
- Nitrate+Nitrite-Nitrogen
- Ammonia-nitrogen
- Total Kjeldahl nitrogen
- Total Organic Carbon
- Sodium*
- Potassium*

* Samples for these parameters will be field filtered prior to preservation.

The rationale for analyzing for VOCs using SAS is to obtain lower detection limits. Twenty wells will be selected for analysis of the full CLP organic and inorganic target lists (see Appendix B for lists).

Hydraulic conductivity will be determined at 20 wells. Tests are to be performed in water table wells by removing or displacing a slug of water from the well and measuring recovery through the use of a pressure transducer and data logger. The tests will be conducted in piezometers using air pressure to provide additional head within the well. Response will be measured with a pressure transducer and data logger. The data that are collected will be used to calculate the hydraulic conductivity of the aquifer.

Subtask 2.1.9 - Groundwater Level Monitoring

At the completion of the well installation program, water levels will be measured at all on-site wells. Water levels will also be measured concurrent with each groundwater sampling effort. Six additional rounds of water levels will be collected during the course of the RI to record fluctuations in water levels. Water levels will be measured using fiberglass tape and attached sounding device or an electronic water level indicator. Groundwater levels will be measured to determine vertical and horizontal groundwater hydraulic gradients at the site.

Subtask 2.1.10 - Sampling Summary

Table 2 provides a summary of all anticipated sampling that will occur in conjunction with the RI/FS at the Wausau NPL site. The table is compiled by subtask and matrix type. The table lists parameters, number of samples collected, the analyzing lab and the estimated number of QC samples.

4.0 - PROJECT ORGANIZATION

4.1 - Overall Responsibility

- Edward Kruel - Project Manager - WDNR
- Margaret Guerriero - Remedial Project Manager - U.S. EPA Region V
- Dennis Iverson, Project Manager, Warzyn Engineering Inc.
- Daniel W. Hall, Project Administrator, Warzyn Engineering Inc.
- RI/FS Reports prepared by Warzyn Engineering Inc.

4.2 - Monitoring Sample Operators and QC

- Principal Engineering Firm - Warzyn Engineering Inc.
  One Science Court, Madison, WI
- Well Installation - to be determined
- Sampling and Monitoring - Supervised by Warzyn Engineering Inc.
- Surveying - Warzyn Engineering Inc.
- Quality Control - Warzyn Engineering Inc.
  Richard Maurer (Quality Assurance Officer)

4.3 - Laboratory Analysis and QC

- Contract Laboratory Program (RAS and SAS) - CPMS, CRL, Region V
  - VOCs
  - Alkalinity, chloride, sulfate, nitrate-nitrogen, TKN, TOC, calcium, magnesium, sodium, potassium, and iron
- Field analysis of pH, specific conductivity and on-site GC screening, laboratory analysis of VOC - Warzyn Engineering Inc. - Michael Linskens, Manager of Analytical Services

4.4 - Specialized Responsibilities for Laboratory Services

Contract Laboratory Program (CLP)

- CLP Routine Analytical Services (RAS)
  - Request initiated by Warzyn
  - Support Services Branch, Office Emergency and Remedial Response, U.S. EPA headquarters - overall management of CLP
  - U.S. EPA EMSL, Las Vegas - Quality Assurance oversight of CLP laboratories
  - Final data review - U.S. Region V Contract Project Management Section, CRL
  - Review of tentatively identified compounds and assessment of need for confirmation - Warzyn Engineering Inc.

- CLP Special Analytical Services (SAS)
  - Request initiated by Warzyn Engineering Inc.
  - Request coordinated through U.S. EPA Region V Environmental Services Division or U.S. EPA Region V Remedial Response Branch or U.S. EPA RPM
  - Review of SAS specification - U.S. EPA Region V QA Officer and CRL
  - Final data reviewed - U.S. EPA Region V CPMS, CRL

4.5 - Quality Assurance

- Overall QA Responsibility - Warzyn Engineering Inc. Quality Assurance Officer (except CLP analyses)

- Warzyn Engineering Inc. and Warzyn subcontracted activities - WDNR, U.S. EPA Region V

- CLP (RAS)
  - Support Services Branch, OERR, EPA headquarters
  - EML Las Vegas, EPA
  - CPMS, CRL,
  - EPA, QAO Region V

- CLP (SAS)
  - CPMS, CRL
  - Region V QAO
  - Warzyn Engineering INc.

- Field Analysis - Warzyn Engineering Inc.

- QAPP Review - QAO, Region V

4.6 - Performance and Systems Audits

- Field operations - QA officer, Warzyn Engineering Inc.

- CLP-Support Services Branch, OERR, EPA and EMSL-Las Vegas, EPA

- Evidence Audit - EPA

- Field GC analysis - Warzyn Engineering Inc.

A project organization chart is shown in Figure 11.

5.0 - QUALITY ASSURANCE OBJECTIVES

Measurements will be made during this study to characterize the nature and extent of site contamination and to aid in identifying appropriate sampling locations for site characterization. For the former, the overall QA objective is to develop and implement procedures of field sampling, chain-of-custody, laboratory analysis and quality control (QC) reporting that will provide legally defensible results of documentable quality. These data will be used to identify the nature and extent of contamination in the aquifer, including the source area(s), evaluate potential remedial actions and evaluate potential risks to human health and the environment. Specific procedures to be used for sampling, chain-of-custody, calibration, laboratory analysis, reporting, internal quality control, audits, preventative maintenance, and corrective actions are described in other sections of this QAPP. For analysis used to aid sampling site selection, the overall QA objective is to obtain data of documentable quality that will allow a relative ranking of potential sampling locations to be made.

5.1 - Level of Quality Control Effort

5.1.A - Field Sampling Program

The quality of data from the field sampling program will be evaluated through the collection of field duplicate, matrix spike/matrix spike duplicate and field and trip blank samples. Duplicate samples will be used to assess the combined effect of sample collection, handling and analysis on data precision. Blank samples will be used to check for procedural contamination or naturally occurring conditions at the site that may cause contamination. The general level of effort for all matrices will be one field duplicate per 10 investigative samples and one field blank per 10 investigative samples. However, field blanks will be collected at a frequency of one per collection method per sampling event or day. For organics analysis of water samples, triple the normal sample volume will be collected for matrix spike/matrix spike duplicate analysis at a frequency of one per 10 investigative samples. One trip blank will be included with each batch of water samples for volatile analysis.

Accuracy and reproduceability standards for survey activities will follow guidelines in the standard survey reference, Classification Standards of Accuracy and General Specifications of Geodetic Control Survey, prepared by the Bureau of Land Management. Horizontal locations will be obtained to an accuracy of ±0.1 foot. Vertical elevations will have an accuracy of±0.1 foot for the ground surface and ±0.1 foot for well casings.

5.1.B - Laboratory Analysis

Surface water and the majority of groundwater samples collected will be analyzed using the U.S. EPA Contract Laboratory Program (CLP). The QC goals of CLP RAS are established under guidelines stated in Invitation for BID (IFB) documents WA-85-J664/J680 for organics and WA-85-J838/J839 for inorganics. The level of laboratory QC effort for CLP Special Analytical Services (SAS) Analyses is described in the individual SAS Requests, attached in Appendix D.

5.1.C - Field Measurement of pH, Specific Conductance, and VOCs in Groundwater

Level of QC effort for field measurements of pH will consist of daily precalibration using two buffer solutions and calibration verification at regular intervals (at least every ten samples).

Level of QC effort for specific conductance measurements will consist of initial and continuing calibration verification using a standard solution of known conductivity (at least every ten samples). Procedures for operation and maintenance will follow those recommended by the manufacturer (see Appendices C-1 and C-4).

Level of QC effort for field GC will consist of initial and continuing calibration verification. Procedures for operation and maintenance will follow those described Appendix F. The level of QC effort described for field sampling activity (Task 5.1.A.) will also apply to field GC analysis for VOCs.

5.2 - Accuracy, Precision, and Sensitivity of Laboratory Analyses

The majority of groundwater and all surface water, soil and sediment samples collected will be analyzed using the Contract Laboratory Program (CLP). The QA goals of routine analyses provided by the CLP are established under guidelines stated in IFBs WA-85-J664/J680 for organics and WA-85-J838/J839 for inorganics. Goals for CLP Special Analytical Services (SAS) requests are listed with methods descriptions in Appendix D. The QA goals of the analytical testing to be performed by Warzyn are provided in Appendix G.

5.3 - Completeness, Representativeness and Comparability

It is anticipated that at least 95% of analyses will provide results meeting acceptance criteria. Sampling methods and locations are designed to provide results representative of the matrix at the sampling point. Analytical methods used will provide comparable data which will supplement data previously collected at the site.

5.4 - Field Measurements (for which samples are not collected)

Measurement data will be generated in many field activities that are incidental to collecting samples for analytical testing or unrelated to sampling. These activities include, but are not limited to, the following:

- Documenting time and weather conditions
- Semi-quantitative total organic vapor screening of soil and water samples using a photoionization detector (e.g., HNu or equivalent) and/or a flame ionization detector (e.g., OVA).
- Determination of depths to water in a borehole or well
- Natural gamma ray logging (Mount Sopris 1000C)
- Verifying well development and pre-sampling purge volumes

The general QC objectives for such measurement data are to obtain reproducible and comparable measurements to a degree of accuracy consistent with the intended use of data through the documented use of standardized procedures.

6.0 - SAMPLING PROCEDURES

Specific sampling procedures are described in the Sampling Plan (Appendix A) Table 2 summarizes numbers of samples to be collected. Table 3 summarizes containers, preservatives, holding times, transport and storage methods.

7.0 - SAMPLE AND DOCUMENT CUSTODY PROCEDURES

Except for samples collected to aid in locating appropriate sampling locations, chain of custody protocol will follow procedures detailed in NEIC Policies and Procedures, (EPA-330/9-78-001R, May, 1978, Revised May 1986). This includes use of chain of custody forms, custody seals, sample tags, traffic reports, container labels and field notebooks for sample documentation. This documentation will include sampling time, location, tag numbers, designation and samplers. Pertinent field screening (e.g., PID) readings, weather conditions, and field modifications of sampling strategy will be recorded. Original field notes and field documents will be maintained by Warzyn in an evidence file until transferred to EPA where the final evidence file will be maintained. The CRL will maintain CLP related records.

Documentation for samples analyzed by field GC will be recorded in a bound field laboratory notebook and in field notebooks kept by sampling team members. Field laboratory and field sampling methods will be entered into the file maintained by Warzyn following completion of field work.

8.0 - CALIBRATION PROCEDURES, FREQUENCY AND PREVENTATIVE MAINTENANCE FOR FIELD INSTRUMENTS

Field instruments will be inspected and calibrated at Warzyn's analytical laboratory prior to being taken to the field. Calibration and maintenance of pH and specific conductance meters are detailed in Appendices C-1 through C-4.

In the field, the pH meter will be calibrated using the two-buffer standardization method prior to use and recalibrated using a pH 7 buffer every tenth sample. The calibration of the conductivity meter will be tested using a check standard. If readings vary more than 5% from expected values, the unit will be replaced.

Calibration of survey instruments used for health and safety purposes will follow procedures recommended by the manufacturer (Appendices C-5 through C-7). The HNu and TIP will be calibrated at the beginning of each work day using standard calibration gas (isobutylene) supplied by HNu. The OVA will be calibrated once per month (using methane) as specified by the manufacturer, unless conditions warrant more frequent calibrations. These conditions might include extensive use of instruments, particularly rough treatment or alteration of initial calibration conditions.

9.0 - ANALYTICAL SERVICES

9.1 - CLP RAS

9.1.A - Analytical Calibration Procedures

All samples analyzed by CLP RAS will conform to the guidelines in the User's Guide to the U.S. EPA Contract Laboratory Program and to those specified in IFBs W-85-J664/J680 for organics and WA-85-J838/J839 for inorganics.

9.1.B - Internal Quality Control

Internal Quality Control procedures for analyses will follow CLP guidelines as specified in IFBs W-85-J664/J680 for organics and WA-85-J838/J839 for inorganics. Field blank samples will be collected to check for contamination resulting from ineffective decontamination subsequent handling or procedures. Duplicate samples will be collected to assess data precision.

9.1.C - Performance and System Audit

Performance and systems audits for the CLP are the responsibility of the Support Services Branch, OERR, EPA and EMSL-Las Vegas, EPA.

9.1.D - Data Assessment/Validation

The assessment of data accuracy and precision is the responsibility of CPMS and the CRL QC coordinator. Guidelines for validation are contained in U.S. EPA Technical Directive Document No. HQ-8410-01. Laboratory Data Validation, Functional Guidelines for Evaluating Organics Analyses, May, 1985, for organic analyses and contained in Laboratory Data Validation, Functional Guidelines for Evaluating Inorganics Analyses, November, 1985, for inorganics analyses. The fraction of analysis results meeting specified QC-criteria (data completeness) will be checked by Warzyn and the SMO. Where test data have been reduced, the method of reduction will be described.

9.1.E - Accuracy/Precision Definitions

Accuracy and precision definitions for analyses performed by CLP RAS are listed in IFBs WA-85-J664/J680 and WA-85-J838/J839 for organics and inorganics, respectively.

9.1.F - Corrective Action

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

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

9.2 CLP SAS

9.2.A - Analytical and Calibration Procedures

Analytical procedures for samples analyzed by SAS are specified in SAS-Regional Request Forms found in Appendix D. Calibration of instruments will

follow procedures specified in analysis methods cited, except where noted on SAS requests.

9.2.B - Internal Quality Control

Quality control requirements for each of the SAS are analyses are specified in Appendix D. Field blank and duplicate samples will be collected and submitted for analysis to determine if sample contamination is due to field sampling equipment and data precision, respectively.

9.2.C - Performance and Systems Audits

Systems audits and required performance limits are specified for each CLP-SAS analysis in Appendix D.

9.2.D - Data Assessment/Validation

The assessment of data accuracy and precision is the responsibility of the CPMS and the CRL QC coordinator. The fraction of analyses results meeting specified QC-criteria (data completeness) will be checked by Warzyn and the SMO. Where test data have been reduced, the method of reduction will be described. Performance criteria for data validation are listed with methods descriptions in Appendix D.

9.2.E - Accuracy and Precision Definitions

Accuracy and precision definitions are specified for each CLP-SAS analysis in Appendix D.

9.2.F - Corrective Actions

If quality control audits detect unacceptable conditions or data, samples should be re-analyzed if holding time criteria permits. The Program Coordinator (Charles Elly or Jay Thakkan) of the Contract Project Management Section will be contacted by the performing laboratory, if requirements are not met upon reanalysis of samples.

10.0 - QUALITY ASSURANCE REPORTS

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

KDF/jap/RCW/DWH
[jap-600-30]

TABLE 1
SOIL GAS SAMPLING LOCATIONS
WAUSAU WELL FIELD RI/FS

LOCATION	NUMBER OF SAMPLES	COMMENTS
East Side		
1-Railroad Line	15	200 foot spacing along rail line with double line of samples on southern half of area
2-Marathon Press	8	locations based on plant survey
3-Marathon Box	8	locations based on plant survey
4-Wausau Chemical	12	locations based on review of the most recent reports
5-Wausau Energy	6	locations based on plant survey
West Side		
6-Marathon Electric	16	locations based on plant survey and observed TCE concentrations at Monitoring Wells C25, R4D
7-Outer Arc-Well CW6	15	200 foot sample spacing around 3000 feet of arc West and South of Well CW6.
8-Well CW6	13	200 foot sample spacing around Well CW6 to evaluate potential sources within an outer arc extending from Pearson Street north of Well CW6 to Burns Street SW of Well CW6 (see Figure 7).
	Total 93 samples	

13076.12
CSR/jap/DLI
[jap-600-30g]

TABLE 2
SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
WAUSAU NPL SITE

1) Matrix	2) Field Parameters	3) Lab	Number of Samples	Duplicates	Field Blanks	4) MS/MSD	5) Matrix Total	6) Test Parameters
Groundwater existing wells, Subtask 2.1.4, Phase I	pH, specific conductivity, temperature	CLP (SAS) CLP (SAS)	56 56	6 6	6 6	6 **	68 68	VOC, VOC, ALK, Sulfate, Chloride Nitrite + Nitrate Nitrogen TKN, TOC, Ca, Mg, Fe, Na, K, NH_3
Groundwater new and existing wells Subtask 2.1.8 Phase I	pH, specific conductivity, temperature	CLP (SAS) CLP (SAS)	122 122	12 12	12 12	12 **	146 146	VOC, ALK., Sulfate, Chloride Nitrate + Nitrite Nitrogen TKN, TOC, Ca, Mg, Fe, Na, K, NH_3
		CLP (RAS)	16	2	2	2	20	HSL Parameters
Surface Water Subtask 2.1.7 Phase I	pH, specific conductivity,	CLP (SAS) CLP (SAS)	6 6	1 1	1 1	1 1	8 8	VOC Alk, Sulfate, Chloride, Nitrate+Nitrite Nitogen, TKN, TOC, Ca, Mg, Fe, Na, K, NH3
Sediment Subtask 2.1.7 Phase I		CLP (RAS)	6	1	1	1	8	VOC
Soil Gas Subtask 2.1.5 Phase I	On-site GC VOC-screen	Field GC	93	*	*	*	93	VOC
Groundwater during drilling Subtask 2.1.6 Phase I	On-site GC VOC-screen	Field GC CLP (SAS)	106 20	* 2	* 2	* 2	106† 24	VOC VOC
Soil Subtask 2.1.6 Phase I		CLP (SAS) CLP (SAS) CLP (RAS)	26 13 26	3 1 3	1 1 1	-- --	30 15 30	Grain Size Distribution Natural Organic Content VOC, Base/neutral and acid extractables

* ** Duplicates, blanks and spikes will be analyzed on a per day basis for on-site GC analyses as outined in Appendix F.
** As outlined in Appendix D SAS requests.
*** Column 5 matrix total does not include matrix spikes and matrix spike duplices.

SUMMARY OF SAMPLING
(Table 2 Continued)

1) Samples are to be considered low concentration

2) Field parameters run by Warzyn sampling personnel. Samples for metal analysis will be filtered prior to preservation.

3) Contract Laboratory Program, RAS, SAS

4) Triple the sample volume will be collected for matrix spike/matrix spike duplicate analysis

5) The matrix spike/matrix spike duplicate samples are at a frequency of one per twenty investigative samples.

6) See Appendix D for requested analysis methods.

7) Sample blank numbers are estimated. Actual numbers may vary based on field conditions.

SGW/jap
[jap-600-30c]

TABLE 3

SAMPLE QUANTITIES, BOTTLES, PRESERVATIVES AND PACKAGING
FOR SOIL, SEDIMENT AND WATER SAMLES FROM WAUSAU NPL SITE

Analysis	Bottles and Jars	Preservation	Holding time	Volume of Samples	Shipping	Normal Packaging
WATER AND LIQUIDS						
Routine Analytical Services (RAS) Low Concentration (Organics)						
Acid Extractables, base/neutral extractables, pesiticides/PCBs	Two 1/2-gallon amber bottles (teflon-lined caps)	Iced to 4°C	5 days until extraction	Fill bottle to neck	Federal Express Priority I	No. 1 foam liner or vermiculite
Low Concentration (Inorganics)						
Metals	One 1-liter high density polyethylene bottle	Filter through 0.45 um filter 1:1 HNO_3 to pH<2 Iced to 4°C	6 months	Fill to shoulder of bottle	Federal Express Priority I	No. 2 foam liner or vermiculite
Cyanide	One 1-liter high density polyethylene bottle	$ 6N NaOH to pH>12 Iced to 4°C	14 days	Fill to shoulder of bottle	Federal Express Priority I	No. 2 foam liner or vermiculate

TABLE 3
(cont.)

Analysis	Bottles and Jars	Preservation	Holding time	Volume of Samples	Shipping	Normal Packaging
Special Analytical Services (SAS)						
Water						
Low Concentration (Organics)						
Volatiles	Two 40-ml volatile organic analysis (VOA vials)	Iced to 4°C	7 days	Fill completely no headspace	Delivered daily to performing laboratory	No. 1 foam liner or vermiculite
Low Concentration (Inorganics)						
TKN, TOC, Nitrate + Nitrite-N NH_3	One 1-liter high density polyethylene bottle	1 ml/l 1:1 H_2SO_4 Iced to 4°C	28 days	Fill to shoulder of bottle	Federal Express Priority I	Vermiculite
Sulfate, Chlorides, Alkalinity	One 1-liter high density polyethylene	Iced to 4°C	28 days (14 days for alkalinity)	Fill to shoulder of bottle	Federal Express Priority I	No. 2 foam liner or vermiculite
Metals	One 1-liter high density ploye thylene	Filtered through 0.45 um filter, 1:1 HNO_3 to pH<2 Iced to 4°C	16 months	Fill to shoulder of bottle	Deliver daily to performing Laboratory	Vermiculite
SOILS AND SOLIDS						
Routine Analytical Services (RAS)						
Low or Med Concentration (Organics)						
Volatiles	Two 120-ml VOA vials	Iced to 4°C	7 days	Fill completely no headspace	Federal Express Priority I (Med w/attached shipper's certificate for restricted articles)	Vermiculite (Med in cans/ vermiculite)

[jap-600-30b]

TABLE 4

SUMMARY OF PROPOSED GROUNDWATER QUALITY MONITORING
WAUSAU WELL FIELD RI/FS

Sampling Event	Total Number of Samples	Sample Blanks	Duplicate Samples	Matrix Spikes	Water Quality Parameters	Analytical Methods	Sample Locations
							West
1. Existing Well	74[a]	6	6	6	VOCs Inorganic Parameters Identified under Subtask 2.1.4	GC (Warzyn) CLP (SAS)	CW6, CW7, CW9, C2S, C4D, R1S, R1D, R2S, R2D, R3S, R3D, R4D, W1A, W4A, W4B, W4C, W6, W7, W9, GM1S, GM4S, GM4D, Plum St. Test Well Total 23
							East
							WW6, MW7, MW7A, MW10A, MW10B, WC1, WC2, WC3, WC3A, WC3B, WC3C, WC5A, WC6, WC6A, FVD2, FVD5, FVD7, Wergin, CW3, CW4, GM5D, GM6D, GM7D, GM8D, GM9S, TCT40, TCT41, TCT42, TCT43, TCT44, WC7, WC7A Total 33
2. Sampling During Drilling	126[a]	10	10	*	VOCs	126 GC (On Site) low detection	Sample Interval 10 foot for west investigation area wells
						20 CLP GC/MS with low detection SAS	Sample interval 15 foot for east investigation area wells
3. Complete Groundwater Sampling Round	158[a]	12	12	12	VOCs	(CLP) GC/MS with low detection SAS	All new and existing monitoring wells
					Inorganic parameters identified under Subtask 2.1.8	CLP (SAS)	
					20 HSL samples	HSL (RAS)	

NOTES

[a] Includes quality control samples, matrix spikes and matrix spike duplicates

* To be determined on site

[jap-600-30k]

TABLE 5

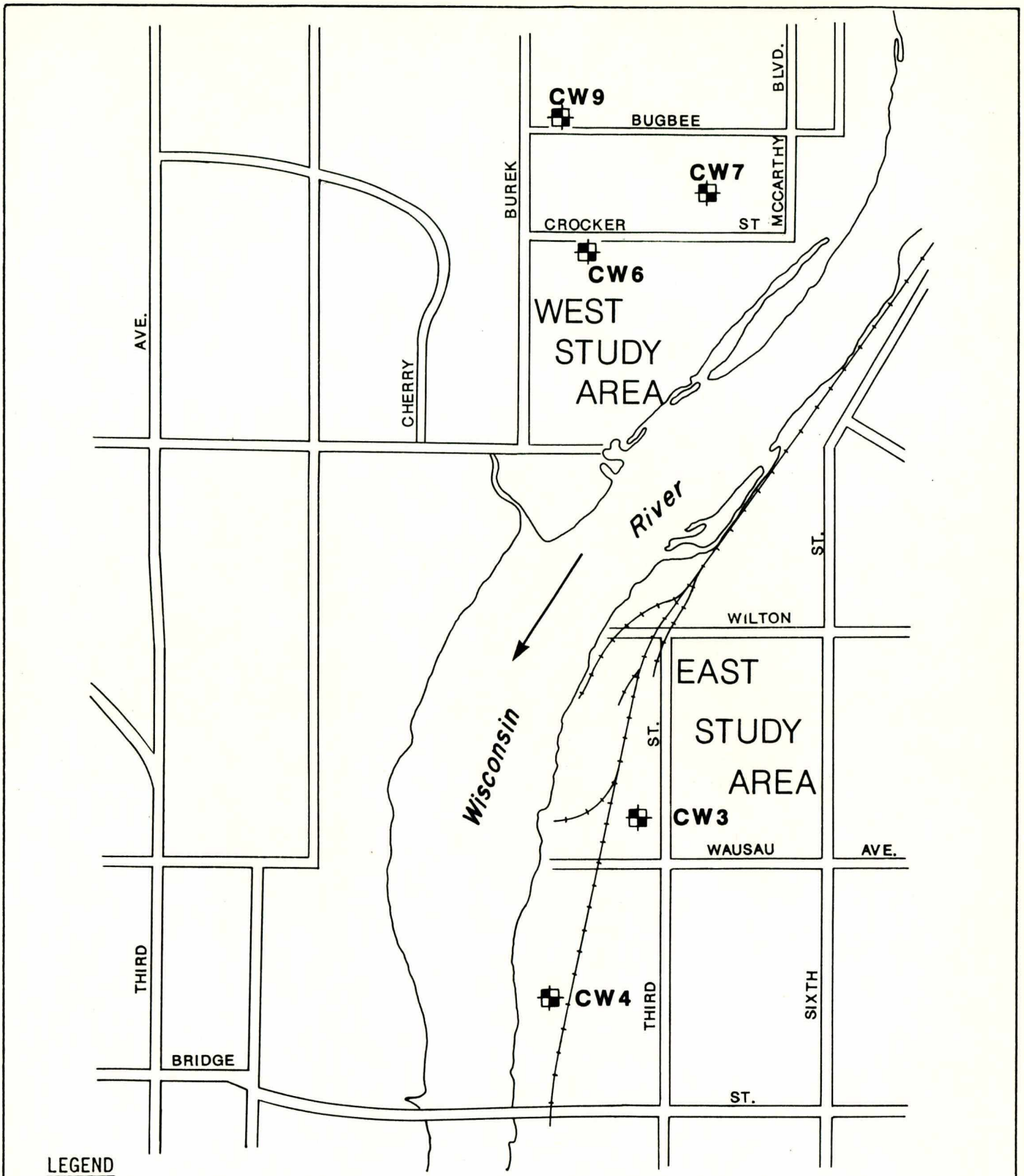
CROSS INDEX OF EXISTING WELL DESIGNATIONS
WAUSAU NPL SITE

Well Identification Code Appearing on Log	Well Identification Code Appearing in This Report	Well Identification Code Appearing on Log	Well Identification Code Appearing In This Report
WEST STUDY AREA			
R1D	Same	WMW-1A	W1A
R2D	Same	WMW-2A	W2A
R3D	Same	WMW-3A	W3A
R4D	Same	WMW-3B	W3B
City Well #6	CW6	WMW-4A	W4A
City Well #7	CW7	WMW-4B	W4B
City Well #9	CW9	WMW-4C	W4C
1S	C1S	WMW-5	W5
B-2S	C2S	WMW-6	W6
B-3S	C3S	WMW-7	W7
B-4S	C4S	WMW-9	W9
4D	C4D	GM-1S	GM1S
B-6D	C6S	GM-2S	GM2S
B-7S	C7S	GM-3S	GM3S
		GM-4S	GM4S
		GM-4D	GM4D
EAST STUDY AREA			
B1	WC1	MW11 (EPA 11)	MW11
B2	WC2	MW12 (EPA 12)	MW12
B3	WC3	MW13 (EPA 13)	MW13
B3A	WC3A	MW14 (EPA 14)	MW14
B3B	WC3B	TCT40	Same
B3C	WC3C	TCT41	Same
B4	WC4	TCT42	Same

TABLE 5
(cont.)

Well Identification Code Appearing on Log	Well Identification Code Appearing in This Report
B4B	WC4B
B5	WC5
B5A	WC5A
B6	WC6
B6A	WC6A
B7	WC7
B7A	WC7A
MW1	WW1
MW2	WW2
MW3	WW3
MW4	WW4
MW5	WW5
MW6	WW6
MW7	WW7
MW7A (EPA 7A)	MW7A
MW8	Same
WGS9	Same
MW9A	Same
WGS10	Same
MW10A (EPA 10A)	MW10A
MW10B (EPA 10B)	MW10B
TCT42	Same
TCT44	Same
FVD1	Same
FVD2	Same
FVD5	Same
FVD7	Same
GM5D	Same
GM6D	Same
GM7D	Same
GM8D	Same
GM9S	Same
WERGIN	Same
CITY WELL #3	CW3
CITY WELL #4	CW4

CSR/sss/
13076.12
[sss-600-54c]



LEGEND

CW6 CITY SUPPLY WELL

north

SCALE: 1" = 1000'

NOTE:

BASE MAP DEVELOPED FROM U.S.G.S. 15 MIN. QUADRANGLE MAPS WAUSAU EAST & WAUSAU WEST DATED 1963, PHOTOREVISED 1978.

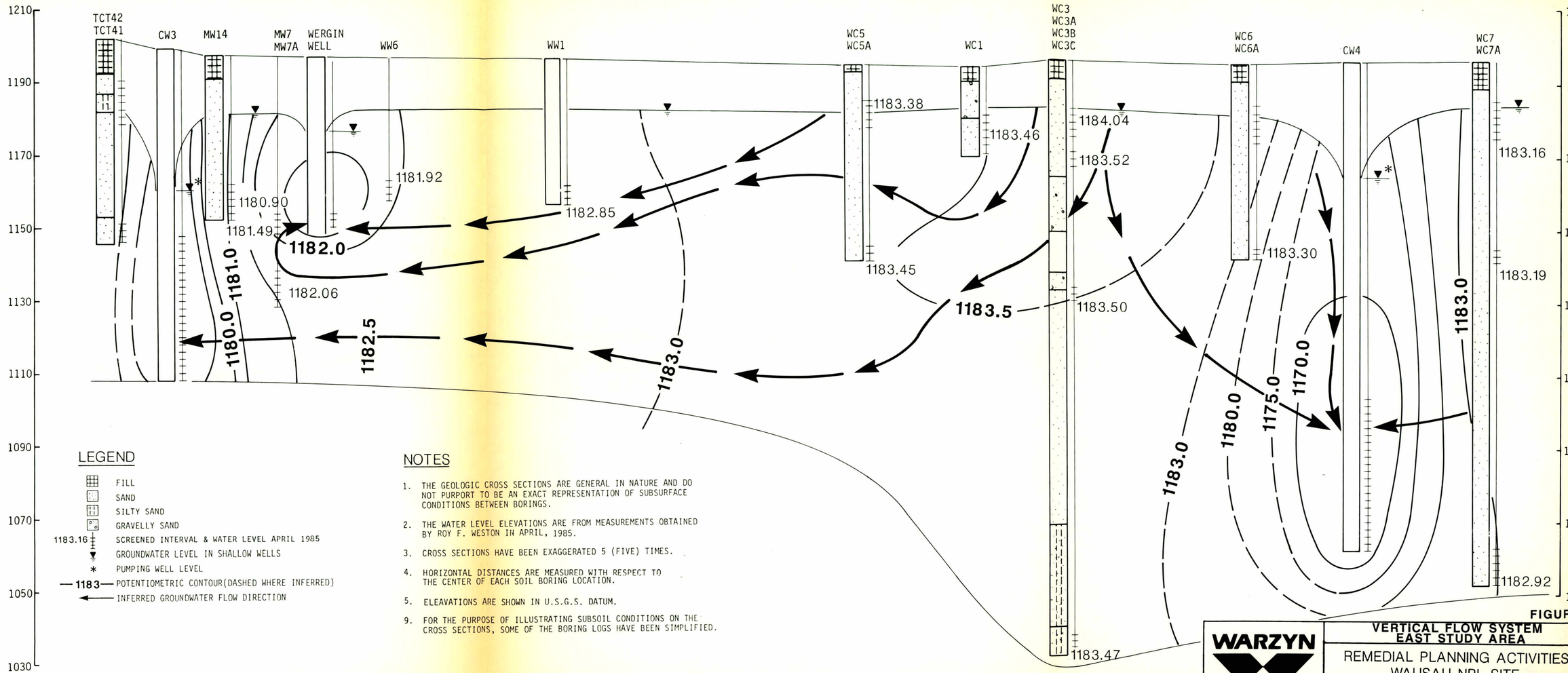
FIGURE 1

DWN SJP	APP'D CSR	DATE 9-2-87	13076-A1

WARZYN ENGINEERING INC

SITE LOCATION MAP
REMEDIAL PLANNING ACTIVITIES
WAUSUA NPL SITE
WAUSAU, WISCONSIN

TELEDYNE POST N67178



LEGEND

- FILL
- SAND
- SILTY SAND
- GRAVELLY SAND
- 1183.16 SCREENED INTERVAL & WATER LEVEL APRIL 1985
- GROUNDWATER LEVEL IN SHALLOW WELLS
- * PUMPING WELL LEVEL
- —1183— POTENTIOMETRIC CONTOUR(DASHED WHERE INFERRED)
- INFERRED GROUNDWATER FLOW DIRECTION

NOTES

1. THE GEOLOGIC CROSS SECTIONS ARE GENERAL IN NATURE AND DO NOT PURPORT TO BE AN EXACT REPRESENTATION OF SUBSURFACE CONDITIONS BETWEEN BORINGS.
2. THE WATER LEVEL ELEVATIONS ARE FROM MEASUREMENTS OBTAINED BY ROY F. WESTON IN APRIL, 1985.
3. CROSS SECTIONS HAVE BEEN EXAGGERATED 5 (FIVE) TIMES.
4. HORIZONTAL DISTANCES ARE MEASURED WITH RESPECT TO THE CENTER OF EACH SOIL BORING LOCATION.
5. ELEAVATIONS ARE SHOWN IN U.S.G.S. DATUM.
9. FOR THE PURPOSE OF ILLUSTRATING SUBSOIL CONDITIONS ON THE CROSS SECTIONS, SOME OF THE BORING LOGS HAVE BEEN SIMPLIFIED.

SCALE: V 1" = 20'
H 1" = 100'

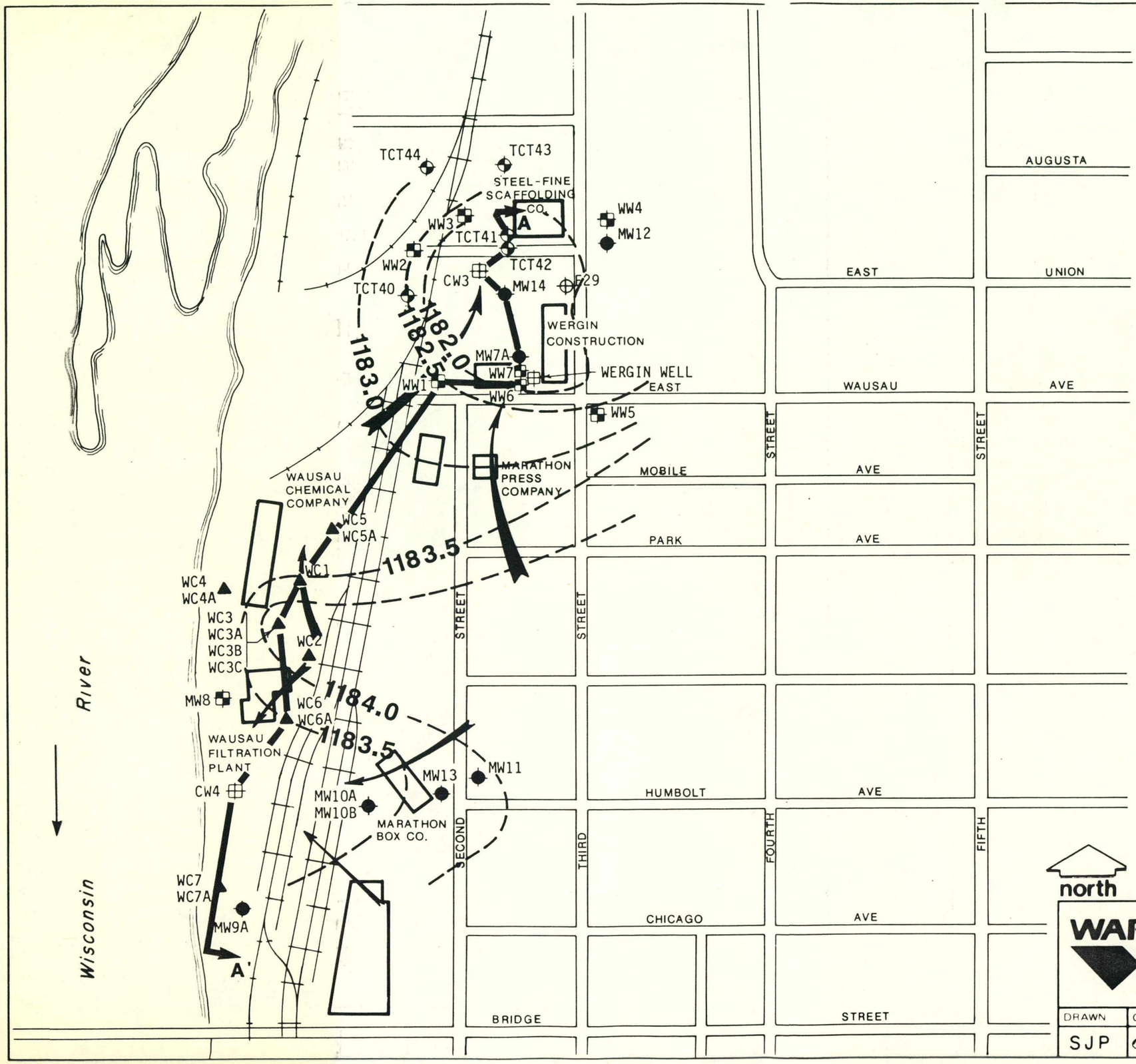
FIGURE 2

WARZYN

VERTICAL FLOW SYSTEM EAST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO.
SJP	CSR	Craig Rawlinson		——	13076 - B1



LEGEND

- CW9 — CITY SUPPLY WELL
- WW2 — CITY MONITORING WELL
- MW12 — WESTON MONITORING WELL FOR EPA
- TCT40 — TWIN CITY TESTING FOR WIS. DNR
- WC1 — WAUSAU CHEMICAL CO. MONITORING WELL (STS CONSULTANTS)
- —1182.0— GROUNDWATER CONTOURS FROM WESTON, 1985
- INFERRED GROUNDWATER FLOW DIRECTION
- A A' CROSS SECTION LOCATION

NOTES

1. WESTON REPORT (1985). BASE MAP REDRAWN BASED ON U.S.G.S. TOPOGRAPHIC MAP, AND TCT REPORT, 1986.
2. REFER TO FIGURE 8 FOR LOCATION OF ADDITIONAL RECENTLY INSTALLED MONITORING WELLS.

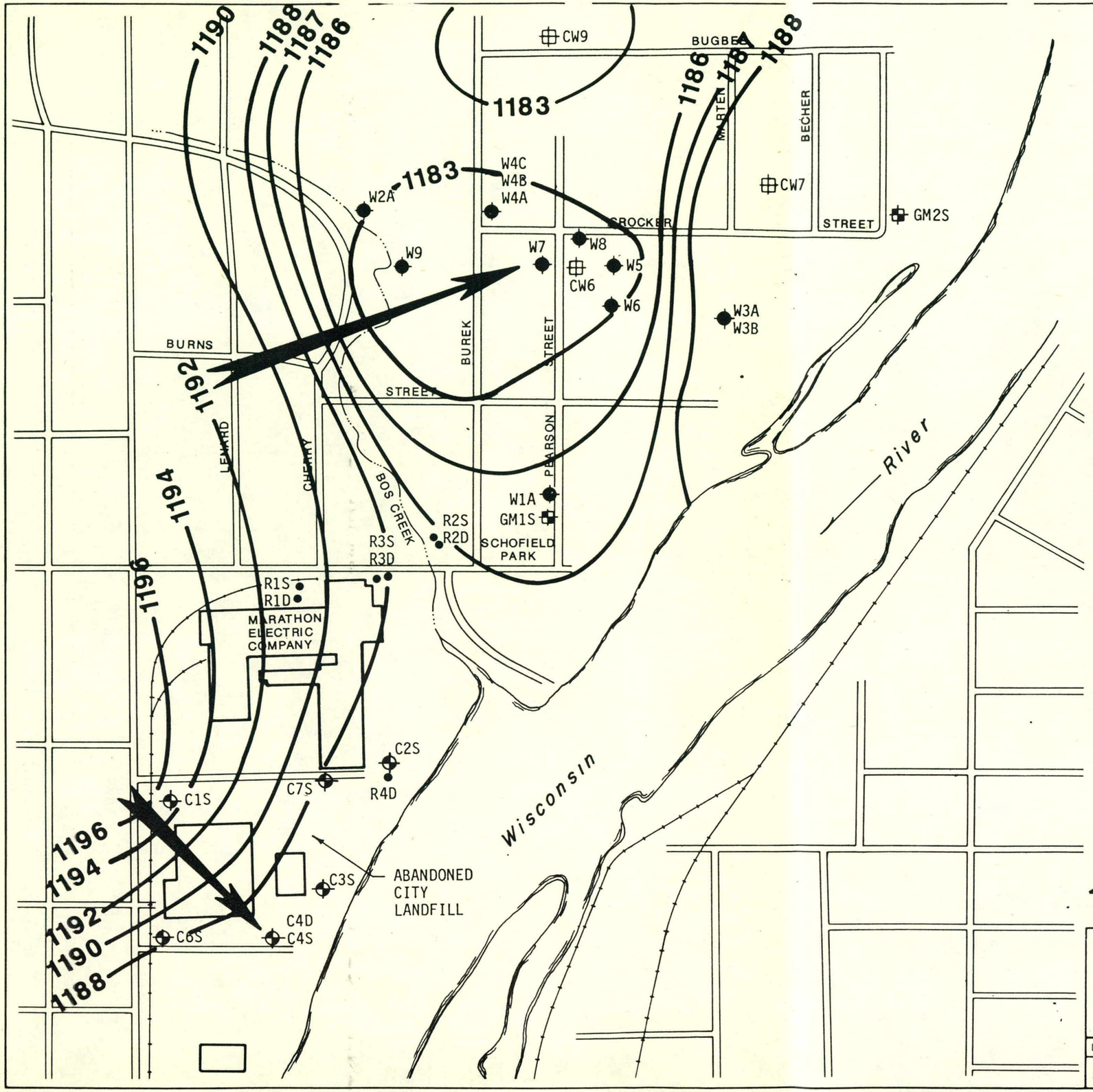
FIGURE 3

WARZYN

WATER TABLE MAP EAST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO
SJP	CSR	Craig Rawlinson		1"=250'	13076-B2



LEGEND

- CITY SUPPLY WELL
- EPA WELL
- DNR WELL
- CITY MONITORING WELL
- MARATHON ELECTRIC WELL
- PROPOSED MONITORING WELL LOCATION "A" DENOTES NESTED WELL
- PROPOSED SOIL BORING LOCATION
- (6) SOIL GAS SAMPLING LOCATION & NUMBER
- —1190— GROUNDWATER CONTOURS
- INFERRED GROUNDWATER FLOW DIRECTION

NOTES

1. WATER TABLE MAP REDRAWN FROM GERAGHTY & MILLER/RMT 1987, FIGURE 7.

north

FIGURE 4

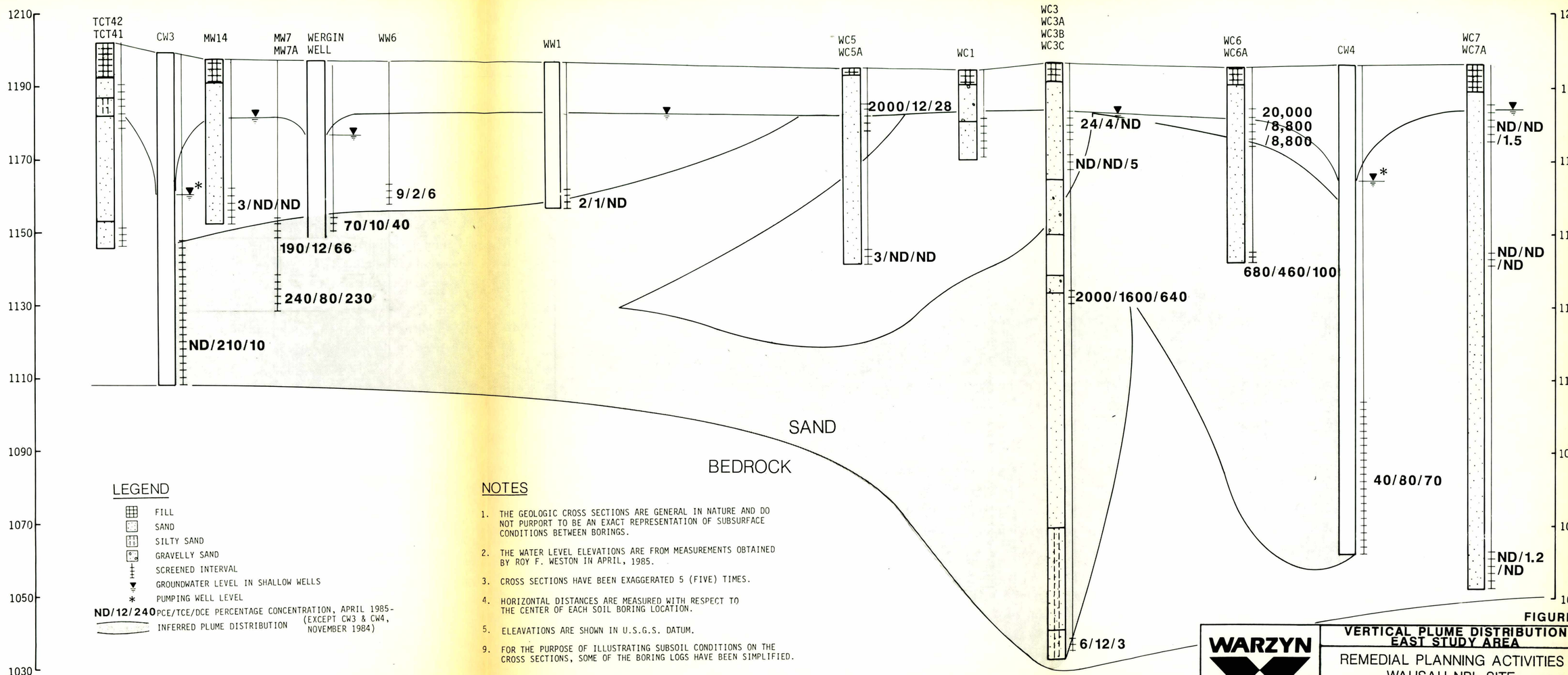
WARZYN

WATER TABLE MAP (5-22-86)
WEST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO.
SJP	CSR	Craig Rawlinson		1" = 400'	13076-B3

TELEDYNE POST N75674



LEGEND

- FILL
- SAND
- SILTY SAND
- GRAVELLY SAND
- SCREENED INTERVAL
- GROUNDWATER LEVEL IN SHALLOW WELLS
- * PUMPING WELL LEVEL

ND/12/240 PCE/TCE/DCE PERCENTAGE CONCENTRATION, APRIL 1985- (EXCEPT CW3 & CW4, NOVEMBER 1984)

INFERRED PLUME DISTRIBUTION

NOTES

1. THE GEOLOGIC CROSS SECTIONS ARE GENERAL IN NATURE AND DO NOT PURPORT TO BE AN EXACT REPRESENTATION OF SUBSURFACE CONDITIONS BETWEEN BORINGS.
2. THE WATER LEVEL ELEVATIONS ARE FROM MEASUREMENTS OBTAINED BY ROY F. WESTON IN APRIL, 1985.
3. CROSS SECTIONS HAVE BEEN EXAGGERATED 5 (FIVE) TIMES.
4. HORIZONTAL DISTANCES ARE MEASURED WITH RESPECT TO THE CENTER OF EACH SOIL BORING LOCATION.
5. ELEAVATIONS ARE SHOWN IN U.S.G.S. DATUM.
9. FOR THE PURPOSE OF ILLUSTRATING SUBSOIL CONDITIONS ON THE CROSS SECTIONS, SOME OF THE BORING LOGS HAVE BEEN SIMPLIFIED.

SCALE: V 1" = 20'
H 1" = 100'

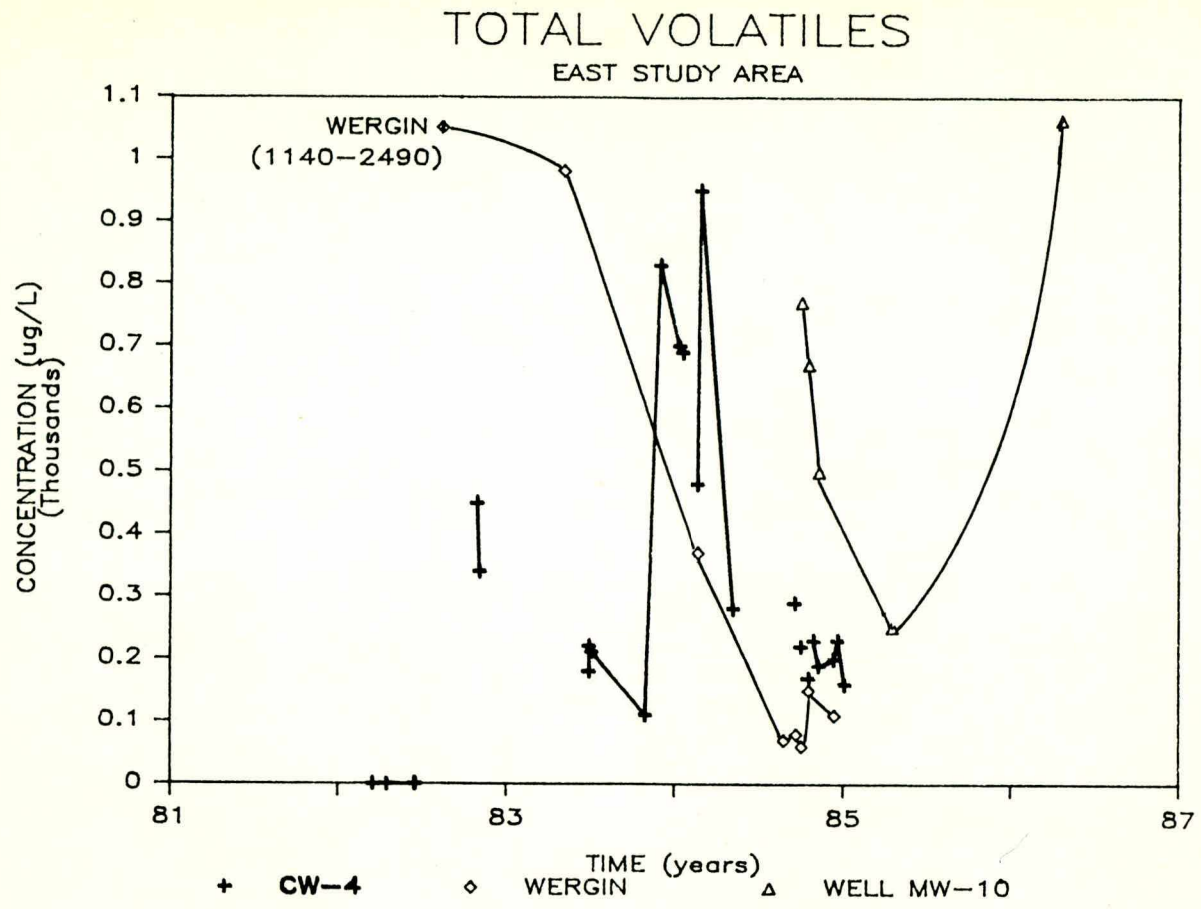
FIGURE 5

WARZYN

VERTICAL PLUME DISTRIBUTION
EAST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO.
SJP	CSR	Craig Rawlinson		—	13076-B4



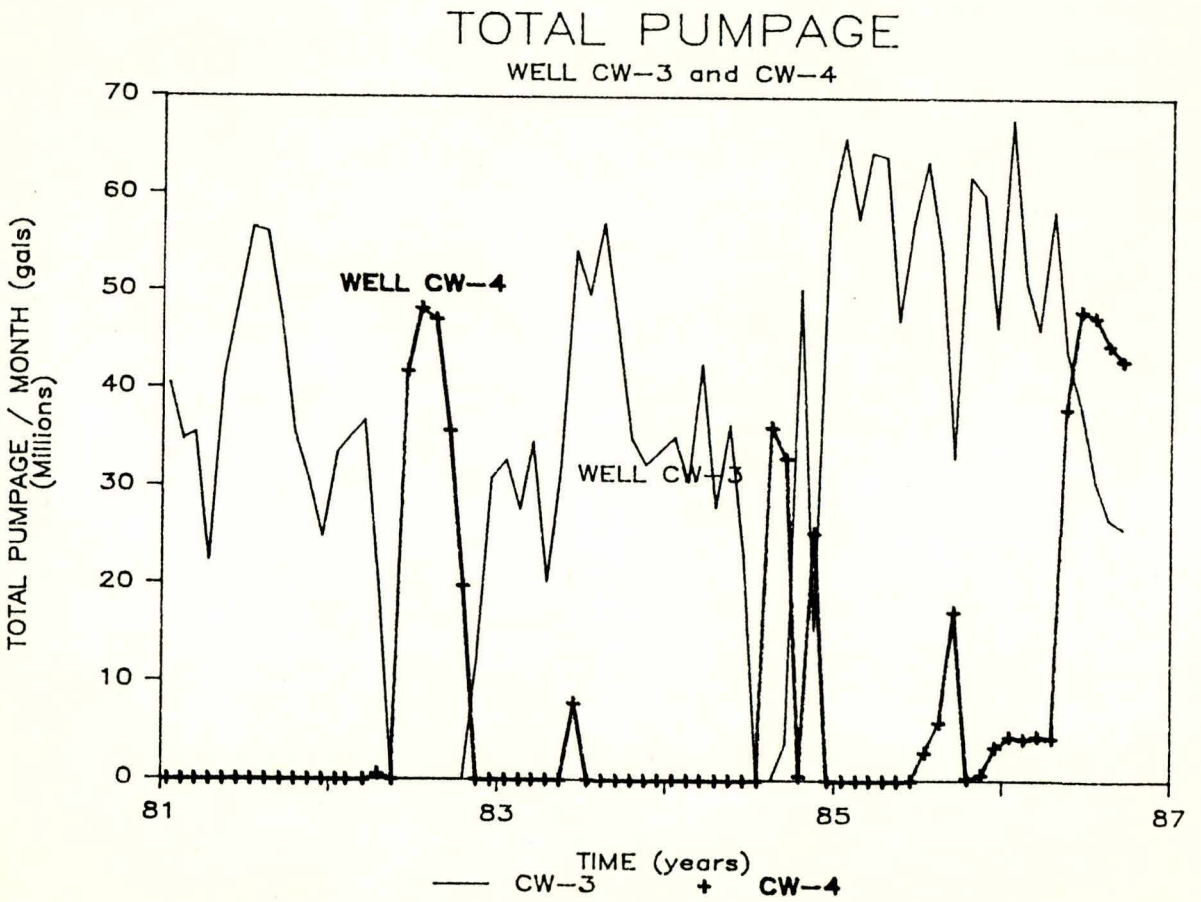
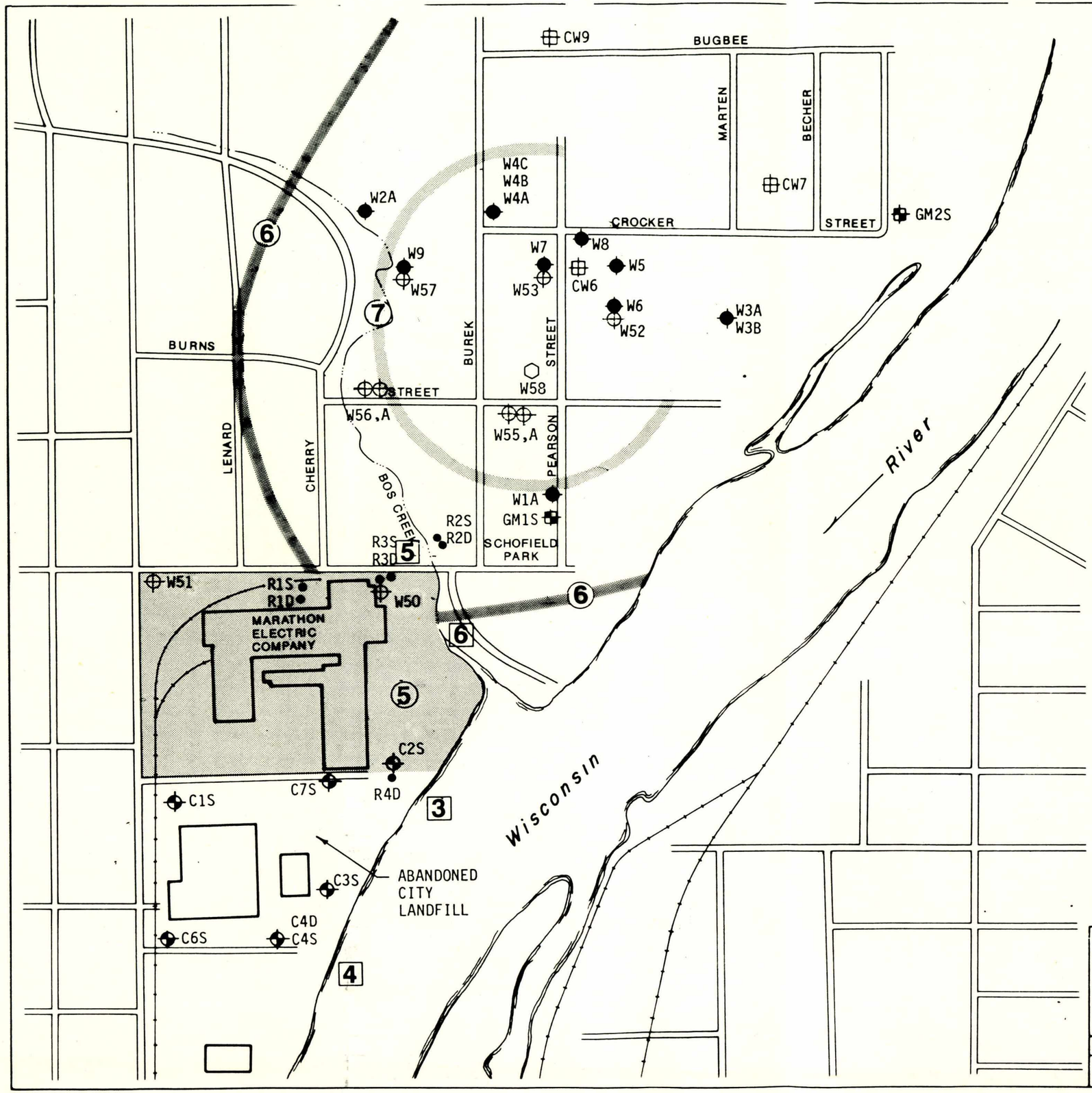


FIGURE 6

PUMPING HISTORY & VOC CONCENTRATIONS



LEGEND

- CITY SUPPLY WELL
- EPA WELL
- DNR WELL
- CITY MONITORING WELL
- MARATHON ELECTRIC WELL
- PROPOSED MONITORING WELL LOCATION "A" DENOTES NESTED WELL
- PROPOSED SOIL BORING LOCATION
- SOIL GAS SAMPLING LOCATION & NUMBER
- PROPOSED SURFACE WATER AND SEDIMENT SAMPLING LOCATION

PROPOSED WELL LOCATION RATIONAL
WAUSAU WELL FIELD - WEST

West Study Area

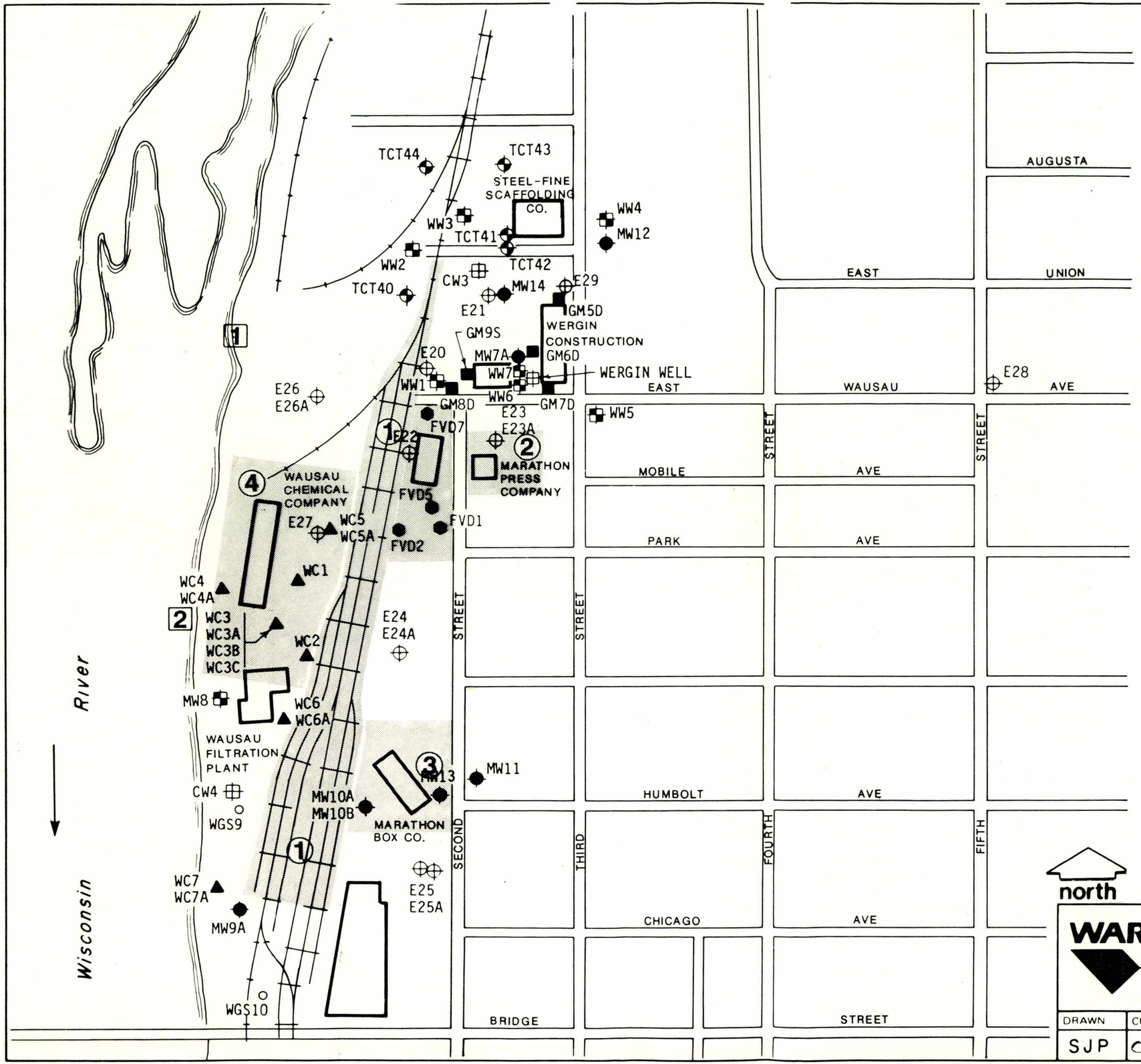
Well No.	Depth (Feet)	Rational
W50	80	Monitor water quality and levels at intermediate depth at well nest R3S/R3D.
W51	45	Monitor levels for better flow control and water quality northwest of Marathon Electric.
W52	80	Monitor water quality and levels at depth adjacent to W6, southeast of CW-6.
W53	80	Monitor water quality and levels at depth adjacent to W7, west of CW-6.
W54 W54A	80 45	Well nest location to be located north of Marathon Electric, exact location based on soil gas and groundwater samples collected during drilling.
W55	80	Monitor groundwater quality and levels between well CW-6 and Marathon Electric.
W55A	45	Monitor groundwater quality and levels between well CW-6 and Marathon Electric.
W56	80	Monitor water quality and levels adjacent to Bos Creek, north of Marathon Electric.
W56A	30	Monitor water quality and levels adjacent to Bos Creek, north of Marathon Electric.
W57	120	Monitor water quality at depth adjacent to W9, confirm existence of bedrock high.
W58	120	Soil boring to confirm the existence of the bedrock high delineated by seismic investigation.
W59 through W65	35	Three wells tentatively planned for Marathon Electric and three wells for one additional source which may be identified through on-site analyses of soil gas and water quality.
Total Projected Footage	1095	375 feet hollow stem auger 820 feet rotary drilled with water quality sampling 17 wells 1 soil borings

FIGURE 7

PROPOSED SAMPLING LOCATIONS
WEST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO
SJP	CSR	Ken Quinn	9/2/87	1" = 400'	13076-B5



LEGEND

- WELL 3 — CITY SUPPLY WELL
- WW2 — CITY MONITORING WELL
- MW12 — WESTON MONITORING WELL FOR EPA
- TCT40 — TWIN CITY TESTING FOR WIS. DNR
- WC1 — WAUSAU CHEMICAL CO. MONITORING WELL (STS CONSULTANTS)
- GM-5D — GERAGHTY & MILLER MONITORING WELL FOR CITY OF WAUSAU
- FVD2 — FOTH & VAN DYKE MONITORING WELL FOR WAUSAU ENERGY
- E27 — PROPOSED MONITORING WELL LOCATION "A" DENOTES NESTED WELL
- WGS9 — WISCONSIN GEOLOGICAL SOCIETY WELL
- 1 — PROPOSED SURFACE WATER AND SEDIMENT SAMPLING LOCATION
- 1 — SOIL GAS SAMPLING LOCATION AND NUMBER

PROPOSED WELL LOCATION RATIONALE
WAUSAU WELL FIELD - EAST

East Study Area

Well No.	Depth	Purpose
E20	60	Monitor water quality at intermediate depth between MW-1 (26') and GM-8D (122.5') to determine if plume is present as inferred from Vertical Contaminant Distribution, Figure 5.
E21	85	Water quality at depth at well MW14
E22	95	Water quality at depth west of Wausau Energy
E23 E23A	85 25	Monitor water levels and quality south of the Wergin Well and north of Marathon Box and Marathon Press Company
E24 E24A	85 25	Monitor water levels and quality between Marathon Press Company and Marathon Box, and upgradient of Wausau Chemical
E25 E25A	120 30	Monitor water levels and quality southeast of Marathon Box property storage building
E26	95 20	Determine extent of possible plume between Wausau Chemical and Well CW-3
E27	110	Monitor water quality below Well WC-5/WC-5A to determine if plume is below this well nest
E28	30	Monitor water table elevation upgradient of pumping wells to establish better control on flow system
E29	30	Monitor shallow groundwater quality adjacent to GM-5D, east of Municipal Well 3.
E30 through E37	25 ft each shallow well(7) 80 ft for deep well (1)	Two shallow wells each on Marathon Box, the railroad yard and Marathon Press Company are dependent on soil gas results. One shallow well is planned for Wausau Chemical depending on review of current on-site work. One additional well nest is dependent on results of on-site analysis of soil gas and water quality.

Total 1150 Feet - 850 feet of rotary drilled with water sampling
300 feet follow stem auger
22 wells at 12 new location

north

FIGURE 8

WARZYN

PROPOSED SAMPLING LOCATIONS EAST STUDY AREA

REMEDIAL PLANNING ACTIVITIES
WAUSAU NPL SITE
WAUSAU, WISCONSIN

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO
SJP	CSR	Ken Quinn	9/2/87	1" = 250'	13076-B6

TELEDYNE POST N75674

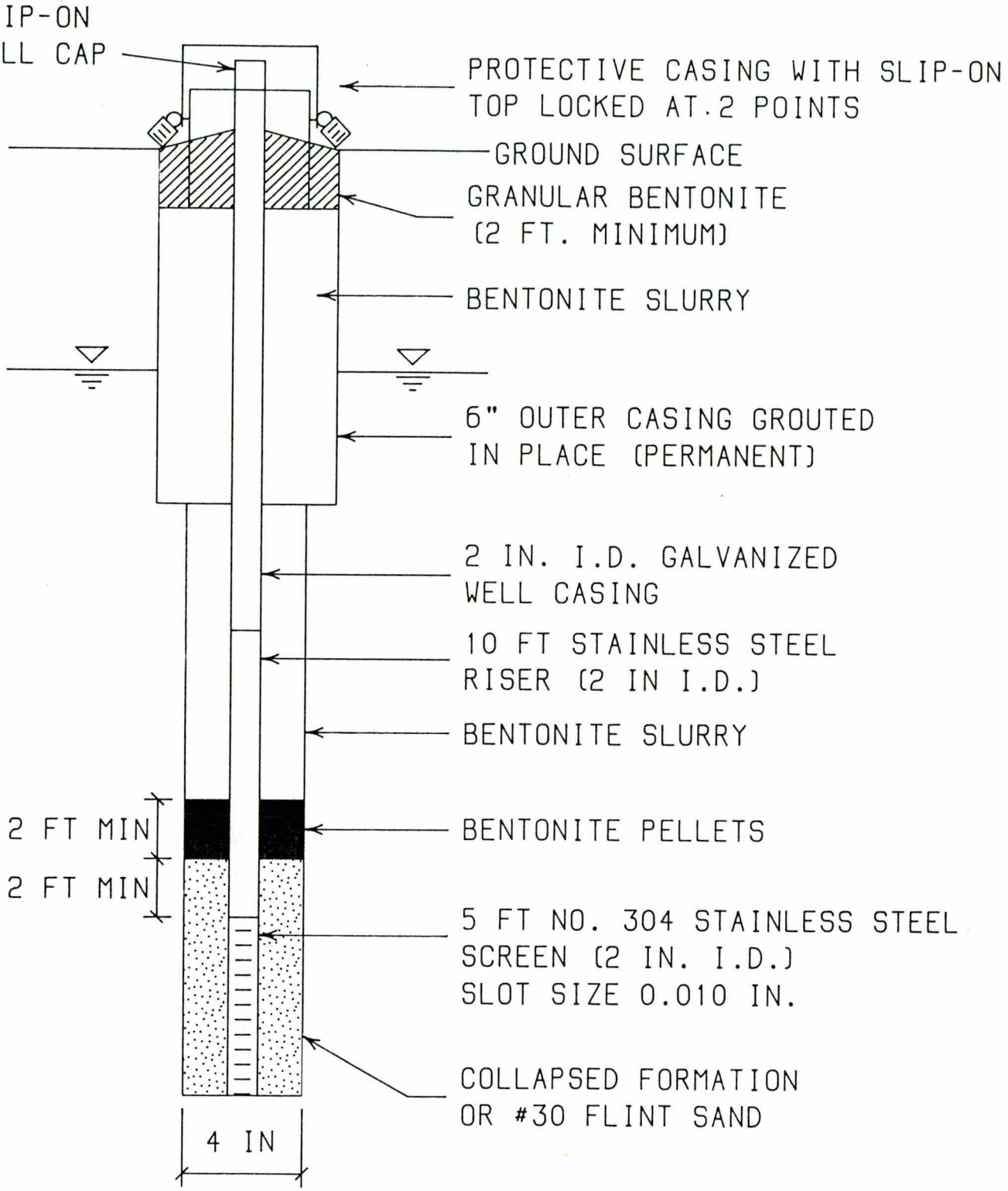


FIGURE 9

DWN ALH	APP'D	DATE 9/2/97	13076-A3	WARZYN ENGINEERING INC	DEEP (PIEZOMETER) WELL REMEDIAL PLANNING ACTIVITIES WAUSAU NPL SITE WAUSAU, WISCONSIN

TELEDYNE POST N67178

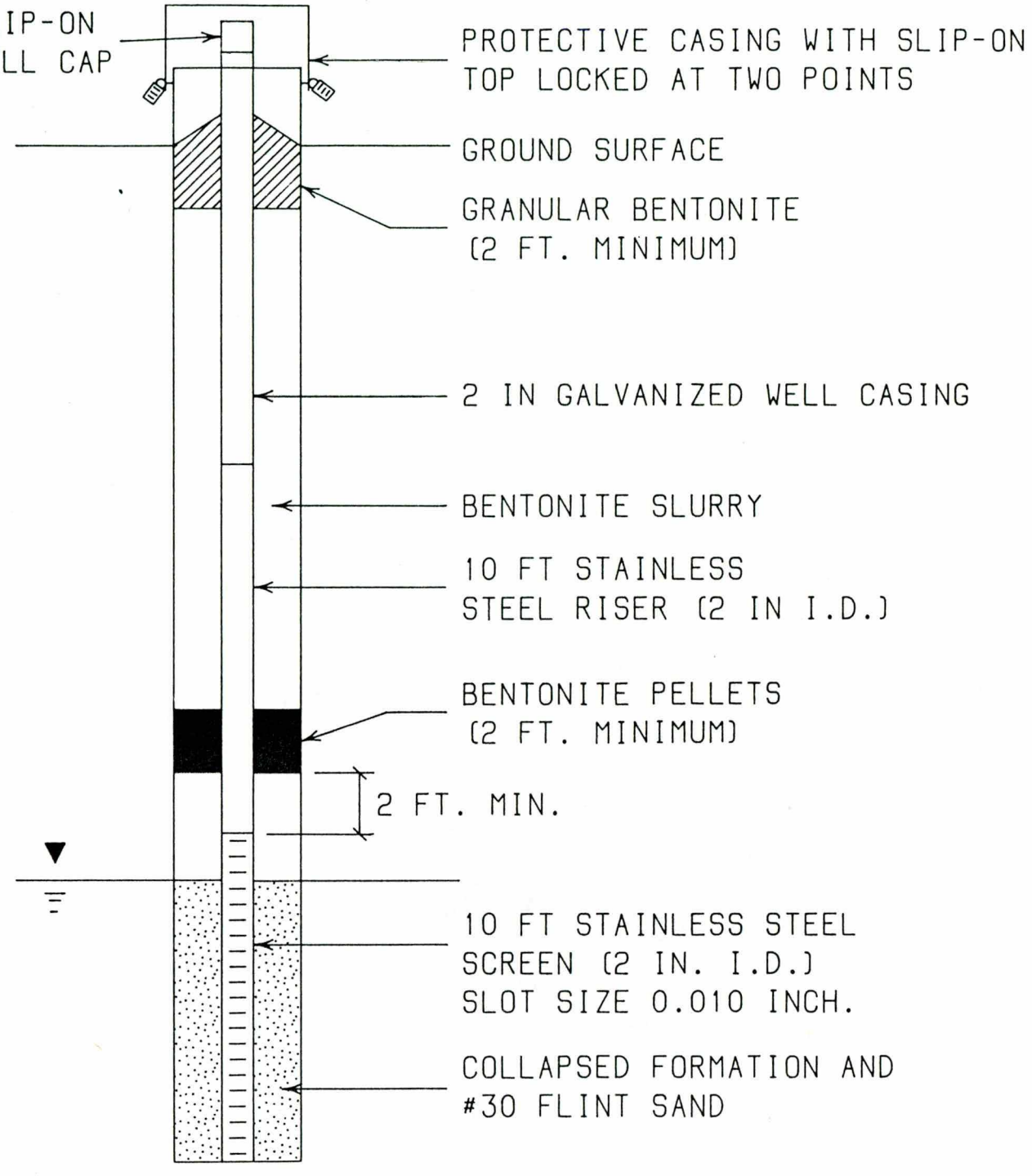


FIGURE 10

DWN ALH	APP'D RJ	DATE 9/2/81	13076-A4	WARZYN ENGINEERING INC	WATER TABLE (SHALLOW) WELL REMEDIAL PLANNING ACITIVIES WAUSAU NPL SITE WAUSAU, WISCONSIN

TELEDYNE POST N67178

PROJECT ORGANIZATION CHART
FIGURE 11

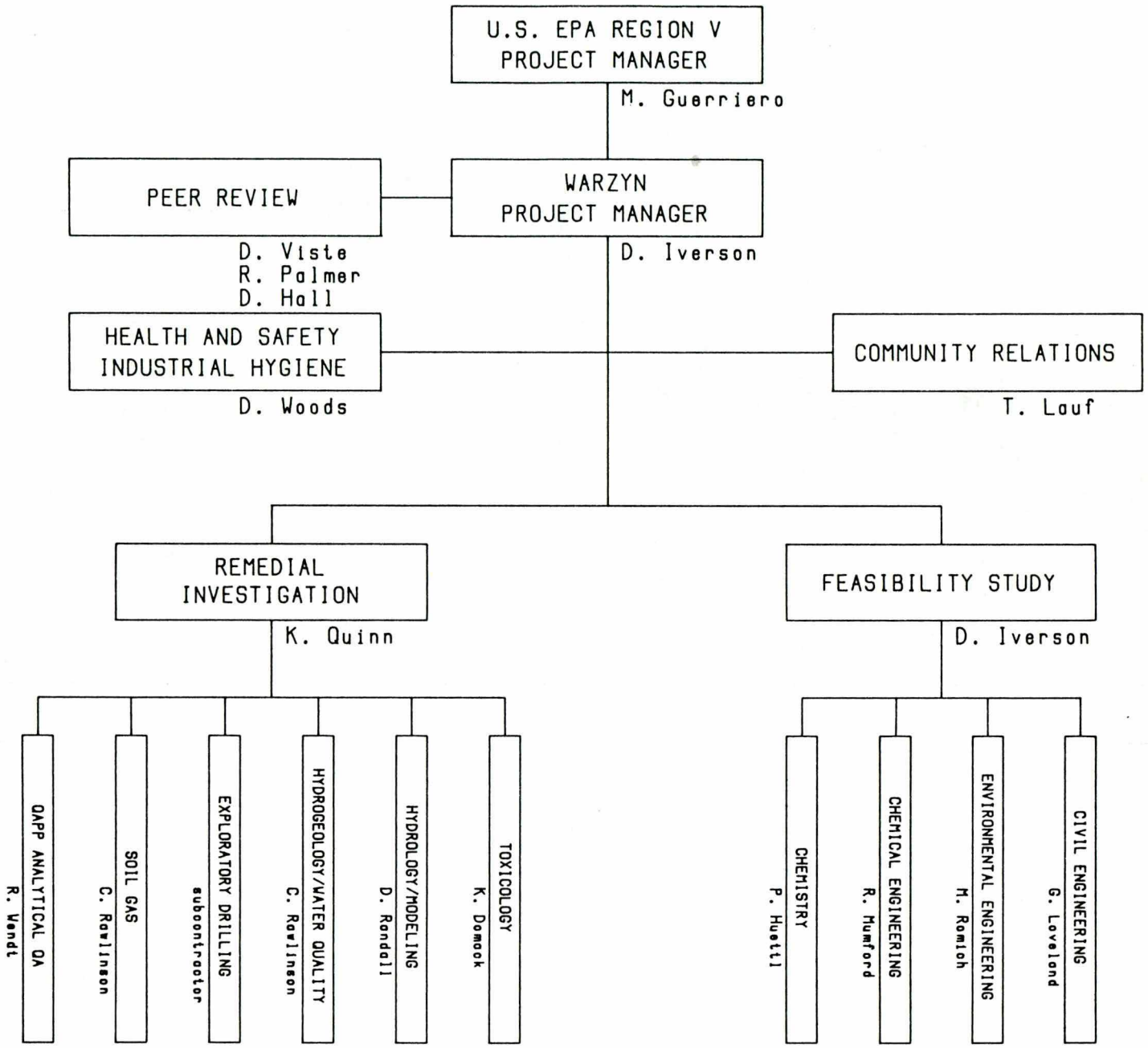
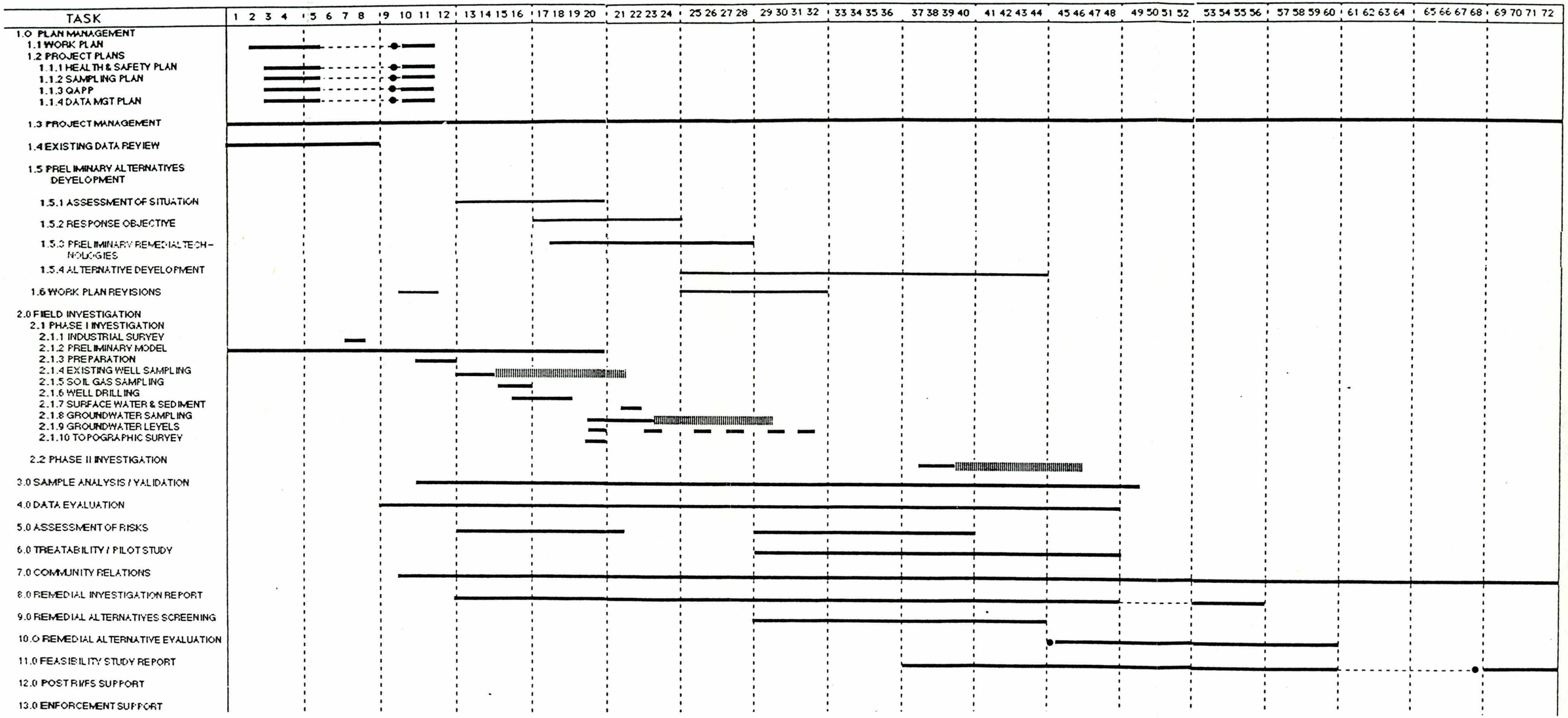


FIGURE 12
PROJECT SCHEDULE
WAUSAU WELL FIELD RI/FS



NOTES
- - EPA REVIEW TIME
▬ PROJECT ACTIVITIES
▥ LAB TURNAROUND TIME
● MEETING

SAMPLING PLAN
WAUSAU NPL SITE
13076.12

1.0 OBJECTIVE

The primary objective of the Phase I sampling activities at the Wausau Well Field is to obtain representative data, which will be used as the basis for the RI/FS analysis. The Remedial Investigation will be performed to gather and assess information needed to accomplish the following general objectives:

- Identification of contaminant sources;
- Characterize on-site physical features and facilities that could affect contaminant migration and remediation;
- Determination of the nature and extent of impacts;
- Determination of probable fate of contaminants through time;
- Assess the dangers to public health associated with the contamination; and
- Support future enforcement action under CERCLA.

2.0 SCOPE

This Sampling Plan details the procedures and practices to be used in obtaining soil, groundwater, surface water, sediment and soil gas samples for the Phase I of the Wausau Well Field RI/FS. These procedures include a description of the sample designation system, the personnel and their responsibilities, and the methods to be used. Also included in this Sampling Plan is a description of anticipated drilling techniques, geophysical logging procedures, hydraulic conductivity testing procedures and water level monitoring practices.

Phase I groundwater sampling is divided into three specific sampling events. The first event consists of sampling and analysis of selected existing monitoring wells in order to provide updated information on the plume distribution since previous sampling. The second sampling event will be

conducted during drilling, and will be used as an investigative tool, to optimize screen location. Third round samples will be obtained from Warzyn installed monitoring wells and from pre-existing monitoring wells. The analytical results of the third round sampling will form the primary basis for the RI analysis, including source identification, extent of contamination, determination of mass of contaminants present, and the evaluation of remedial action alternatives. All three sampling events will be completed at both the east and west study areas (refer to Figure 1). If necessary, an additional round of samples will be collected and analysed during the Phase II investigation.

3.0 SAMPLING LOCATIONS AND NUMBERS OF SAMPLES

Groundwater, surface water, soil, sediment and soil gas samples will be collected at various locations in the City of Wausau. As previously stated, at least three separate groundwater sampling events will be conducted at both the east and west investigation sites. If warranted by results of the Phase I groundwater sampling, an additional round of groundwater samples may be collected from selected wells during Phase II. An addendum to this sampling plan will be submitted if additional sampling or well drilling is deemed necessary during the Phase II investigation. Specific locations and numbers of samples to be obtained during the Phase I investigation are presented in Table 4 (QAPP) and are summarized below. Refer to Figures 7 and 8 (QAPP) for locations of sampling points. The sampling and analysis program is summarized in Table 2 (QAPP).

A. Groundwater Samples

A.1 Existing Well Sampling

Fifty-six (56) monitoring and water supply wells will be sampled, collecting 6 duplicates, 6 field blanks, and 6 matrix spikes, for a total of 74 samples. Specific wells to be sampled are listed in Table 4 (QAPP) and shown on Figures 7 and 8 (QAPP).

Twenty-three (23) water samples will be collected from the following existing well locations west of the Wisconsin River.

- Municipal water supply wells CW6, CW7 and CW9;
- Three (3) shallow and four (4) deep monitoring wells installed for Marathon Electric;
- Two (2) DNR monitoring wells;
- Four (4) City of Wausau monitoring wells; and
- Seven (7) EPA monitoring wells.

Thirty-three (33) groundwater samples will be collected from the following existing well locations east of the Wisconsin River:

- Municipal water supply wells CW3 and CW4;
- Wergin Construction water supply well;
- Thirty (30) monitoring and test wells which were installed during previous hydrogeologic investigations.

A.2 Samples Collected During Drilling

Groundwater samples will be collected during drilling from selected locations through the aquifer to help in identifying the plume distribution prior to setting the well screen. These samples will be analyzed for volatile organic compounds (VOCs) using an on-site GC. Results of the field GC analyses will provide an indication of vertical and horizontal plume distribution, so that the interval to be screened and additional well locations may be logically selected. A total of 106 samples are anticipated. Duplicates, blanks and spikes will be analyzed on a per day basis as outlined in Appendix F. In addition to onsite GC analyses, 26 samples will be submitted to the CLP for low detection limit GC/MS verification of VOC concentrations.

A.3 Groundwater Sampling

Following the installation of site monitoring wells, a comprehensive groundwater sampling round will be conducted at accessible groundwater monitoring and production wells. One hundred and twenty-two (122) groundwater

samples, 12 duplicate, 12 blank and 12 spikes will be collected and analyzed. Forty-seven (47) monitoring wells (28 existing and 19 new) and three (3) production wells will be sampled at the west study area. Sixty-eight (68) monitoring wells (46 existing and 22 new), 2 public and 1 private production well will be sampled at the east study area.

A.4 Additional Groundwater Sampling

The need for an additional round of sampling of the municipal, industrial, and monitoring wells previously installed, in addition to those wells installed during Phase I, will be evaluated. The number of wells sampled during the second round of water sampling may be reduced pending Phase 1 sampling results.

B. Surface and Sediment Samples

Surface water and sediment samples will be collected from six locations on the Wisconsin River and Bos Creek. One duplicate, one blank and one spike sample will also be submitted for analysis. The samples will be used to supplement the data collected during the WDNR survey. Locations of surface water and sediment sampling stations were selected to assess the affect of potential VOC source areas on surface water bodies. Surface water and sediment samples will be collected from the following locations:

- Wisconsin River, near the east bank upstream from Wausau Chemical Co. site treatment system discharge;
- Wisconsin River, near the east bank downstream from Wausau Chemical Co. site treatment system discharge;
- Wisconsin River, near the west bank upstream of the former City of Wausau landfill, Marathon Electric and Bos Creek;
- Wisconsin River, near the west bank downstream of the former City of Wausau landfill;
- Bos Creek in the backwater north of Randolf Street.
- Bos Creek downstream of Marathon Electric.

Refer to Figures 7 and 8 for locations of surface water and sediment sampling stations.

A second set of samples may be collected during the Phase II investigation if laboratory analyses indicate the presence of VOCs. Additional surface water and sediment samples may also be required during Phase II, in order to evaluate the feasibility of developing alternative municipal water supplies.

C. Soil Gas Survey

Soil gas sampling with on-site GC analysis will be conducted to help screen potential source areas in an attempt to map the distribution of VOCs at the water table surface, near a potential sources. Results of onsite analyses will be used to guide the selection of well locations.

Soil gas sampling will be conducted at an estimated 10 facility locations. Figures 7 and 8 (QAPP) show the tentative locations for 7 of the soil gas investigation locations. The remaining 3 are reserved for additional sources which may be identified. Table 1 (QAPP) presents a list of the proposed soil gas survey locations and the tentative number of samples. However, the actual scope of the soil gas survey at each potential source area will be determined based on the size of the facility, the type of operation conducted (past or present) at the site, and the results of the Industrial Site Survey.

A total of 93 soil gas samples are planned to be collected for the purpose of evaluation of potential sources. If additional potential sources are identified during the Industrial Site Survey, an additional 3 samples per new site are planned. Assuming the possibility of locating up to three source areas, a maximum of 102 soil gas samples are planned, plus 10 sample blanks and 10 duplicates.

4.0 SAMPLE DESIGNATION

A sample numbering system will be used to identify each sample, duplicate and blank. Each sample identifier will include the project identifier code, sample type and location code, and a sampling event code. The Team Leader will maintain a log book containing the sample identification listings.

A. Project Identifier Code

A 2-letter designation will be implemented to identify the sampling site. The project identifier will include "W" for Wausau Well Field project followed by "E" for Environmental Protection Agency (EPA) lead project.

B. Sample Type and Location Code

Each sample collected will be identified by a 1- or 2-digit code to identify the sample type. The sample type codes are:

- G - groundwater sample from completed well
- S - split spoon soil sample
- SW - surface water sample
- SD - sediment sample
- SG - soil gas sample
- SB - sample blank
- TB - trip blank
- MS - matrix spike

The location code (QAPP) will follow the sample type code. The location code consists of a two to five digit numberic or alpha-numeric code that indicates the sample location. Surface water, sediment, and soil gas sampling will use a consecutive numbering system, starting at 01. Groundwater location codes are identical to the well labels presented in Figures 7 and 8 (QAPP). A cross reference to well identification labels used in previous reports are presented in Table 5 (QAPP). For water quality samples collected during drilling, the location code will be followed by the sample depth. The depth listed will be the depth of casing installed in the hole during sampling.

Sampling Round Code/Duplicate Code

Groundwater samples obtained from completed wells will have a numeric identifier to signify the sampling round. Groundwater and soil samples collected during drilling, and surface water, sediment and soil gas samples will not have round codes, because they represent a one time sampling at a unique location. Additional soil gas, surface water or sediment samples collected from the same general area will be designated by a different sample location code. Duplicate samples will be designated by the round code, if used, preceded by 9. Sampling round codes are summarized as follows.

Round	Subtask (s)	Sampling Activity
01	2.1.4	• Existing well sampling
02	2.1.8	• Groundwater sampling following installation of new monitoring wells.
03	2.2	• Phase II groundwater sampling, if warranted by previous analyses.

D. Examples of Sample Numbers

Examples of sample number codes are as follows:

- WE-GTCT42-01 = Wausau (EPA Lead), groundwater sample from Monitoring Well TCT 42, sampling Round 1.
- WE-MSTCT42-01 = Wausau (EPA Lead) matrix spike sample from Monitoring Well TCT42, sampling Round 1.
- WE-GE24A-27 = Wausau (EPA Lead) groundwater sample collected while drilling from Monitoring Well E24A with casing to a depth of 27 feet.
- WE-SW02-9 = Wausau (EPA Lead), surface water location 2, duplicate.
- WE-SB10-03 = Wausau (EPA Lead), sample blank number 10, sampling Round 3.
- WE-SG05 = Wausau (EPA Lead), Soil gas sample from location 05.

5.0 GENERAL SAMPLING EQUIPMENT AND PROCEDURES

A. Groundwater Quality Sampling (Subtasks 2.14, 2.18 and 2.2)

1. Objective

The objective of this activity is to collect groundwater quality samples representative of the aquifer at the screened interval of the well.

2. Personnel and Responsibilities

Sampling teams - Two teams of two people each will be responsible for purging wells, collecting water quality samples, providing site safety monitoring during sampling, decontamination of equipment and proper disposal of purged water.

Chain-of-Custody Technician - This person will be responsible for chain-of-custody records, preparing sample bottles for the sampling teams, packaging and shipping samples with assistance from the sampling team members. This person will also assist the analytical technician in sample filtering, preserving and pH and conductivity measurements, particularly in Subtasks 2.1.4 and 2.1.8.

Analytical Technician - This person will be responsible for conducting the on-site volatile organic analyses (Subtasks 2.1.5 and 2.1.6), filtering, preserving, conducting the pH and conductivity measurements and assisting in sample preparation, particularly in Tasks 2.1.4, 2.1.8 and 2.2.

3. Methods - Monitoring Wells

Monitoring well purging and sampling techniques to be utilized during Round 1, 2 and 3 groundwater sampling are summarized below. Sampling techniques to be utilized during drilling are presented in Section C.3 of this chapter.

Water Levels - A water level will be obtained using a weighted tape and sounding device or an electric water level meter, measuring to the nearest $\pm$0.01 ft. If a floating oil layer is suspected to be present, based on drilling or previous sampling observations, an oil water interface probe will be used to measure the depth to fluid and depth to water.

Purging

1. If floating product is observed, a stainless steel bailer will be used to collect a sample of the floating product without purging. The stainless steel bailer will then be used to purge the well of three volumes and to collect required samples (See Table 3 of the QAPP for required bottles, preservatives and handling).

2. At deep wells (piezometers), a Johnson Keck submersible sampling pump with packer will be used to purge and collect the samples.

3. The pump will be set within the screened interval and the packer inflated above the pump within the stainless steel riser section above the screen.

4. The pump will be run to remove a minimum of three volumes of the isolated zone of the well. Volume to be removed is 0.16 gallons/ft of the 2-inch well times the length of the isolated zone.

5. Water levels above the isolated zone will be monitored regularly to determine whether any leakage past the packer is occurring. If more than 10% of the purged volume comes from leakage past the packer (0.1 ft of head drop above the isolated zone per foot of isolated zone), the packer will be deflated and reset. If a second attempt is unsuccessful, the entire volume of the well will be purged.

6. Purge water discharge will be collected in a tank.

7. When the purged water tank is full, it will be discharged to the City sanitary sewer at a point directed by the City of Wausau.

Sample Collection

1. Samples will be collected directly from the sampling pump discharge using the bottles listed in Table 3 of the QAPP.

2. All sample bottles will be labeled with the time of sample collection, in addition to the other chain-of-custody items prepared by the Chain-of-Custody Technician.

3. Samples collected from the bailer (those wells with floating product observed) will be collected with a minimum amount of water disturbance.

QC Samples

QC samples will be collected at the following rate:

- 1 duplicate/10 samples or 1/day, whichever is less
- 1 sample blank/10 samples; sample blanks will be collected by using the sampling device (the pump or bailer) and collecting a sample immediately after decontamination
- 1 Trip blank submitted per sample shipment
- 1 matrix spike/10 samples

Refer to Table 2 (QAPP) for a summary of Quality Control samples to be collected.

Sample Handling Preparation and Sample Analysis

- All samples will be iced immediately after collection
- Groundwater samples undergoing metals analyses will be filtered through a 0.45 um pressure filtration device as soon as possible after sample collection
- Preservation will be conducted as specified in Table 3 of the QAPP
- pH and conductivity will be measured as specified in Appendices C1 through C4 of the QAPP.

Decontamination

- Decontamination will be conducted by washing in TSP solution using City water followed by two rinses with distilled water
- The pump and with discharge tubing will be immersed in the wash water with a minimum of two volumes of water pumped through it, followed by two rinses. The first rinse will have water pumped into the wash tank until the TSP is substantially removed from the pump and discharge hose. The second rinse will follow a similar procedure
- Bailers used to sample oily groundwater will be decontaminated by rinsing with acetone followed by the same wash and rinse sequence.

B. Soil Gas Sampling and Analysis (Subtask 2.1.5)

1. Objective

To collect samples of near-surface soil gas for analysis by the on site GC. Results of the analyses will be used to optimize monitoring well locations, additional soil gas sampling locations and to screen potential VOC source areas.

2. Personnel and Responsibilities

Sampling Team - Two-person sampling team to drive the sampling probe and collect the soil gas sample for analysis.

Laboratory Technician - Will be responsible for GC analysis of the soil gas samples.

3. Methods

Sample Collection

- The steel sampling probe will be driven into the ground to a depth of a minimum of 2.5 ft.
- The drive head will be removed from the probe and the sampling head attached with teflon tubing connected to the sample bottle in line with the pump upstream of the sample bottle. Sample bottle will be a 250 ml bottle with a septum and 2 stop cocks
- The probe and tubing will be purged to remove a minimum of one volume of air from the probe, tubing and sample bottle, while not exceeding 20 centibars of vacuum
- A sample will be collected following purging by closing the stop cocks on the sample bottle. The sample will be immediately wrapped in aluminum foil and put in a dark area. The samples will not be cooled so as not to cause condensation of moisture within the sample bottle.

Decontamination

- The sample probe and tubing will be decontaminated by drawing a minimum of 10 volumes of ambient air through the probe and tubing. If ambient air results in contaminated blanks, decontamination will be completed using the GC carrier gas

- Sample bottles will be decontaminated by flushing a minimum of 10 volumes of high purity air from the GC.

Sample Analysis

Sample analysis and QC will be conducted as described in the QAPP (Appendix F).

C. Well Drilling and Sampling (Subtask 2.1.6)

1. Objective and Scope

The ultimate objective of monitoring well installation is to obtain representative groundwater quality and water level information. This data will be used to characterize groundwater flow conditions and the distribution of the contaminant plume(s).

A total of thirty-nine wells and one soil borings are planned for the Wausau well field investigations. Seventeen wells (975 ft) and one soil borings (120 ft) are anticipated during the Phase I investigation of the west study area. Nine of the proposed well locations and one proposed soil boring locations are shown Figure 7 (QAPP) Also included on Figure 7 (QAPP) is the rationale for proposed well locations. The remaining 8 well locations will be determined in the field, based on the results of the soil gas sampling program, preliminary testing of existing wells, analysis of water samples collected during drilling and geophysical logging of previously installed wells.

An additional twenty-two wells (1150 ft) are anticipated during the Phase I investigation of the east study area. Fourteen of the proposed well locations are shown on Figure 8 (QAPP), along with the rationale for selecting the proposed locations. The remaining eight well locations will be determined in the field, based on sampling of existing wells, soil gas sampling and analysis of samples collected during drilling.

A total of sixty-six 12-hour drilling rig-days (22 field days for three rigs) are anticipated to complete the specified drilling and well installation. In addition to the drilling rig operators and helpers, the field crew will consist of three geologists, one Site Safety Officer (SSO) (between three drilling rigs), an on site GC operator and a Team Leader.

2. Personnel and Responsibilities

Team Leader - The Team Leader will coordinate the three drilling rigs, soil gas sampling and analytical work. The Team Leader will also interpret results of the soil gas and water quality analyses and will make decisions in cooperation with the Warzyn Project Manager and the U.S. EPA project officer on the depth of screened interval and location of future drilling sites.

Site Geologist/Geotechnical Engineer - A geologist or geotechnical engineer will be assigned to each individual drilling rig working on the site to collect and classify soil samples, collect water quality samples, prepare boring logs and well details, document the methods used for well construction and development, and provide site safety monitoring of drilling operations in Level D protection.

Site Safety Officer - The Site Safety Officer will be responsible for coordinating site safety activities on each of the three drilling rigs and other concurrent operations (soil gas sampling) and providing assistance on any sampling activity where Level B or C work would be on-going. The Site Safety Officer will also advise the Team Leader as to the sequencing of activities to avoid the need for more than one activity taking place in Level B or C protection.

3. Methods

Drilling - Shallow borings will be drilled using 4 1/4-in. ID hollow-stem augers with a screened lead auger. If a water head is needed to eliminate sand moving up into the augers, City water can be added to the augers. If City water is used, a daily VOC analysis of the City water will be completed using the on-site GC.

It is anticipated that deep borings (>65 feet) will be drilled using clear water rotary methods. A daily VOC analysis of the water supply will be conducted using the on site GC. However, if clear water rotary drilling becomes infeasible, drilling muds may be used. Initially, 6 in. casing will be advanced into the borehole. If necessary, 4 in. casing will be telescoped within the 6 in. casing and advanced to the total depth of the boring.

Soil Sampling and Analysis - Soil sampling will be conducted according to ASTM D1586 using a 2-in. split-spoon sampler. Samples will be collected at a 5-ft interval to a depth of 25 ft, and 10-ft intervals to the bottom of the borehole or at changes in soil type. Thirty (30) soil samples will be submitted to the CLP for analysis of grain size distribution and 15 for natural organic content using the Wet Combustion method (see Appendix D for SAS). In addition, thirty (30) soil samples will be submitted to the CLP for analysis of VOCs, base/neutral and acid extractables by GC/MS to characterize contamination in the unsaturated zone at potential source areas.

Water Quality Sampling and Analysis

Water quality samples will be collected during drilling from the 40 wells which will be installed during Phase I (Refer to Figures 7 and 8). Deep borings performed in the west study area will be sampled on an approximate 10 ft interval from a depth of about 40 ft to termination (±80 ft). An average of five samples per deep well and one sample from each of the individual shallow wells are anticipated to be collected. Approximately 48 groundwater samples will be collected and analyzed during the installation of monitoring wells for the west study area.

Due to the greater proposed depth of several monitoring wells on the east side of the Wisconsin River, groundwater quality will be sampled on a 15 ft interval from a depth of approximately 30 ft to boring termination. Approximately 58 groundwater samples are anticipated to be collected during installation of monitoring wells for the east study area. A total of approximately 106 samples will be collected during drilling.

A duplicate and blank sample will be collected and analyzed for every 10 groundwater samples or per day, whichever is less. City water used for drilling will also be analyzed daily. The on-site analytical procedures are presented in Appendix F and summarized in Table 2.

The borehole sampling zone will be isolated by either driving a 2-inch, 3-ft long schedule 80 galvanized steel well point into the sand ahead of the casing or by setting a Johnson Keck submersible pump with packer within the bottom of the casing and sampling from the riser pipe. If the well point is used, a solid rod may have to be driven first to loosen the soils. The riser pipe will be purged of three well volumes using a Brainard Kilman 1.7 inch OD. pump. Four VOA vials will be collected, two for on-site GC analysis and two for possible lab analysis. All samples will be collected with no head space. Samples will be analyzed on-site according to methods in Appendix F of the QAPP.

Geophysical Logging - Geophysical logging will be conducted in the deep borehole after completion of the boring. A natural gamma log will be run by the site geologist using a constant logging speed of not more than 1/2 crank/second on the Mount Sopris 1000 C logging unit on the 4:1 drive shaft (less than 0.25 ft/second). The geophysical logger will be calibrated as described in Appendix E of the QAPP.

Well Installation - The wells will be installed as shown on Figures 9 and 10 of the QAPP. All joints will be sealed with teflon tape. The site geologist will measure total depth of the hole prior to installation of the well materials and will measure the depth to the top of each material placed prior to placing the next layer.

The bentonite slurry will be placed using a tremie pipe, maintaining the tremie pipe below the top of the bentonite slurry.

Wells located in high traffic areas (i.e., parking lots, driveways) will be constructed so that protective casings are flush with the ground surface.

Development - Each well will be developed using either air lift pumping or pumping with the Brainard Kilman pump until the water is visually clear, on-site pH and conductivity measurements have stabilized or until 10 well volumes (0.16 gal/ft of water column in the well) have been removed.

Decontamination - The split spoon sampling device will be decontaminated using a TSP wash followed by two distilled water rinses between each sample.

The drill rig tools will be steam cleaned immediately after each boring, except where a shallow boring is conducted with the same rig immediately after completion of an adjacent deep well. Steam cleaning will be conducted at the decontamination pad.

The Brainard Kilman pump will be decontaminated using a TSP wash and clear water rinse or steam cleaned following each water quality sample. If water quality results from the on site lab indicate compounds of concern are below detection prior to the next sample, no decontamination will be necessary.

Waste Disposal - Cuttings from the drilling operation will be contained if they are found to be contaminated by screening with a PID or OVA. If on-site disposal of cuttings create aesthetic problems, they will be removed and disposed of off-site. The off-site disposal area will be coordinated between the City, EPA project officer and the Team Leader.

Drilling fluids, purge water from water quality sampling and well development water will be contained and disposed of to the City sewer at a location agreed upon by the City.

D. SURFACE WATER AND SEDIMENT SAMPLING

1. Objective

Surface water and sediment samples from six (6) different locations will be collected and submitted to the CLP for VOC and general water quality parameter analyses. The results of these analyses will be used to determine if

potentially identified sources have had an impact on VOC concentrations of surface water and sediment. Inorganic parameter analyses of surface water samples will be used to evaluate future potential alternative water supplies and to compare groundwater and surface water chemical characteristics. GC/MS Analyses of sediment samples will be used to evaluate VOC impact on surface water bodies in the area.

2. Personnel and Responsibilities

Sampling Team - One team of two people will collect surface water and sediment samples and provide their own site safety monitoring.

Chain-of-Custody Technician - This person will prepare sample labels and provide chain-of-custody records and package and ship samples.

3. Methods

Surface Water Sampling - Samples will be collected in quiet water areas near the bank of the stream or river. Surface water samples will be collected prior to sediment samples at 6 locations shown on Figures 7 and 8.

Surface water samples will be collected using stainless steel sampling equipment. Sampling equipment will be decontaminated using TSP wash and double clear water rinse. Surface water samples undergoing metals analyses will be filtered through a 0.45 um pressure filtration device as soon as possible after sample collection. Sample handling, preservation, bottles and packaging are summarized in Table 3 of the QAPP.

Surface water samples will be analyzed for pH and conductivity using the methods in Appendices C1 through C4 of the QAPP.

Sediment Sampling - Sediment samples will be collected following surface water samples using a hand-corer. The hand-corer will be driven to a depth of 6 inches sediment sample splits of the material will be collected in the sample bottles listed in Table 3 of the QAPP. The VOA sample will be collected as soon as possible after sample removal. The hand-corer will be decontaminated in the same manner as the surface water sampling equipment.

QC Samples

- 1 duplicate of each media
- 1 sample blank of each media collected by using the sampling device immediately after decontamination. Silica sand will be used for the sediment sample blank
- 1 matrix spike for each media

E. HYDRAULIC CONDUCTIVITY TESTS

1. Objective

The hydraulic conductivity tests are intended to obtain an estimate of the hydraulic conductivity of the screened interval of the well tested.

2. Personnel and Responsibilities

The site geologist will conduct the test.

3. Methods

The test methods will follow the procedures outlined in Appendix G of the QAPP, and will include the following steps:

- Hydraulic conductivity tests will not be conducted at wells with total VOC concentrations greater than 750 ug/L
- Measure water level with a tape and sounding device.
- Place the pressure transducer into the well and allow approximately three minutes for the probe to equilibrate to the water temperature and pressure
- Install the well head device to seal the well head (for piezometers only)
- Enter the reference water level into the data logger and check the water level using the pressure transducer to water level reading is stable
- After a stabilized water level reading is obtained from the pressure transducer, the well is pressurized with sufficient air pressure to displace 10 ft of water (0.4 PSI/ft of water) (for piezometers only)

- Air pressure is maintained until the water level reading from the transducer is constant (for piezometers only)
- The air pressure is then be instantaneously released while running the pressure transducer recorder in the log sampling mode (for piezometers only)
- At water table wells a single bailer is removed to reduce the water level, while running the pressure transducer recorder in the log sampling mode
- The test results are immediately printed out to obtain a hard copy
- Data is transferred at the end of the day to a micro computer.

SAMPLE DOCUMENTATION

Samples will be handled under chain-of-custody procedures. Standard forms including sample tags, traffic reports, chain-of-custody forms, and custody seals used for sample tracking will be maintained. Pertinent information regarding the samples will be recorded in the site log book maintained by the Team Leader and in logs maintained by each sampling crew. The information will include sampling time, location, tag numbers, designation and samplers. Pertinent PID readings, weather conditions and field modifications of sampling strategy will be recorded. The log book will be maintained in indelible ink and will be in a bound volume unless weather conditions dictate otherwise.

CLP Sample Documentation Required by the U.S. EPA. The following documents are numbered and must be accounted for. If a document is voided, it will not be destroyed, rather, it will be saved and returned to the REM II Sample Coordinator. Copies of the multiple-copy forms will accompany samples to the laboratory. The other copies will be sent to the Sampling Coordinator immediately following sample shipment.

A) Chain of Custody Form

1) One Form per shipping container (cooler).

2) Carrier service does not need to sign form if custody seals remain intact.

3) Use for each project sample.

B) Chain of Custody Seals

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

2) Cover seals with clear tape.

3) Record seal numbers on Chain of Custody Form.

4) Use for each project sample.

C) Organic and Inorganic Traffic Reports

1) For low and medium samples, one form required for each sample undergoing RAS organic or inorganic analysis by CLP.

2) Preprinted stickers on forms should be fixed to appropriate sample containers.

3) These numbers are recorded on Chain of Custody Forms.

D) Sample Tags

1) Each sample container will have a Sample Tag affixed to it with string or wire.

2) Traffic Report Numbers and Case numbers are recorded in the "Remarks" section of the tag.

3) Sample Tag Numbers are recorded on the Chain of Custody Forms.

4) Use for each project sample.

E) High Hazard Traffic Reports

1) One form is required for each High Concentration sample being analyzed by the CLP.

2) The stickers provided are to be affixed to the appropriate sample when packaging.

Other Sample Documentation Required

A) Special Analytical Service (SAS) Packing List

1) Used in place of Traffic Report Forms for SAS requests.

2) Does not replace Chain of Custody Form - used in conjunction with it.

B) CRL Basic Data Forms

1) For samples sent to CRL, these replace Traffic Report Forms and are shipped with samples.

C) CRL Sample Data Report

1) Will be completed for all CLP samples.

2) For samples sent to CLP Laboratories, these forms are sent to Sampling Coordinator to be forwarded to the RSCC.

3) These forms are necessary for the U.S. EPA to track the samples for data validation purposes.

D) Sample Identification Record Form

1) Will provide a means of recording crucial sample shipping and tracking information.

2) Will contain information to be entered into the Remtech DataBase such as:

a) Case Number
b) CRL Number
c) Sample Matrix
d) Site Number
e) Sample Location Code
f) Sample Round
g) Sample Type (blank, replicate)
h) Number of bottles
i) Traffic Report Numbers
j) Chain of Custody Number
k) Lab Code
l) Date Sampled
m) Date shipped
n) Airbill Number
o) Sample Tag Number

3) This form must be maintained for each sample shipment and forwarded to the Sampling Coordinator upon sample shipment.

Paperwork accompanying the samples being shipped to CRL and CLP Laboratories will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the chain-of-custody forms and other paperwork (if possible), will be retained for the field files.

Two sample seals will be placed on opposite sides of the lid and extending down the sides of the cooler. The lid will be securely taped shut prior to shipment.

Schedule of Sampling Events

The Phase I investigation sampling will begin with existing well sampling approximately one week after receipt of approval of the QAPP and associated plans. Existing well sampling will take approximately 2 weeks and will be followed immediately by the soil gas investigation. It is anticipated that the soil gas investigation will take approximately two weeks to complete. Monitoring well installation is tentatively scheduled to occur simultaneously with the second week of the soil gas investigation. However, the well installation schedule is subject to the availability of a drilling subcontractor chosen through an open bid procedure. Approximately twenty-two drilling days for three rigs are anticipated. Following well installation, monitoring wells will be developed and surveyed for location and elevation. Surface water, sediment and groundwater sampling of all monitoring wells and production wells will be initiated approximately two weeks after monitoring well installation. Finally, groundwater levels will be recorded at six different time periods following monitoring well installation. The schedule of sampling activities is summarized in Figure 12.

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[jap-600-30j]

APPENDIX B

EPA CLP TARGET COMPOUND LIST - RAS
ORGANIC AND INORGANICS

WARZYN

TABLE A-1

CLP TARGET COMPOUND LIST AND CONTRACT REQUIRED DETECTION LIMITS (CRDL)*

	Volatiles	CAS Number	Detection Limits(1) Low Water(2) ug/l	Low Soil Sediment(3) ug/kg
1.	Chloromethane	74-87-3	10	10
2.	Bromomethane	74-83-9	10	10
3.	Vinyl Chloride	75-01-4	10	10
4.	Chloroethane	75-00-3	10	10
5.	Methylene Chloride	75-09-2	5	5
6.	Acetone	67-64-1	10	10
7.	Carbon Disulfide	75-15-0	5	5
8.	1,1-Dichloroethene	75-35-4	5	5
9.	1,1-Dichloroethane	75-35-3	5	5
10.	trans-1,2-Dichloroethene	156-60-5	5	5
11.	Chloroform	67-66-3	5	5
12.	1,2-Dichloroethane	107-06-2	5	5
13.	2-Butanone	78-93-3	10	10
14.	1,1,1-Trichloroethane	71-55-6	5	5
15.	Carbon Tetrachloride	56-23-5	5	5
16.	Vinyl Acetate	108-05-4	10	10
17.	Bromodichloromethane	75-27-4	5	5
18.	1,1,2,2-Tetrachloroethane	79-34-5	5	5
19.	1,2-Dichloropropane	78-87-5	5	5
20.	trans-1,3-Dichloropropene	10061-02-6	5	5
21.	Trichloroethene	79-01-6	5	5
22.	Dibromocloromethane	124-48-1	5	5
23.	1,1,2-Trichloroethane	79-00-5	5	5
24.	Benzene	71-43-2	5	5
25.	cis-1,3-Dichloropropene	10061-01-5	5	5
26.	2-Chloroethyl Vinyl Ether	110-75-8	10	10
27.	Bromoform	75-25-2	5	5
28.	2-Hexanone	591-78-6	10	10
29.	4-Methyl-2-pentanone	108-10-1	10	10
30.	Tetrachloroethene	127-18-4	5	5
31.	Toluene	108-88-3	5	5
32.	Chlorobenzene	108-90-7	5	5
33.	Ethyl Benzene	100-41-4	5	5
34.	Styrene	100-41-4	5	5
35.	Total Xylenes	100-42-5	5	5

TABLE A-1

HAZARDOUS SUBSTANCE LIST (HSL) AND
CONTRACT REQUIRED DETECTION LIMITS (CRDL)*

	Volatiles	CAS Number	Detection Limits(1) Low Water(2) ug/l	Low Soil Sediment(3) ug/kg
1.	Chloromethane	74-87-3	10	10
2.	Bromomethane	74-83-9	10	10
3.	Vinyl Chloride	75-01-4	10	10
4.	Chloroethane	75-00-3	10	10
5.	Methylene Chloride	75-09-2	5	5
6.	Acetone	67-64-1	10	10
7.	Carbon Disulfide	75-15-0	5	5
8.	1,1-Dichloroethene	75-35-4	5	5
9.	1,1-Dichloroethane	75-35-3	5	5
10.	trans-1,2-Dichloroethene	156-60-5	5	5
11.	Chloroform	67-66-3	5	5
12.	1,2-Dichloroethane	107-06-2	5	5
13.	2-Butanone	78-93-3	10	10
14.	1,1,1-Trichloroethane	71-55-6	5	5
15.	Carbon Tetrachloride	56-23-5	5	5
16.	Vinyl Acetate	108-05-4	10	10
17.	Bromodichloromethane	75-27-4	5	5
18.	1,1,2,2-Tetrachloroethane	79-34-5	5	5
19.	1,2-Dichloropropane	78-87-5	5	5
20.	trans-1,3-Dichloropropene	10061-02-6	5	5
21.	Trichloroethene	79-01-6	5	5
22.	Dibromocloromethane	124-48-1	5	5
23.	1,1,2-Trichloroethane	79-00-5	5	5
24.	Benzene	71-43-2	5	5
25.	cis-1,3-Dichloropropene	10061-01-5	5	5
26.	2-Chloroethyl Vinyl Ether	110-75-8	10	10
27.	Bromoform	75-25-2	5	5
28.	2-Hexanone	591-78-6	10	10
29.	4-Methyl-2-pentanone	108-10-1	10	10
30.	Tetrachloroethene	127-18-4	5	5
31.	Toluene	108-88-3	5	5
32.	Chlorobenzene	108-90-7	5	5
33.	Ethyl Benzene	100-41-4	5	5
34.	Styrene	100-41-4	5	5
35.	Total Xylenes	100-42-5	5	5

Table A-1, continued

	Semi-Volatiles	CAS Number	Detection Limits(1) Low Water(4) ug/l	Low Soil Sediment(5) ug/kg
36.	Phenol	108-95-2	10	330
37.	bis(2-Chloroethyl)ether	111-44-4	10	330
38.	2-Chlorophenol	95-57-8	10	330
39.	1,3-Dichlorobenzene	541-73-1	10	330
40.	1,4-Dichlorobenzene	106-46-7	10	330
41.	Benzyl Alcohol	100-51-6	10	330
42.	1,2-Dichlorobenzene	95-50-1	10	330
43.	2-Methylphenol	95-48-7	10	330
44.	bis(2-Chloroisopropyl)ether	39638-32-9	10	330
45.	4-Methylphenol	106-44-5	10	330
46.	N-Nitroso-Dipropylamine	621-64-7	10	330
47.	Hexachloroethane	67-72-1	10	330
48.	Nitrobenzene	98-95-3	10	330
49.	Isophorone	78-59-1	10	330
50.	2-Nitrophenol	88-75-5	10	330
51.	2,4-Dimethylphenol	105-67-9	10	330
52.	Benzoic Acid	65-85-0	50	1600
53.	bis(2-Chloroethoxy)methane	111-91-1	10	330
54.	2,4-Dichlorophenol	120-83-2	10	330
55.	1,2,4-Trichlorobenzene	120-82-1	10	330
56.	Naphthalene	91-20-3	10	330
57.	4-Chloroaniline	106-47-8	10	330
58.	Hexachlorobutadiene	87-68-3	10	330
59.	4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
60.	2-Methylnaphthalene	91-57-6	10	330
61.	Hexachlorocyclopentadiene	77-47-4	10	330
62.	2,4,6-Trichlorophenol	88-06-2	10	330
63.	2,4,5-Trichlorophenol	95-95-4	50	1600
64.	2-Chloronaphthalene	91-58-7	10	330
65.	2-Nitroaniline	88-74-4	50	1600
66.	Dimethyl Phthalate	131-11-3	10	330
67.	Acenaphthylene	208-96-8	10	330
68.	3-Nitroaniline	99-09-2	50	1600
69.	Acenaphthene	83-32-9	10	330
70.	2,4-Dinitrophenol	51-28-5	50	1600
71.	4-Nitrophenol	100-02-7	50	1600
72.	Dibenzofuran	132-64-9	10	330
73.	2,4-Dinitrotoluene	121-14-2	10	330
74.	2,6-Dinitrotoluene	606-20-2	10	330
75.	Diethylphthalate	84-66-2	10	330

Table A-1, continued

	Semi-Volatiles	CAS Number	Detection Limits(1) Low Water(4) ug/l	Low Soil Sediment(5) ug/kg
76.	4-Chlorophenyl Phenyl ether	7005-72-3	10	330
77.	Fluorene	86-73-7	10	330
78.	4-Nitroaniline	100-01-6	50	1600
79.	4,6-Dinitro-2-methylphenol	534-52-1	50	1600
80.	N-nitrosodiphenylamine	86-30-6	10	330
81.	4-Bromophenyl Phenyl ether	101-55-3	10	330
82.	Hexachlorobenzene	118-74-1	10	330
83.	Pentachlorophenol	87-86-5	50	1600
84.	Phenanthrene	85-01-8	10	330
85.	ANTHRACENE	120-12-7	10	330
86.	Di-n-butylphthalate	84-74-2	10	330
87.	Fluoranthene	206-44-0	10	330
88.	Pyrene	129-00-0	10	330
89.	Butyl Benzyl Phthalate	85-68-7	10	330
90.	3,3'-Dichlorobenzidine	91-94-1	20	660
91.	Benzo(a)anthracene	56-55-3	10	330
92.	bis(2-ethylhexyl)phthalate	117-81-7	10	330
93.	Chrysene	218-01-9	10	330
94.	Di-n-octyl Phthalate	117-84-0	10	330
95.	Benzo(b)fluoranthene	205-99-2	10	330
96.	Benzo(k)fluoranthene	207-08-9	10	330
97.	Benzo(a)pyrene	50-32-8	10	330
98.	Indeno(1,2,3-cd)pyrene	193-39-5	10	330
99.	Dibenz(a,h)anthracene	53-70-3	19	330
100.	Benzo(g,h,i)perylene	191-24-2	10	330

Table A-1, continued

	Pesticides	CAS Number	Detection Limits(1) Low Water(6) ug/l	Low Soil Sediment(7) ug/kg
101.	alpha-BHC	319-84-6	0.05	8.0
102.	beta-BHC	319-85-7	0.05	8.0
103.	delta-BHC	319-86-8	0.05	8.0
104.	gamma-BHC (Lindane)	58-89-9	0.05	8.0
105.	Heptachlor	76-44-8	0.05	8.0
106.	Aldrin	309-00-2	0.05	8.0
107.	Heptachlor Epoxide	1024-57-3	0.05	8.0
108.	Endosulfan I	959-98-8	0.05	8.0
109.	Dieldrin	60-57-1	0.10	16.0
110.	4,4'-DDE	75-55-9	0.10	16.0
111.	Endrin	72-20-8	0.10	16.0
112.	Endosulfan II	33213-65-9	0.10	16.0
113.	4,4-DDD	72-54-8	0.10	16.0
114.	Endosulfan Sulfate	1031-07-8	0.10	16.0
115.	4,4'-DDT	50-29-3	0.10	16.0
116.	Endrin Ketone	53494-70-5	0.10	16.0
117.	Methoxychlor	72-43-5	0.5	80.0
118.	Chlordane	57-74-9	0.5	80.0
119.	Toxaphene	8001-35-2	1.0	160.0
120.	AROCLOR-1016	12674-11-2	0.5	80.0
121.	AROCLOR-1221	11104-28-2	0.5	80.0
122.	AROCLOR-1232	11141-16-5	0.5	80.0
123.	AROCLOR-1242	53469-21-9	0.5	80.0
124.	AROCLOR-1248	12672-29-6	0.5	80.0
125.	AROCLOR-1254	11097-69-1	1.0	160.0
126.	AROCLOR-1260	11096-82-5	1.0	160.0

Table A-1, continued

NOTES

(1) Detection limits listed for soil/sediment are based on net weight. The detection limits calculated by the laboratory for soil/sediments will be on dry weight basis and will be higher.

(2) Medium Water Contract Required Detection Limits (CRDL) for Volatile Hazardous Substances List (HSL) Compounds are 100 times the individual Low Water DL.

(3) Medium Soil/Sediment CRDL for Volatile HSL Compunds are 100 times the individual Low Water CRDL.

(4) Medium Water CRDL for Semi-Volatile HSL Compounds are 100 times the individual Low Water CRDL.

(5) Meidum Soil/Sediment CRDL for Semi-Volatile HSL Compunds are 60 times the individual Low Soil/Sediment CRDL.

(6) Medium Water CRDL for Pesticide HSL Compounds are 100 times the individual Low Water CRDL.

(7) Medium Soil/Sediment CRDL for Pesticide HSL Compunds are 15 times the individual Low Soil/Sediment CRDL.

* Specific detection limits are highly matrix dependent. The detection limit listed herein are provided for guidance and may not always be achievable.

[jap-400-66]

TABLE A-2

ELEMENTS DETERMINED BY INDUCTIVELY COUPLED PLASMA EMISSION OR ATOMIC ABSORPTION SPECTROSCOPY

Metal	Required Detection Level(1) ug/l
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Other	
Cyanide	10

NOTES

(1) Any analytical method specified in Exhibit D of IFB WA 84-J091/J092 may be utilized as long as the documented instrument or method detection limits meet the CRDL requirements. Higher detection levels may only be used in the following circumstances.

If the sample concentration exceeds two times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the CRDL.

[jap-400-65]

APPENDIX C-1

FIELD MEASUREMENT OF pH

WARZYN

pH

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible

Reagents and Apparatus:

1. pH meter (Orion 901 or 407A for lab use, Orion 211, 221, and 230 for field use).
2. Combination pH electrode.
3. Magnetic stirrer and stir bars (for lab use).
4. Beakers or plastic cups.
5. pH buffer solutions, pH 4.00, 7.00, and 10.00.
6. Deionized water in squirt bottle.

Calibration:

1. Place combination electrode in pH 7.00 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibration dial until reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4.00 or pH 10.00 buffer solution. Use pH 7.00 and 4.00 for samples with pH <8, and buffers 7.00 and 10.00 for samples with pH >8.
4. Wait several minutes and then turn slope adjustment dial until reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00, follow Steps 2-5 again.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into clean beaker or plastic cup.

3. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.

4. Check temperature of sample. It should be ± 2°C of the buffer solutions.

5. Rinse electrode with deionized water.

6. Immerse electrode in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record pH. Rinse electrode with deionized water.

7. Recheck calibration with pH 7.00 buffer solution after every 20 samples and at the end of the analytical run.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be ± 0.2 pH units. Average the results.

2. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.

Notes:

1. The pH test is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C of each other. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.

3. Electrode should be stored in pH 4.00 buffer.

4. Before leaving laboratory for field work:

    a. Check batteries.
    b. Do quick calibration at pH 7.00 and 4.00 to check electrode response and batteries.
    c. Obtain fresh pH buffer solutions.

5. Following field measurements:

   a. Report any problems with meter or electrode.
   b. Clean meter and meter case.
   c. Make sure electrode is stored in pH 4.00 buffer.

Approved 7/22/86

Michael Linskens

Michael J. Linskens
Laboratory Manager

[ALM-1-26]

APPENDIX C-2

INSTRUCTIONS MANUAL ORION MODEL 211
pH METER

WARZYN

INSTRUCTION MANUAL

model 211
digital pH meter

ORION RESEARCH

contents

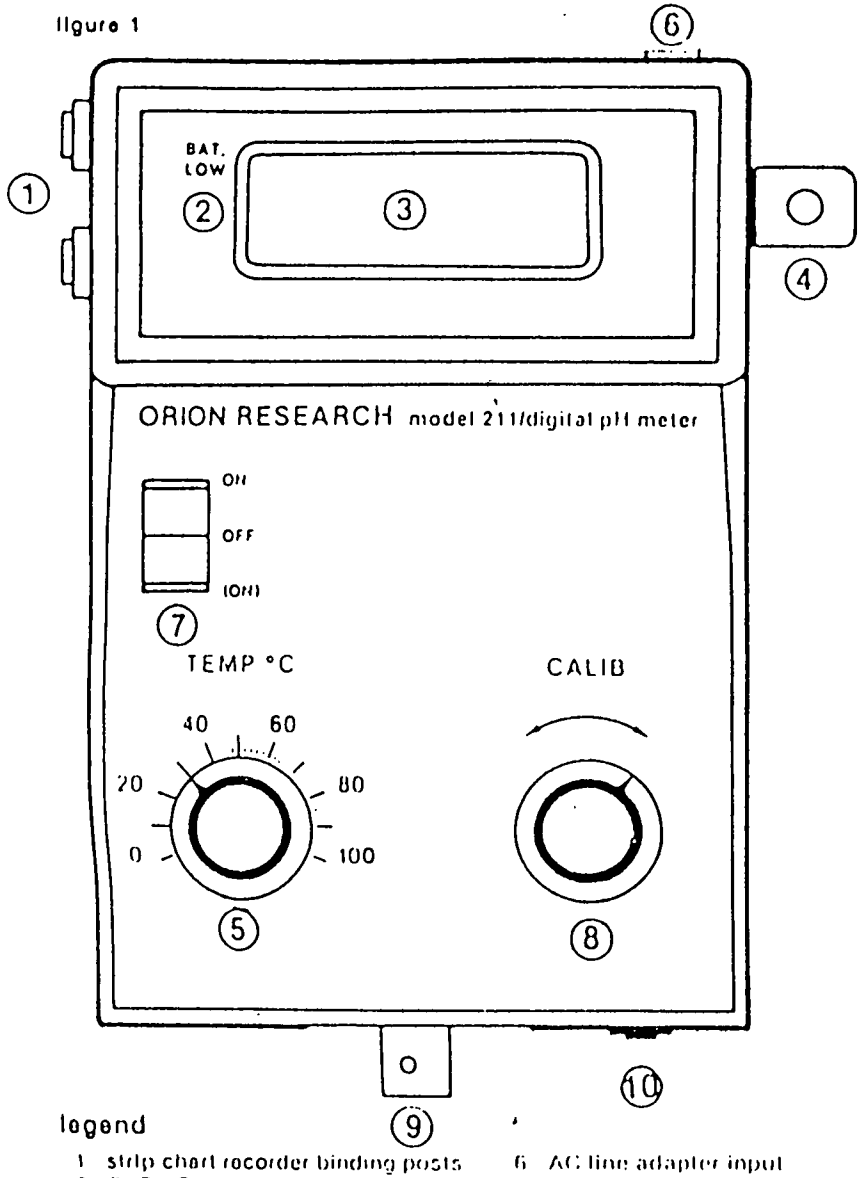
repair/service

For information on repair or replacement of this instrument, contact Orion Research toll-free. Ask for Customer Service.

ORION RESEARCH INCORPORATED
Customer Service
840 Memorial Drive
Cambridge, Massachusetts 02139 U.S.A.
800-225-1480 (Continental U.S.)
617-864-5400 (Massachusetts, Alaska, Hawaii, Canada)
Telex: 921466

Form IM211/3860
Printed in U.S.A.

figure 1



legend

1. strip chart recorder binding posts
2. BAT LOW
3. LC display
4. support rod clip
5. temperature indicator control
6. AC line adapter input
7. function control
8. calibration control
9. electrode connector
10. slope control

introduction

The Model 211 is a battery- or line-operated (110/220 V AC adapter) digital pH meter for field or laboratory use. The meter is complete with strip chart recorder binding posts and is supplied with an unbreakable, gel-filled combination pH electrode, one packet of pH 7 buffer powder, one bottle for pH 7 buffer, one bottle for distilled water, support rod, electrode holder, AC adapter, six 1.5 V batteries, shorting plug, and carrying case.

instrument description

See figure 1.

1. strip chart recorder binding posts: black post is low (ground) and red post is high input side of recorder. See page 8.
2. BAT LOW: an arrow pointing towards BAT LOW appears on the display when battery requires replacement.
3. LC display: pH display over the range of 0 - 14 with ± .01 pH units resolution.
4. support rod clip: holds steel rod used to mount electrode holder.
5. temperature indicator control (TEMP °C): compensates for variation in electrode slope or temperature changes. Used in two-buffer calibration.
6. AC line adapter input: jack used to insert AC line adapter. With AC line adapter operational, the internal battery is bypassed.
7. function control: rocker switch with three positions - ON, OFF and (ON). Depress (ON) for a momentary reading. The switch will return to OFF when released.
8. calibration control (CALIB): used to calibrate the meter with buffers of known pH.
9. electrode connector: accepts BNC connector from pH electrode.
10. slope control: screwdriver adjustment used to set second buffer in two-buffer calibration.

instrument set-up

support rod

1. Insert steel support rod into the hole in the support rod clip on side of the meter.
2. Mount electrode holder on the rod by pinching to compress the spring. Release to hold in place.

power source

The Model 211 operates on six nonrechargeable 1.5 volt batteries or on 110 or 220 ± 20% V with an approved AC adapter (specify voltage when ordering). Low battery is indicated by the BAT LOW indicator on the display.

NOTE: Batteries are not rechargeable - use of line adapter whenever possible will prevent the unit's batteries from being discharged. If battery operation is desired, follow installation instructions under battery replacement.

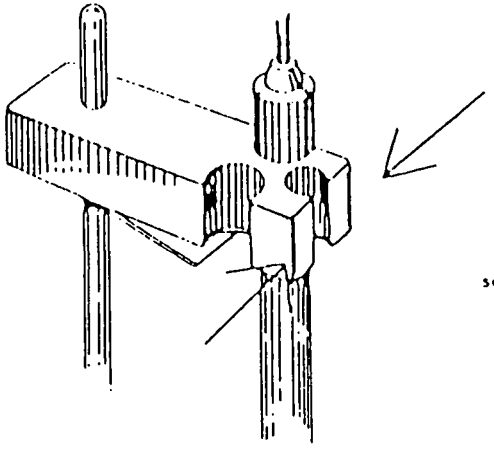
meter check-out

1. Install six AA batteries in the meter. Orient the (+) and (-) battery terminals to match the orientation shown in the battery compartment.
2. Depress ON button on the front panel. If the BAT. LOW indicator on the front display lights up, the batteries must be replaced.
3. If battery mode is not to be used, disregard steps 1 and 2. Insert pin end of appropriate AC line adapter into the meter, and the other end into the appropriate grounded AC line receptacle.
4. Attach BNC shorting plug to BNC input on the bottom side of the meter. Depress ON button on the front panel. Turn CAL/B knob so display reads a steady 7.00. If this cannot be done consult ORION Technical Service.
5. Remove the shorting plug. Successful completion of steps 1-4 show the [illegible] is ready for use.

connecting electrode

1. Insert the BNC connector into the electrode jack on the bottom panel of the meter. Turn connector clockwise until it seats firmly.
2. Mount electrode in the electrode holder by spreading the electrode clip open and sliding the electrode into the holder so that the clip closes on electrode cap. See figure 2.
3. Follow measurement procedures to use the meter to measure pH.
4. Disconnnect electrode by turning connector counterclockwise until released from pin.

figure 2



squeeze as shown to insert electrode

measurement procedures

general measurement technique

temperature: All samples and buffers should be at the same temperature, as small variations in temperature can cause errors in measurement. The slope of the pH electrode, the potential of the reference electrode, and the pH of the buffer are temperature-dependent.

cleaning electrodes: Electrode should be rinsed and shaken between measurements to remove drops and to prevent solution carryover.

stirring: Stir measured solutions moderately to obtain good contact between the glass bulb and the solution. Insert electrode to a depth of about 3 cm.

pH measurements

single-buffer standardization
(where maximum precision is not required)

NOTE: For maximum accuracy it is recommended that a two-buffer calibration be performed once at the beginning of each day (see page 7). This procedure ensures the correct setting of the slope control. Subsequent measurements during the day may be made using a single point calibration.

1. Place the electrode in a buffer solution whose pH is near the expected pH of the sample. Insert electrode to a depth of about 3 cm and stir moderately.
2. Set the temperature indicator control to the temperature of the buffer.
3. Set the function control to ON and allow the buffer reading to stabilize. Adjust the CALIB so that the display indicates the pH of the buffer at the solution temperature. See Table 1.
4. Remove the electrode from the buffer solution and rinse by stirring moderately in distilled water. Shake off excess drops of water.
5. Place electrode in the sample to a depth of about 3 cm and stir moderately. Set the function control to ON and allow the reading to stabilize. Record the steady pH reading.

two-buffer standardization
(where maximum precision is required)

1. Select two buffers to bracket the expected pH of the sample, with one buffer having a pH of 7.
2. Place the electrode in the pH 7 buffer to a depth of about 3 cm and stir moderately. Set the temperature indicator control to the temperature of the buffer. Set the function control to ON and allow the reading to stabilize. Turn CALIB until the display indicates the pH of the buffer at the solution temperature. See table 1.
3. Remove electrode from the first buffer and rinse by stirring moderately in distilled water. Shake off excess drops of water.
4. Place the electrode in the second buffer to a depth of about 3 cm and stir moderately. Set the function control to ON and adjust the slope control until the pH at the solution temperature is displayed. See Table 1.
5. Remove the electrode and rinse by stirring moderately in distilled water. Shake off excess drops of water.
6. Place the electrode in the sample to a depth of about 3 cm and stir moderately. Set the function control to ON and allow the reading to stabilize. Record the steady pH reading.

TABLE 1

TEMP (°C)	pH 7.00 Buffer	pH 4.01 Buffer	pH 10.01 Buffer
5	7.08	4.00	10.25
10	7.06	4.00	10.18
15	7.03	4.00	10.12
20	7.01	4.00	10.06
25	7.00	4.01	10.01
30	6.98	4.02	9.97
35	6.98	4.02	9.93
40	6.97	4.03	9.80
50	6.97	4.06	9.83
60	6.98	4.09	--

battery replacement

To replace the batteries, remove the panel on the back of the meter. Be sure to observe the polarity marking when inserting new batteries.

recorder output

The red and black binding posts at the side of the meter provide an output for strip chart recording of absolute mV independent of function mode. For recorders with input impedance of 100 Kilohms or greater, the output is fixed to about 100 mV/pH. pH 14.00 output is 1.40 V. Lower impedance recorders may be used but full-scale output is reduced.

1. Connect the lead from the high (input side of the recorder) to the red binding post and the lead from the low (ground) side to the black binding post.
2. Proceed according to directions in the strip chart recorder instruction manual.

repair and service

ORION warranty covers failures due to manufacturer's workmanship or material defect from the date of purchase by the user. User should return the warranty card to ORION and retain proof of purchase. Warranty is void if product has been abused, misused, or repairs attempted by unauthorized persons.

Warranties herein are for products sold/installed for use only in the United States and Canada. For ORION products purchased for use in all other countries consult local in-country, authorized ORION sales agent/distributor for product warranty information.

A Return Authorization Number must be obtained from ORION Laboratory Products Customer Service before returning any product for in-warranty repair, replacement or credit.

"No Lemon" Instrument Warranty

The instrument is covered by the ORION "No Lemon" warranty. If the instrument fails within twelve months from date of purchase for any reason other than abuse, the purchaser may elect to have it repaired or replaced at no charge. This warranty covers the original or replacement/repaired meter from date of original meter purchase; the warranty is not extended beyond the buyer's original warranty date.

accessories

815600	Ross™ epoxy body, bulb guard combination pH electrode
9104BN	Laboratory grade combination pH electrode (BNC connector)
910600	GX-series epoxy body, gel-filled combination electrode (BNC connector)
912600	GX-series epoxy body, gel-filled flask combination electrode (BNC connector)
913600	GX-series epoxy body, gel-filled flat surface combination pH electrode (BNC connector)
915600	RX-series refillable, epoxy body combination pH electrode (BNC connector)
9162BN	Combination pH electrode with rugged bulb (BNC connector)
9163BN	Combination pH electrode with needle shape (BNC connector)
910004	pH 4 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910007	pH 7 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910009	pH 9 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910104	pH 4.01 buffer, 475 ml bottle
910107	pH 7.00 buffer, 475 ml bottle
910110	pH 10.01 buffer, 475 ml bottle
970899	Dissolved oxygen electrode
910002	Electrode holder
020030	Shorting plug
020120	110V AC line adapter
020121	220V AC line adapter

specifications

package contents	model 211 digital pH meter, with model 910600 gel-filled unbreakable combination pH electrode, support rod, electrode holder, bottles for pH 7 buffer and distilled water, one packet pH 7 buffer powder, AC adapter, six 1.5 V batteries, and carrying case
range	0 to 14 pH
resolution	± .01 pH
temperature compensation	manual (0 to 100°C)
isopotential point	pH 7 (fixed)
power requirement	six 1.5 V batteries; battery life: 3000 ten second intermittent measurements when line adapter is not used. line adapter: 110 or 220 V ± 20%, 50/60 Hz
dimensions	14 cm high x 9 cm wide x 4.5 cm deep
weight	0.4 kg

specifications subject to change without notice

notice of compliance

The Model 211 may generate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class B computing device in accordance with specifications in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If the Model 211 does cause interference to radio or television reception, which can be determined by turning the unit off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- reorient the receiving antenna
- relocate the Model 211 with respect to the receiver
- move the Model 211 away from the receiver
- plug the Model 211 into a different outlet so that the meter and receiver are on different branch circuits

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet prepared by the Federal Communications Commission helpful:

"How to Identify and Resolve Radio-TV Interference Problems"

This booklet is available from the U.S. Government Printing Office, Washington, DC 20402, Stock No. 004-000-00345-4.

APPENDIX C-3

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE AND TEMPERATURE

WARZYN

CONDUCTIVITY
(YSI METER)

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance (Electrical Conductivity), umhos/cm @ 25°C

Reference: EPA 1983, p. 120.1.

Detection Limit: 1 umhos/cm @ 25°C

Sample Handling: Determine on-site

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. Conductivity standard, 1413 umhos/cm @ 25°C.

Procedure:

1. With mode switch of meter in off position, check zero setting. If not zeroed, use meter adjusting screw to zero.
2. Plug probe into jack located on side of meter.
3. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if meter cannot be aligned.
4. Analyse the conductivity standard. If the result is within the specified control ranges, analyse samples. A control should be analysed after every 20 samples and at the end of every analytical run.
5. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.
6. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use scale that produces a mid-range output on meter.
7. Wait for needle to stabilize (about 15 seconds) and record conductivity as indicated. Multiply reading by scale setting.
8. While gently agitating probe, take sample temperature (°C) to nearest 1°C and record.
9. Rinse probe with deionized water.
10. Record specific conductivity and temperature.

Quality Control:

1. A quality control check standard of 1413 umhos/cm is to be analyzed before and after every 10 samples. The check standard must be within the critical levels or the samples run prior to the last check standard are to be reanalyzed. Before the samples are reanalayzed, the analyst must diagnose the problem and consult with the laboratory supervisor until the problem has been resolved and approved. Record the result of the check standard in the quality control check standard book.

2. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be within 95%. Average the results.

3. All glassware is to be soap and water washed, tap rinsed and deionized rinsed prior to analysis.

Notes:

1. Calculate specific conductivity at 25°C using following formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

G_{25} = Specific conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

2. Analyze and record the conductivity standard solution (1413 umhos/cm @ 25°C) with each data set.

3. Record on field sheet which meter and probe were used. The meter should be wiped clean as necessary.

Reagent Preparation:

1. Conductivity Standard: Dissolve 0.7456g anhydrous KCl in deionized water and dilute to 1000 mL at 25°C in a volumetric flask. Specific conductance is 1413 umhos/cm at 25°C.

approved 9/6/86

Michael Linskens

Michael J. Linskens
Laboratory Manager

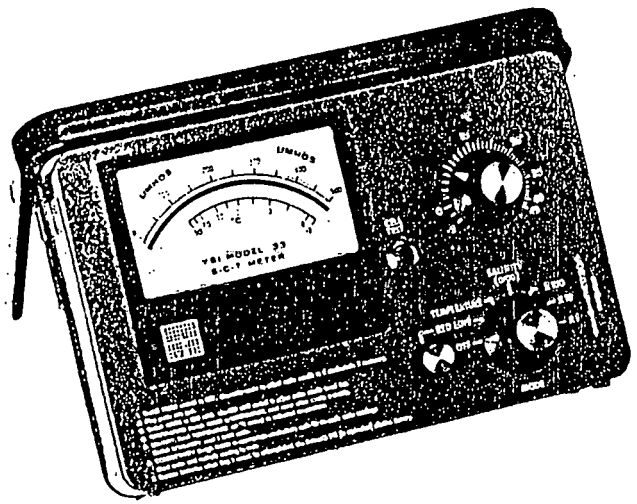
[AL-1-20]

APPENDIX C-4

OPERATING INSTRUCTIONS YSI MODEL 33
CONDUCTIVITY METER

WARZYN

INSTRUCTIONS FOR YSI MODEL 33 AND 33M S-C-T METERS



Scientific Division
Yellow Springs Instrument Co., Inc.
Yellow Springs, Ohio 45387 • Phone 513-767-7241

PRICE INCLUDING HANDLING $5.00

TABLE OF CONTENTS

GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged, plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as micromhos/centimeter (μmhos/cm); with the 33M, it's millisiemens/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1cm cube. (Conversion information: 1 μmho/cm = 0.1 mS/m.) Salinity is the number of grams of salt/kilogram of sample (‰ = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated; however, a temperature function is provided on the instrument to aid with calculation of corrections. Also, when just temperature and conductivity are known it is possible to calculate salinity, and when only temperature and salinity are known it is possible to calculate conductivity.

SPECIFICATIONS

Model 33 Conductivity

Ranges:	0-500, 0-5,000, 0-50,000 μmhos/cm with YSI 3300 Series Probes. (Note: The "μmho" designations on the meter are a shorthand form for "μmho/cm".)
Accuracy:	±2.5% max. error at 500, 5,000 and 50,000 plus probe. ±3.0% max. error at 250, 2,500 and 25,000 plus probe. See Error Section.

Readability:	2.5 μmhos/cm on 500 μmho/cm range. 25 μmhos/cm on 5,000 μmho/cm range. 250 μmhos/cm on 50,000 μmho/cm range.
Temperature Compensation:	None

Model 33M Conductivity

Ranges:	0-50, 0-500, 0-5,000 mS/m with YSI 3300 Series Probes.
Accuracy:	±2.5% max. error at 50, 500, and 5,000 plus probe. ±3.0% max. error at 25, 250, and 2,500 plus probe. See Error Section.
Readability:	0.25 mS/m on 50 mS/m range. 2.5 mS/m on 500 mS/m range. 25.0 mS/m on 5,000 mS/m range.
Temperature Compensation:	None.

Salinity

Range:	0-40 ‰ in temperature range of -2 to +45°C.
Accuracy:	Above 4°C, ±0.9 ‰ at 40 ‰ and ±0.7 ‰ at 20 ‰ plus conductivity probe. Below 4°C, ±1.1 ‰ at 40 ‰ and ±0.9 ‰ at 20 ‰ plus conductivity probe. See Error Section.
Readability:	0.2 ‰ on 0-40 ‰ range.
Temperature Compensation:	Manual by direct dial from -2 to +45°C.

Temperature	
Range	-2 to +50°C.
Accuracy	±0.1°C at -2°C, ±0.6°C at 45°C plus probe. See Error Section.
Readability	±0.15°C at -2°C to ±0.37°C at 45°C.
Power Supply	Two D-size alkaline batteries, Eveready E95 or equivalent, provide approximately 200 hrs. of operation.
Probe	YSI 3300 Series Conductivity/Temperature Probe. Nominal Probe Constant: K = 5/cm
Accuracy	±2% of reading for conductivity and salinity. Error of ±0.1°C at 0°C and ±0.3°C at 40°C.
Instrument	
Ambient Range	Satisfactory operation -5 to +45°C. A maximum error of ±0.1% of the reading per °C change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading.

OPERATION PROCEDURE

1. Setup
   (a) Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity scale.
   (b) Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.
   (c) Plug the probe into the probe jack on the side of the instrument.
   (d) Put the probe in the solution to be measured. (See Probe Use.)
2. Temperature
   Set the MODE control to TEMPERATURE. Read the temperature on the bottom scale of the meter in degrees Celsius. Allow time for the probe temperature to come to equilibrium with that of the water before reading.
3. Salinity
   (a) Transfer the temperature reading from Step 2 to the °C scale on the instrument.
   (b) Switch the MODE control to the SALINITY position and read salinity on the red 0-40 ‰ meter range.
   (c) Depress the CELL TEST button. The meter reading should fall less than 2%; if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.
4. Conductivity on Model 33 (Model 33M data are in parentheses.)
   (a) Switch the MODE control to the X100 scale. If the reading is below 50 on the 0-500 range (5.0 on the 0-50 range), switch to the X10 scale. If the reading is still below 50 (5.0), switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in μmhos/cm (mS/m). Measurements are not temperature compensated.

Example:	Meter Reading:	247 (24.7)
	Scale:	X10
	Answer:	2470 μmhos/cm (247.0 mS/m)

(b) When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2%; if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

NOTE: The CELL TEST does not function on the X1 scale.

5. Error

The maximum error in a reading can be calculated by using the graphs in the following sections.

(1) Temperature

The temperature scale is designed to give the minimum salinity error when the temperature readings are used to compensate salinity measurements.

Figure 1 shows total error for probe and instrument versus °C meter reading.

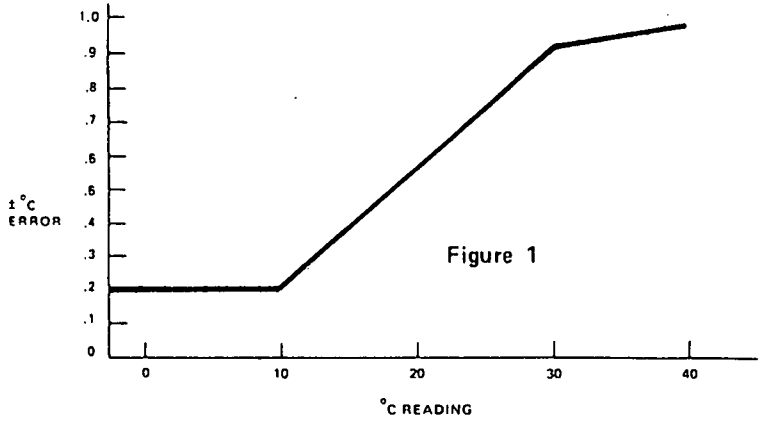


Figure 1

Example: Meter Reading: 15°C

Total Error: 0.4°C

Accuracy: 15°C ± 0.4°C for probe and instrument combined

(2) Conductivity on Model 33 (Model 33M data are in parentheses.)

Figure 2 shows the worst-case conductivity error as a function of the conductivity reading for the probe and instrument combined.

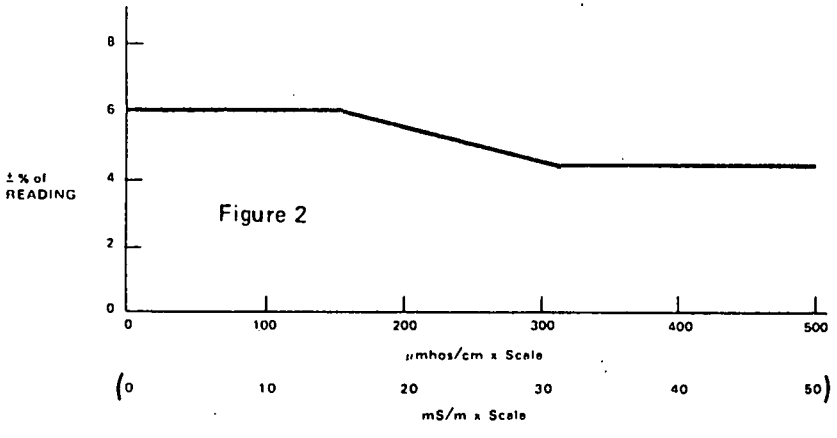


Figure 2

Example: Meter Reading: 360 µmhos/cm (36 mS/m)

Scale: X10

% Reading Error: ± 4.5%

Accuracy: 3600 ± 162 µmhos/cm (360 ± 16.2 mS/m) for probe and instrument

(3) Salinity

The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both. The temperature scale and temperature control have been designed to minimize the temperature error contribution to the salinity error. The error shown in Figure 3 is the total of the temperature and conductivity probe, the temperature scale and the salinity scale error.

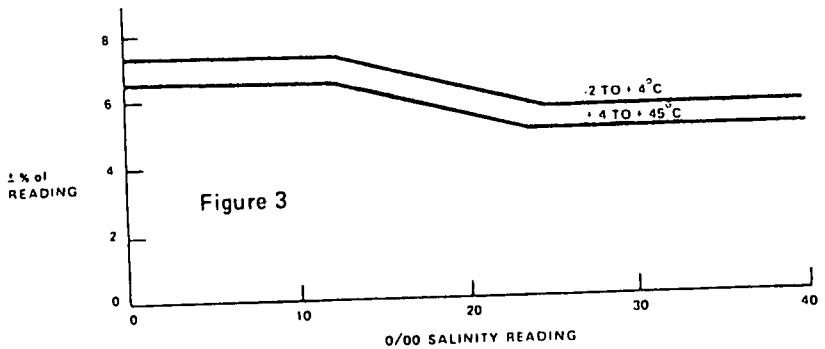
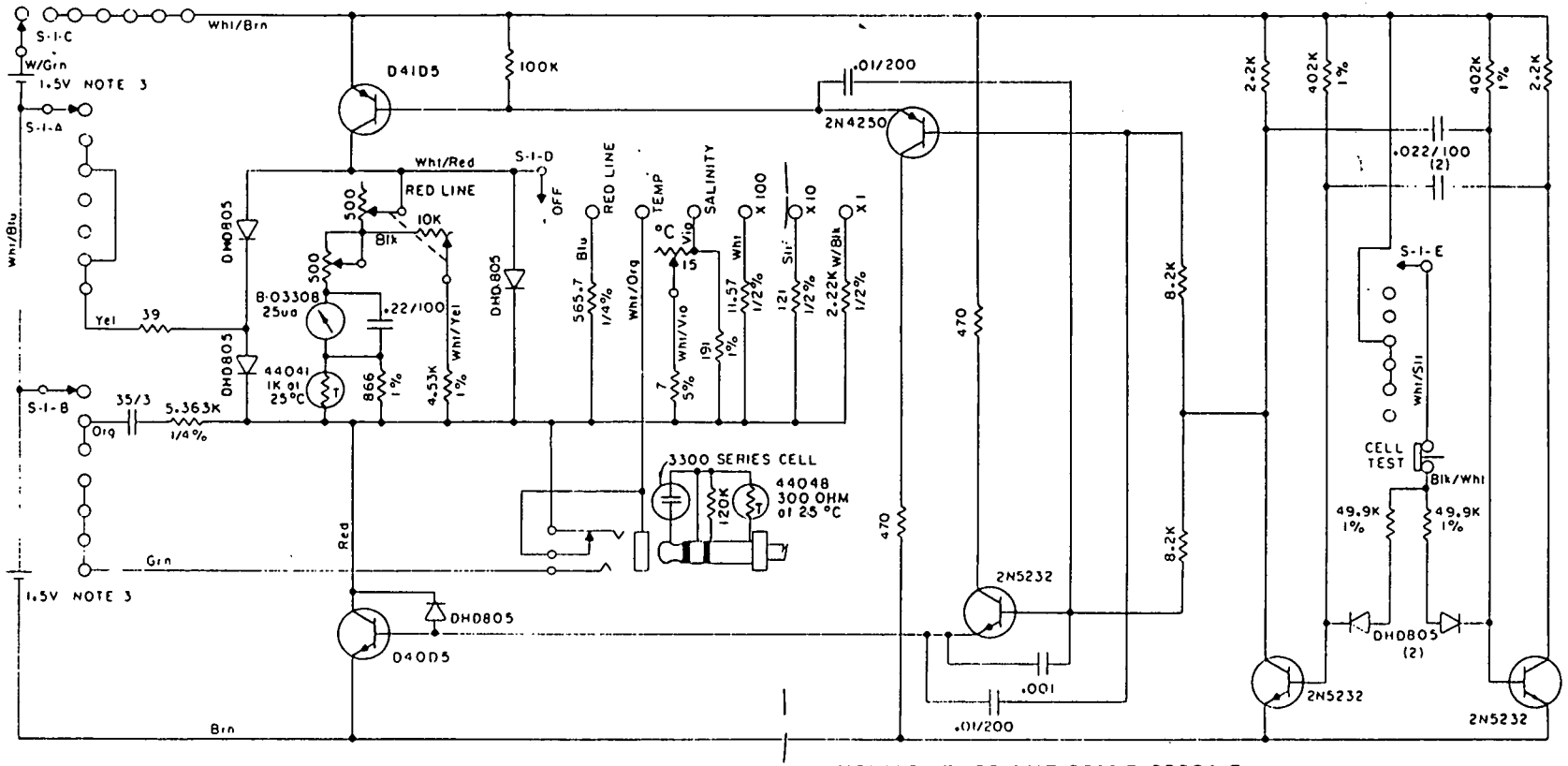


Figure 3

Example: Meter Reading: 10 0/00, @ 10°C

% of Reading Error: 6.5%

Accuracy: 10 ‰ ± 0.65 ‰ for all errors, combined worst case.



YSI MODEL 33 AND 33M B-03321-F

NOTE:

1. Resistance values in ohms, K = 1,000; resistors are ¼W, 10% unless otherwise specified.
2. The values shown on the schematic may differ from those in the instrument; if so, either value can be used for replacement purposes.
3. Battery is "D" size, alkaline only, Eveready E-95 or equal.

CIRCUIT DESCRIPTION, MAINTENANCE AND CALIBRATION

1. Description

The circuit is composed of two parts: a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature, redline and X1 positions the multivibrator operates at 100 Hz. In the salinity, X100 and X10 positions the multivibrator operates at 600 Hz and in these ranges pushing the CELL TEST button drops the frequency to 100 Hz allowing the operator to judge the degree of probe polarization.

2. Maintenance

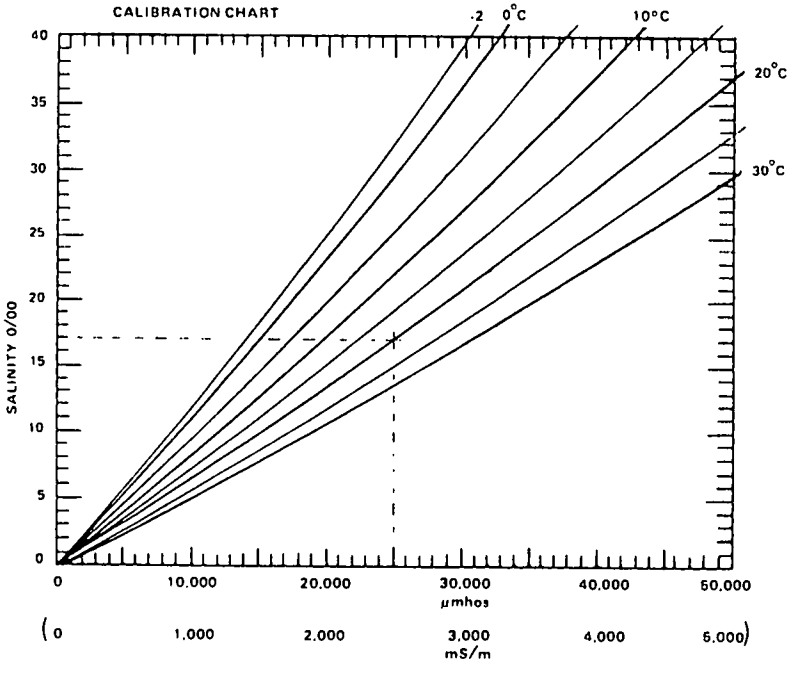
The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs. of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The Positive (+ button) end must go on red.

3. Calibration of Model 33 (Model 33M data are in parentheses.)

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency the dial can be re-positioned. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

(a) Read the temperature and conductivity of the solution. Determine the salinity of the solution by running a line vertically on the graph from this conductance value until it intersects the appropriate °C line (interpolate as required for temperature between the given °C lines). From this intersection extend a line horizontally to the edge of the graph. This determines the salinity for this sample.
Example: 25,000 μmhos/cm and 20°C gives a salinity of 17.
(Example: 2,500 mS/m and 20°C gives a salinity of 17.)



(b) Remove the °C knob, switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in Step (a). In the example given, the value is 17.

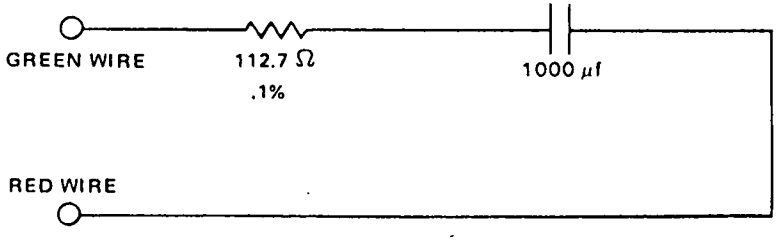
(c) Switch to TEMPERATURE. (Note: This temperature reading must be the same as Step (a); if not, begin again at Step (a).) Place the knob on the control shaft (without turning the control shaft) with the knob pointer at the same temperature as the meter reading and tighten both set screws securely.

At earliest opportunity recalibrate using the following procedure or return the instrument to factory for service.

(a) Set the instrument for a salinity measurement as normal.

(b) Substitute a 1000 μf capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



(c) Turn the temperature dial until the meter reads redline.

Now install the temperature knob with the arrow at 25°C. This is a temporary calibration only. Return the instrument to the factory for proper recalibration.

PROBE

1. Description of YSI 3300 Series Conductivity/Temperature Probe

The YSI 3300 Series Conductivity Probes are designed for field use, embodying construction and design for rugged, accurate service.

Each probe features a built-in cell constant of 5.0 (500.0/M) ±2%, a precision YSI thermistor temperature sensor of ±0.1°C accuracy at 0°C and ±0.3°C at 40°C and a low capacitance cable assembly terminating in a three therminal 0.25" dia. phone type connector.

The 3310 has a 10 ft. cable and the 3311 is a 50 ft. version. Other lengths are available on special order.

The probe has a rigid P.V.C. body, platinized pure nickel electrodes, and a durable cable, providing resistance to a wide range of waterborne substances.

2. Maintenance

(a) Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oils and organic matter are the most likely contaminants.

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as: Dow Chemical "Bathroom Cleaner", Horizon Industries "Rally, Tile, Porcelain, and Chrome Cleaner"; Johnson Wax "Envy, Instant Cleaner" or Lysol Brand "Basin, Tub, Tile Cleaner."

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol and 1 part HCl can be used.

Always rinse the probe after cleaning and before storage.

CAUTION: Do not touch the electrodes inside the probe. Platinum black is soft and can be scraped off.

If cleaning does not restore the probe performance, re-platinizing is required.

(b) Re-Platinizing

Equipment Required —

(1) YSI #3140 Platinizing Solution, 2 fl. oz. (3% platinum chloride dissolved in 0.025% lead acetate solution).
(2) YSI Model 33 or 33M S-C-T Meter.
(3) 50 ml glass breaker or equivalent bottle.
(4) Distilled water.

Procedure —

(1) Clean the probe as in Section (a) — either method.
(2) Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes. Do not cover the top of the probe.
(3) Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrode. Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below:

Meter Reading		Time
μmhos/cm	mS/m	(minutes)
30,000	3,000	5
25,000	2,500	6
20,000	2,000	8
15,000	1,500	11
10,000	1,000	16

(4) After the elapsed time remove the probe and rinse in fresh water.

(5) Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

(c) Storage:
It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.

3. Probe Use

(a) Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.

(b) Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should it become necessary to add more weight to overcome water currents, we suggest limiting the total weight to two pounds (8 pairs). For weights in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.

(c) Gentle agitation by raising and lowering the probe several times during a measurement insures flow of specimen solution through the probe and improves the time response of the temperature sensor.

4. Cell Calibration & Standard Solutions

The YSI #3300 Series Cells are calibrated to absolute accuracy of ±1.5% based on a standard solution. Since the literature on conductivity does not indicate a consistently accepted standardization method, we have chosen the 0.01 demal KCl solution method as determined by Jones and Bradshaw in 1937 as our standard. Recent textbooks, as well as the ASTM standards, concur with this choice.

The solution is prepared by diluting 0.745 grams of pure dry KCl with distilled water until the solution is 1 kilogram. The table below shows the values of conductivity this solution would have if the distilled water were non-conductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

Temperature °C	Conductivity μmhos/cm	mS/m
15	1141.5	114.2
16	1167.5	116.8
17	1193.6	119.4
18	1219.9	122.0
19	1246.4	124.6
20	1273.0	127.3
21	1299.7	130.0
22	1326.6	132.7
23	1353.6	135.4
24	1380.8	138.1
25	1408.1	140.8
26	1436.5	143.7
27	1463.2	146.3
28	1490.9	149.1
29	1518.7	151.9
30	1546.7	154.7

The operator may use the standard solution and the table to check accuracy of a cell's constant or to determine an unknown constant. The formula is shown below:

$$K = \frac{R\,(C_1 + C_2)}{10^6} \quad \text{or} \quad \frac{R\,(S_1 + S_2)}{10^5}$$

where: K = Cell constant
R = Measured resistance in Ω

C_1 = Conductivity in μmhos/cm
C_2 = Conductivity in μmhos/cm of the distilled water used to make solution.
S_1 = Conductivity in mS/m
S_2 = Conductivity in mS/m of the distilled water used to make the solution.

R, C_1 and C_2, or S_1 and S_2, must either be determined at the same temperature or corrected to the same temperature to make the equation valid

Note: For further information on conductivity and the above standard information, refer to ASTM Standards Part 23 — Standard Methods of Test for Electrical Conductivity, or Water and Industrial Waste Water - ASTM Designation D1125-64.

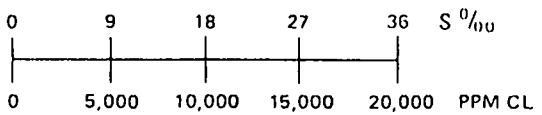
YSI MODEL 33 AND 33M USED WITH YSI 51A, 54 and 57 OXYGEN METERS

If the salinity measurement is to be used for salinity correction on the 51A, the reading should be converted to Chlorosity. The formula is:

$$\text{PPM Chlorosity} = \frac{\text{Salinity } ^0/_{00} - 0.03}{1.8} \times 10^3$$

For these instruments the 0.03 can be neglected so the equation simplifies to:

$$\text{PPM Cl} = \frac{\text{SS } ^0/_{00} \times 10^3}{1.8}$$



For salinity correction when using the Model 57 use the salinity reading direct from the Model 33 or 33M. No conversion is necessary.

Model 33 and 33M salinity readings taken in conjunction with Model 54 dissolved oxygen readings can be used to correct the Model 54 for salinity and to make post-measurement salinity corrections to dissolved oxygen data. Correction tables are available from the factory.

APPENDIX C-5

CALIBRATION AND MAINTENANCE OF PHOTOVAC TIP

WARZYN

CALIBRATION AND MAINTENANCE OF PHOTOVAC TIP

CALIBRATION

Two basic calibration operations must be performed. These are adjustment of zero and the adjustment of span.

The zero adjustment is the easiest. Under conditions where accuracy is very important and sensitivity is less important, it may be sufficient to zero the instrument using outdoor air. In other cases, office area or indoor may prove to be clean enough for zeroing purposes. When rigorously done, a source of "Zero Air" or "Ultra Zero Air" is necessary. These are high purity grades of compressed air available in bottled form. The bottle is fitted with a regulator and can be connected directly to the TIP's input fitting. A very low rate of flow should be used with pressure applied never exceeding 1 psi (6 kPa.).

Having adjusted zero (this is best done with the "Span" control at maximum), we will now turn to span calibration. To assess a situation where there is a high ionizable loading in the air consisting of a mixture of many components, it must be recognized that any reading obtained will be a composite of the various components. With photoionization, response factors vary greatly from compound to compound. This makes the reading on TIP dependent upon both concentration and nature of the mixture involved. TIP, in this case, works as a scoping tool; the user can move around the contaminated area seeking "hot spots".

HNu calibration gas will be used to calibrate the TIP. The TIP probe will be inserted into the gas cylinder feeder base and the gas released. The TIP will then be adjusted to the gas concentration (generally 52 ppm).

MAINTENANCE

Routine maintenance requirements for TIP are minimal. All that is required is to assure the batteries remain close to full charge (during periods of non-use) and to assure that the inlet frit-filter is kept clear of debris. The frit is a sintered, stainless type and must be periodically replaced to assure free-flow of air to the detector.

WARZYN

Replacement of the frit is indicated when the inlet flow falls below 140 mL/min.

Referring to the TIP pictorial diagram, remove the four cover mounting screws holding the detector cover in place. Remove any inlet probe that may have been installed. Make sure the unit is switched "Off". Lift the detector cover straight off the front of TIP with a twisting motion to overcome friction against the seal. Take the cover and place it upside down against a soft, but firm, surface such as a block of wood, so that the inlet fitting will not be damaged. From the inside of the fitting, press out the filter with a tool such as a 1/16th inch hex screwdriver.

Turn the cover right side up and position the new filter squarely in the inlet fitting. Press it into position with the same tool.

Make sure that the black PID seal is in place in its recess in the TIP detector and slip the detector cover into position, twisting it over the seal. Replace the four cover mounting screws.

Further maintenance operations that can be performed by the user involve the cleaning of the ion chamber and the lamp window, replacement of the lamp and replacement of the battery pack.

The ion chamber is reached by removing the detector cover (as previously described), unplugging the yellow collector wire from the printed circuit board of the UHF driver, releasing the red repeller wire at its attachment point on the PID (loosen the small screw and pull gently free) and finally, unscrewing the PID from the lamp holder by grasping gently but firmly the body of the PID and rotating counterclockwise. The lamp will pop up on a spring and may be lifted out for cleaning/replacement. The interior of the ion chamber contains a very delicate wire mesh and must not be touched with any solid object. The lamp window may be cleaned with a cotton swab dipped in methanol and the interior of the ion chamber may be blown free of dust using a gentle compressed air jet. The lamp (or its replacement) is simply put back into the lamp holder and the PID screwed back into place being very careful to avoid "cross threading". The two wires are replaced as before. It is vital

to assure that the PID seal is replaced in its seat before putting the cover back onto the detector.

If the pump and LEDs stay off for longer than a minute or so on a fully charged TIP, the detector lamp driving circuit may need adjustment. Remove the detector cover after which the pump and LEDs should come on as a result of ambient light hitting the photo resistor on the exposed circuit UHF driver circuit board.

Locate the ceramic trimming capacitor on the UHF driver; it has a screwdriver adjustment slot on the top of it. Be sure TIP is switched off, and make a pencil mark on the trimmer capacitor to indicate its original position. Turn the trimmer adjustment slightly (five degrees or so) in one direction or the other, then replace the detector cover and turn TIP "On". Repeat this procedure until the lamp starts. After TIP has run for two minutes or so, turn it off and set the trimmer back to its original position, or very near it, replace the detector cover, and use TIP.

If the lamp will not start regardless of the trimmer capacitor setting, the lamp likely needs replacing. Set the trimmer to its original position and replace the lamp as previously described. Lamp replacement is also indicated if, with fully charged batteries, TIP response drops drastically from one day to the next. Normally, a slight ozone smell will be present at the TIP vent. A failed lamp will not produce ozone.

[cac-65-18]

APPENDIX C-6

CALIBRATION AND MAINTENANCE OF HNu PHOTOIONIZER

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CALIBRATION AND MAINTENANCE
OF HNU PHOTOIONIZATION DETECTOR

(Extracted from Manufacturer's Instruction Manual)

To zero the instrument, turn the function switch to the standby position and rotate the zero photentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotation yields a downscale deflection. Note: no zero gas is needed, since this is an electronic zero adjustment (see below). If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted if necessary. Wait 15 or 20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero.

The instrument is now ready for calibration or measurement by switching the function switch to the proper measurement range. The instrument is supplied calibrated to read directly in ppm (v/v) 0-20, 0-200, 0-2000 of benzene with the span position set at 9.8. For additional sensitivity, the span potentiometer is turned counterclockwise (smaller numbers) to increase the gain. By changing the span setting from 10.0 to 1.0, the sensitivity is increased approximately ten fold. Then, the 0-20, 0-200, 0-2000 ppm scales become 0-2, 0-20, and 0-200 ppm full scale, respectively.

The span control is also utilized to make the instrument scale read directly in ppm of the compound being measured. E.G., it is adjusted to match the value of a calibration gas to that same reading on the instrument scale. The span control can be utilized to calibrate nearly any compound measured by photoionization to be direct reading on the 0-20 ppm range. For example, gain settings of 4.5 or 8.9, respectively, will provide direct reading capability (0-20, 0-200 ppm) for vinyl chloride and trichloroethylene, respectively.

Place the HNU probe top into the calibration gas cylinder discharge hose and open the gas valve. Place the scale selector on 0-200 and adjust to the calibration gas concentration (generally 52 ppm) by turning the potentiometer. The instrument is now calibrated. Recheck and adjust the zero if necessary.

[cac-65-19]

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APPENDIX C-7

CALIBRATION AND MAINTENANCE OF ORGANIC VAPOR ANALYZER (OVA)

WARZYN

Instruction & Service Manual

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2R900AC

CENTURY SYSTEMS

Portable Organic Vapor Analyzer
Model OVA-128

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TABLE OF CONTENTS

INTRODUCTION

The Century Model OVA-128 Portable Organic Vapor Analyzer (OVA) is a highly sensitive instrument designed to measure trace quantities of organic materials in air. It is essentially a hydrogen flame ionization detector such as utilized in laboratory gas chromatographs and has similar analytical capabilities. The flame ionization detector is an almost universal detector for organic compounds with the sensitivity to analyze for them in the parts per million range (V/V) in air in the presence of moisture, nitrogen oxides, carbon monoxide and carbon dioxide.

The instrument has broad application, since it has a continuous, chemically resistant air sampling system and can be readily calibrated to measure almost all organic vapors. It has a single linearly scaled readout from 0 ppm to 10 ppm with a X1, X10, X100 range switch. Designed for use as a portable survey instrument, it can also be readily adapted to fixed remote monitoring or mobile installations. It is ideal for the determination of many organic air pollutants and in the monitoring of air in potentially contaminated areas.

The OVA-128 is certified intrinsically safe by Factory Mutual Research Corporation (FM) for use in Class I, Division 1, Groups A, B, C & D hazardous environments. Similar foreign certifications have been obtained, including BASEEFA and Cerchar approval for Group IIC, Temperature Class T4 and equivalent approval from the Japanese Ministry of Labor. This requirement is especially significant in industries where volatile flammable petroleum or chemical products are manufactured, processed or used and for instruments which are actually used in portable surveying and in analyzing concentrations of gases and vapors. Such instruments must be incapable, under normal or abnormal conditions, of causing ignition of the hazardous atmospheric mixtures. In order to maintain the certified safety, it is important that the precautions outlined in this manual be practiced and that no modification be made to these instruments.

Sections 1 through 6 herein apply to the basic instrument. Section 7 contains information relative to options which are available and which may or may not have been purchased with your OVA.

It is highly recommended that the entire manual be read before operating the instrument. It is essential that all portions relating to safety of operation and maintenance, including Section 5, be thoroughly understood.

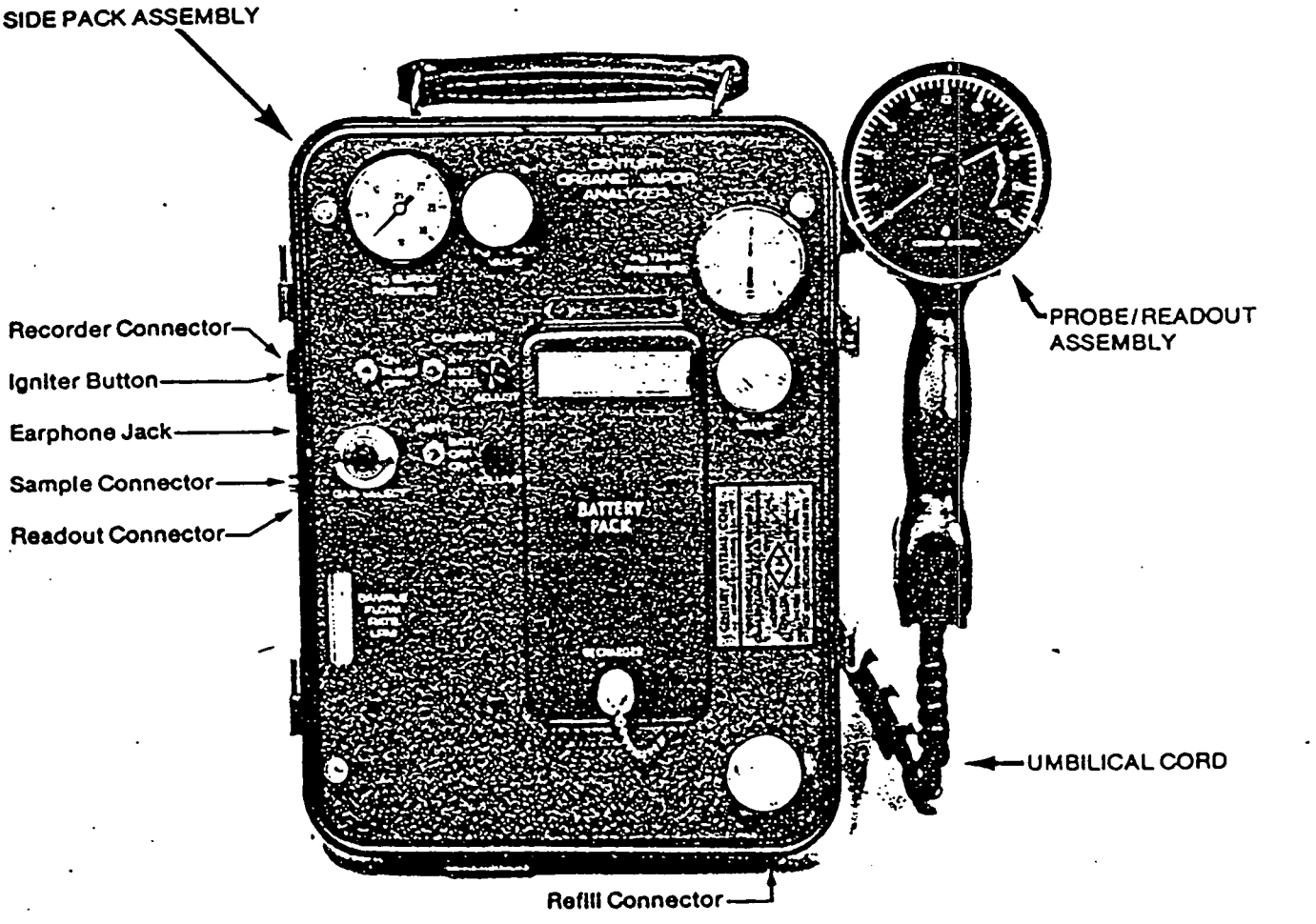


FIGURE 1-1. PORTABLE ORGANIC VAPOR ANALYZER
Model OVA-128

SECTION 1

DESCRIPTION AND LEADING PARTICULARS

1.1 GENERAL

The Century Portable Organic Vapor Analyzer (OVA), illustrated in Figure 1-1, is designed to detect and measure hazardous gases found in almost all industries. It has broad application, since it has a chemically resistant sampling system and can be calibrated to almost all organic vapors. It is extremely sensitive and can provide accurate indication of gas concentration in one of three ranges: 0 to 10 ppm; 0 to 100 ppm; and 0 to 1,000 ppm. While designed as a lightweight portable instrument, it can readily be adapted to remote monitoring applications.

The instrument utilizes the principle of hydrogen flame ionization for detection and measurement of organic vapors. The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated. During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system. The sample flow rate is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber, the sample is exposed to a hydrogen flame which ionizes the organic vapors. When most organic vapors burn, they leave positively charged carbon-containing ions which are collected by a negative collecting electrode in the chamber. An electric field exists between the conductors surrounding the flame and the collecting electrode which drives the ions to the collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated on the input electrode. This current is measured with a linear electrometer preamplifier which has an output signal proportional to the ionization current. A signal conditioning amplifier is used to amplify the signal from the preamp and to condition it for subsequent meter or external recorder display. The meter display is an integral part of the Probe/Readout Assembly and has a scale from 0 to 10.

1.2 TYPICAL APPLICATIONS

(1) Measurement of most toxic organic vapors present in industry for compliance with Occupational Safety and Health Administration (OSHA) requirements.
(2) Process monitoring and evaluation.
(3) Evaluation and monitoring applications in the air pollution field.
(4) Leak detection in storage, transportation and handling equipment.
(5) Survey of gas distribution and transmission lines and equipment for compliance with Office of Pipeline Safety (OPS) requirements.
(6) Forensic science applications.

1.3 OTHER TYPICAL USES

(1) Controlling and monitoring atmospheres in manufacturing and packaging operations.
(2) Mudlogging, gas and mineral exploration.
(3) Leak detection related to volatile fuel handling equipment.

1.4 MAJOR FEATURES

The basic instrument consists of two major assemblies, the Probe/Readout Assembly and the Side Pack Assembly (see Figure 1-1). The recorder is optional on all models, but is normally used with all instruments which incorporate the GC Option. The output meter and alarm level adjustments are incorporated in the Probe/Readout Assembly which is operated with one hand. The Side Pack Assembly contains the remaining operating controls and indicators, the electronic circuitry, detector chamber, hydrogen fuel supply and electrical power supply. It is a quantitative type instrument with sensitivity to 0.1 ppm methane.

Other major features are: 250° linear scale readout, less than two second response time and minimum eight hour service life for fuel supply and battery pack. A battery test feature allows charge condition to be read on the meter. Hydrogen flame-out is signified by an audible alarm plus a visual indication on the meter. The instrument contains a frequency modulated detection alarm which can be preset to sound at a desired concentration level. The frequency of the detection alarm

varies as a function of detected level giving an audible indication of organic vapor concentration. The instrument is designed for one man, one hand operation and the entire unit weighs a total of less than 12 pounds, including fuel supply and battery. An earphone is provided for "only operator" monitoring.

During use, the Side Pack Assembly can be carried by the operator on either his left or right side or as a back pack. The Side Pack Assembly is housed in a high impact plastic case and weighs less than 10 pounds. The Probe/Readout Assembly can be detached from the Side Pack Assembly and broken down for transport and storage. See Figure 1-2 for the breakdown capability of the instrument.

1.5 ADAPTABILITY FEATURES AND STANDARD ACCESSORIES

1.5.1 GENERAL

Maximum flexibility and operability features are included in the instrument design. As shown in Figure 1-2, a variety of pickup fixtures can be used. They can be installed by simply turning a knurled locking nut. Small diameter tubing can be used for remote sampling and electrically insulated flexible extensions can be used for difficult places to reach.

1.5.2 PROBE

The telescoping probe allows the length to be increased or decreased over an eight inch range to suit the individual user. A knurled locking nut is used to lock the probe at the desired length. The probe is attached to the Readout Assembly using a knurled locking nut. For measurements in close areas, the probe is replaced with a Close Area Sampler, which is supplied as a standard accessory.

1.5.3 PARTICLE FILTERS

The primary filter is of porous stainless and located behind the sample inlet connector, see Side Pack Assembly drawing in Appendix "A". In addition, replaceable porous metal filters are installed in the "close area" sampler, the pickup funnel and the tubular sampler.

1.5.4 INSTRUMENT CARRYING CASE

An instrument carrying case is provided to transport, ship and store the disassembled Probe/Readout Assembly, the Side Pack Assembly and other standard equipment.

1.5.5 MOBILE INSTALLATION

The instrument is readily adaptable to a mobile application by simply plugging into vehicle power and hydrogen fuel supply and making provisions for drawing sample from the vehicle primary sampling system.

1.6 SPECIFICATIONS

Sensitivity: 0.1 ppm (methane)
Response time: Less than 2 seconds
Readout: 0 to 10 ppm, 0 to 100 ppm, 0 to 1,000 ppm, 250° linear scaled meter; external monitor connector
Sample flow rate: Nominally 2 units
Fuel supply: 75 cubic centimeter tank of pure hydrogen at maximum pressure of 2300 PSIG, fillable while in case
Primary electrical power: Rechargeable and replaceable battery pack at 12VDC
Service life: Hydrogen supply and battery power-8 hours operating time minimum
Size: Standard Unit: 8-5/8 x 11-5/8 x 4-1/4 FM Unit: 8-5/8 x 11-5/8 x 4-1/2 Probe/Readout Assembly: Variable (see Figure 1-2)
Weight: Standard Unit: Side Pack Assembly, less than 10 lbs. FM Unit: Side Pack Assembly, less than 11 lbs. Probe/Readout Assembly: less than 2 lbs.
Operator requirements: One man, one hand operation
Detection alarm: Frequency modulated audible alarm. Can be preset to desired level. Frequency varies as a function of detection level
Flame-out indication: Audible alarm plus visual meter indication
Battery test: Battery charge condition indicated on readout meter or battery recharger
Pickup fixtures: Variety of types for various applications
Probe: Telescoping adjustment over 8 inches or probe can be completely removed from Readout Assembly
Umbilical cord: Cable between readout and sidepack with connectors for electrical cable and sample hose
Filtering: In-line particle filters and optional activated charcoal filter.
Side Pack case: Molded high impact plastic case with carrying handle and shoulder strap
Electrical protection: Refer to Section 5
Standard accessories:

1) Instrument carrying and storage case
2) Fuel filling hose assembly
3) A.C. battery charger
4) Earphone
5) Various pickup fixtures

Optional accessories:

1) Gas chromatograph option
2) Portable strip chart recorder
3) Activated charcoal filter; also used with desiccant as a moisture trap
4) Dilution valve
5) Septum adapter for use with gas chromatograph option

SECTION 2

DETAILED OPERATING PROCEDURES

2.1 GENERAL

The procedures in this section are broken into five parts: (1) Starting, (2) Operating, (3) Shut Down, (4) Fuel Refilling, and (5) Battery Charging. After familiarization

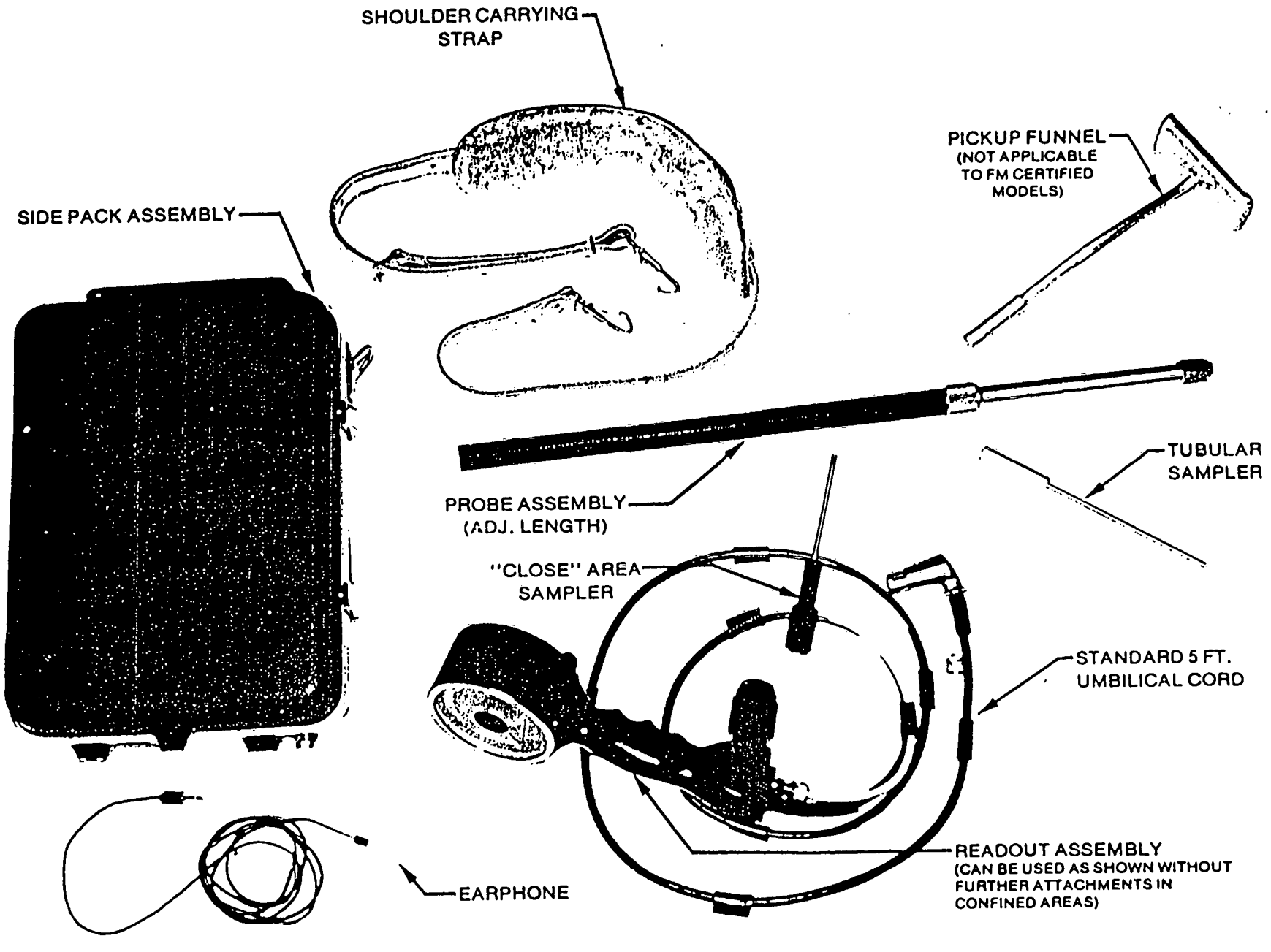


FIGURE 1-2

INSTRUMENT DISASSEMBLED

Typical of All Models Except Where Noted.

with the instrument, the summarized procedures described in Section 3 may be used for simplicity. Because of the many optional applications for the instrument, the comprehensive detailed procedures described in this section may seem complex. However, in normal applications the operating procedures are quite simple. A condensed operating procedure check list is provided inside the cover of the Side Pack Assembly. Refer to Section 7 for operating procedures relative to major optional accessories such as the Gas Chromatograph Option.

2.2 SYSTEM CONTROLS, INDICATORS AND CONNECTORS

Tables 2-1 and 2-2 describe the functions of the various controls, indicators and connectors illustrated in Figure 1-1. Unless otherwise noted, the listings in Tables 2-1 and 2-2 are applicable to both the Model OVA-118 and OVA-128.

TABLE 2-1
SIDE PACK ASSEMBLY

Controls/Indicators — Function

1) INSTR/BATT Test Switch - This 3 position toggle switch turns on all instrument electrical power except the pump and alarm power and also permits display of the battery charge condition on the readout meter.
2) PUMP (ON-OFF) Switch - This toggle switch turns on power to the internal pump and audio alarms.
3) Igniter Switch - This momentary push button switch connects power to the igniter coil in the detector chamber and simultaneously disconnects power to pump.
4) CALIBRATE Switch (range selector) - This 3 position toggle switch selects the desired range: X1 (0-10 ppm); X10 (0-100 ppm); X100 (0-1,000 ppm).
5) CALIBRATE ADJUST (zero) Knob - This potentiometer is used to "zero" the instrument.
6) GAS SELECT Knob (span control) - This ten-turn dial readout potentiometer sets the gain of the instrument commonly referred to as span control.
7) Recorder Connector - This 126 series 5-pin Amphenol connector is used to connect the instrument to an external monitor with the following pin connections.

   Pin E - plus 12VDC
   Pin H - Ground
   Pin A - Signal 0-5VDC (OVA-118 only)
   Pin B - Signal 0-5VDC (OVA-128 only)

8) Recharger Connector - This BNC connector is used to connect the battery pack to the battery recharger assembly.
9) H2 TANK VALVE - This valve is used to supply or close off the fuel supply from the hydrogen tank.
10) H2 TANK PRESSURE Indicator - This high pressure gauge measures the pressure in the hydrogen fuel tank which is an indication of fuel supply.
11) H2 SUPPLY VALVE - This valve is used to supply or close off the hydrogen fuel to the detector chamber.
12) H2 SUPPLY PRESSURE Indicator - This low pressure gauge is used to monitor the hydrogen pressure at the capillary restrictor.
13) SAMPLE FLOW RATE Indicator - This indicator is used to monitor the sample flow rate.
14) Refill Connection - This 1/4" AN fitting is used to connect the hydrogen refill hose to the instrument.
15) REFILL VALVE - This valve is used to open one end of the instrument fuel tank for refilling with hydrogen.
16) Earphone Jack - This jack is used to connect the earphone; it turns off speaker when used.
17) VOLUME Knob - This potentiometer adjusts the volume of the internal speaker and earphone.
18) Readout and Sample Connectors - These connectors are used to connect the sample hose and umbilical cord from the Probe/Readout Assembly to the Side Pack Assembly.

TABLE 2-2
PROBE/READOUT ASSEMBLY

Controls/Indicators — Function

A) Meter - This 250° linear scaled meter displays the output signal level in ppm.
B) Alarm Level Adjust Knob - This potentiometer (located on the back of the Readout Assembly) is used to set the concentration level at which the audible alarm is actuated.

2.3 STARTING PROCEDURE

2.3.1 INITIAL PREPARATION FOR USE

2.3.1.1 INITIAL ASSEMBLY (Reference Figure 1-2)

a) Normal Survey Configuration
   (1) Connect the adjustable length probe to the Readout Assembly with the captive locking nut. Ensure that the probe is seated firmly in the Readout Assembly.
   (2) Select the desired pickup fixture and check that a particle filter is installed.
   (3) Connect the pickup fixture to the probe using the knurled locking nut.
   (4) Connect the umbilical cord and sample hose to the Side Pack Assembly.
b) "Close Area" Survey Configuration
   (1) Check to ensure that a particle filter is installed in the close area sampler.
   (2) Connect the close area sampler directly to the Readout Assembly.
   (3) Connect the umbilical cord and sample hose to the Side Pack Assembly.

2.3.1.2 SERVICING

a) Fueling: Pure, dry hydrogen can normally be

purchased locally or in a high grade from the Matheson Company of East Rutherford, New Jersey. The maximum instrument supply bottle pressure is 2300 PSIG. A high pressure hydrogen filling hose assembly is provided with the instrument. This assembly includes the proper fittings for the instrument and supply bottle, and a three-way fill/bleed valve. Initial fueling and subsequent refilling, using the Century high pressure filling hose, should be accomplished in accordance with the detailed instructions described in Section 2.6 of this manual.

b) Battery Check: Move INSTR/BATT Test Switch to the BATT position and ensure battery is charged by reading the indication on the readout meter.

c) Calibration: Standard factory calibration is performed using methane in air. The GAS SELECT (span) Control is set and locked to the position for calibration to methane (factory setting is 300). If the instrument is calibrated for other organic vapors, the reading on the GAS SELECT Control must be set for that particular vapor.

2.3.1.3 SAFETY PRECAUTIONS

Certain safety precautions must be followed in using the instrument. Hydrogen gas, when mixed with air, is highly flammable. Operating and refueling instructions should be strictly followed to ensure safe, reliable operation. Section 5 of the manual provides detailed safety precautions.

2.3.2 TURN ON PROCEDURE

The GAS SELECT control should be preset to the desired dial indication prior to turn on. The procedure for determining this setting is contained in Section 4 of this manual. The instrument, as received from the factory, is set to measure in terms of methane in air.

a) Move the INSTR Switch to ON and allow five minutes for warm up.

b) To set the audible alarm to a predetermined level, first turn the PUMP Switch to ON, then adjust the meter pointer to the desired alarm level, using the CALIBRATE ADJUST (zero) Knob. Turn the Alarm Level Adjust Knob on the back of the Readout Assembly until the audible alarm just comes on. Adjust speaker volume with VOLUME Knob. If earphone is used, plug in and readjust the volume as desired. The instrument is then preset to activate the alarm when the level exceeds that of the setting.

c) Move the CALIBRATE Switch to X10 and adjust the meter reading to zero with the CALIBRATE ADJUST (zero) Knob.

d) Ensure the PUMP Switch is ON and observe the SAMPLE FLOW RATE Indicator. Indication should be approximately 2 units.

e) Open H2 TANK VALVE one (1) turn and observe the reading on the H2 TANK PRESSURE Indicator. (Approximately 150 psi of pressure is needed for each hour of operation.)

f) Open H2 SUPPLY VALVE 1/2 to 1 turn and observe the reading on the H2 SUPPLY PRESSURE Indicator.

CAUTION

Do not leave H2 SUPPLY VALVE open when the pump is not running, as this will allow hydrogen to accumulate in the detector chamber.

g) Confirm that meter is still reading zero (readjust if required).

h) Depress igniter button. There will be a slight "pop" as the hydrogen ignites and the meter pointer will move upscale of zero. Immediately after ignition, release the igniter button. Do not depress igniter button for more than 6 seconds. If burner does not ignite, let instrument run for several minutes and try again. After ignition, the meter pointer will indicate the background concentration. This background level is nulled out using the CALIBRATE ADJUST (zero) Knob. Reference paragraph 6.2.5.1.

NOTE

Since the OVA utilizes the sample air drawn by the pump into the detector chamber as the only source of air to support the hydrogen flame, without adjustment the instrument will read the actual background concentration (ppm) of all hydrocarbons present at a given location.

i) Move instrument to an area which is representative of the "lowest ambient background concentration" (cleanest air) to be surveyed. Move the CALIBRATE Switch to X1 and adjust the meter to read 1 ppm with the CALIBRATE ADJUST (zero) Knob.

NOTE

Adjustment to 1 ppm (rather than 0) is necessary in the X1 range because of the sensitivity of the OVA. This permits minor downward fluctuations in the normal background level without dropping below 0, which would actuate the flame-out alarm. It is important, therefore, to remember during the subsequent survey that 1 ppm must be subtracted from all readings. Therefore, a 1.8 ppm reading would actually be only 0.8 ppm.

j) If the alarm level is to be set above the normal background detection level, turn the Alarm Level Adjust Knob on the back of the Readout Assembly until it actuates slightly above background.

THE INSTRUMENT IS NOW READY FOR USE.

2.4 OPERATING PROCEDURES

a) Set the CALIBRATE Switch to the desired

range. Using one hand operation, survey the areas of interest while observing the meter and/or listening for the audible alarm indication. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. For broad surveys outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative reading or pinpointing, the pickup fixture should be positioned at the point of interest.

b) When organic vapors are detected, the meter pointer will move upscale and the audible alarm will sound when the preset point is exceeded. The frequency of the audible alarm will increase as the detection level increases.

c) If the flame-out alarm is actuated, ensure that the pump is running, then press the igniter button. Under normal conditions, flame-out results from sampling a gas mixture that is above the lower explosive level which causes the H2 flame to extinguish. If this is the case, reignition is all that is required.
Another possible cause for flame-out would be restriction of the sample flow line which would not allow sufficient air into the chamber to support combustion of the H2 flame. The normal cause for such restriction would be a clogged particle filter or other restriction in the line.
It should be noted that the chamber exhaust port is on the bottom of the case and blocking this port with the hand will cause fluctuations and/or flame-out.

2.5 SHUT DOWN PROCEDURE

The following procedure should be followed for shut down of the instrument:

1) Close H2 SUPPLY VALVE.
2) Close H2 TANK VALVE.
3) Move INSTR Switch to OFF.
4) Wait 5 seconds and move PUMP Switch to OFF. INSTRUMENT IS NOW IN A SHUT DOWN CONFIGURATION.

2.6 FUEL REFILLING

a) The instrument should be completely shut down as described in Section 2.5 herein during hydrogen tank refilling operations. The refilling should be done in a ventilated area. There should be no potential igniters or flame in the area.

b) If you are making the first filling of the instrument or if the filling hose has been allowed to fill with air, the filling hose should be purged with N2 or H2 prior to filling the instrument tank. This purging is not required for subsequent fillings.

c) The filling hose assembly should be left attached to the hydrogen supply tank when possible. Ensure that the FILL/BLEED Valve on the instrument end of the hose is in the OFF position. Connect the hose to the refill connection on the Side Pack Assembly.

d) Open the hydrogen supply bottle valve slightly. Open the REFILL VALVE and the H2 TANK VALVE on the instrument panel and place the FILL/BLEED Valve on the filling hose assembly in the FILL position. The pressure in the instrument tank will now be indicated on the H2 TANK PRESSURE indicator.

e) After the instrument fuel tank is filled, shut off the REFILL VALVE on the panel, the FILL/BLEED Valve on the filling hose assembly and the hydrogen supply bottle valve.

f) The hydrogen trapped in the hose should now be bled off to atmospheric pressure. CAUTION should be used in this operation as described in Step (g) below, since the hose will contain a significant amount of hydrogen at high pressure.

g) The hose is bled by turning the FILL/BLEED Valve on the filling hose assembly to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED Valve should be turned to the FILL position to allow the hydrogen trapped in the connection fittings to go into the hose assembly. Then, again, turn the FILL/BLEED Valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED Valve to OFF to keep the hydrogen at one atmosphere in the hose so that at the time of the next filling there will be no air trapped in the filling line.

h) Close the H2 TANK VALVE.

i) With the H2 TANK VALVE and the H2 SUPPLY VALVE closed, a small amount of H2 at high pressure will be present in the regulators and plumbing. As a leak check, observe the H2 TANK PRESSURE Indicator while the remainder of the system is shut down and ensure that the pressure indication does not go down rapidly, indicating a significant leak. If it does decrease rapidly (greater than 350 PSIG/hr.), there is a significant leak in the H2 supply system.

2.7 BATTERY RECHARGING

a) Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet. Never charge in a hazardous area or environment.

b) Move the battery charger switch to the ON position. The light above the switch button should illuminate.

c) Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right when charging. When fully charged, the pointer will be in line with "charged" marker above the scale.

d) Approximately one hour of charging time is required for each hour of operation. However, an overnight charge is recommended. The charger can be left on indefinitely without

damaging the batteries. When finished, move the battery charger switch to OFF and disconnect from the Side Pack Assembly.

The following are special instructions relative to batteries which have been allowed to completely discharge.

It has been established that the above battery recharging procedures may not be sufficient when the operator of the instrument has inadvertently left the INSTR Switch ON for a period of time without recharging and allowed the battery to completely discharge.

When this happens and the above procedures fail to recharge the battery, the following should be accomplished:

1) Remove the battery from the instrument case.
2) Connect to any variable DC power supply.
3) Apply 40 volts at 1/2 amp maximum.
4) Observe the meter on the power supply frequently and as soon as the battery begins to draw current, reduce the voltage on the power supply at a slow rate until the meter reads approximately 15 volts. NOTE: The time required to reach the 15 volt reading will depend on degree of discharge.
5) Repeat steps a), b), c), and d) above to continue charging.

2.7.2 DC CHARGER

a) The optional DC charger is designed to both charge the battery and to provide power for operating the instrument from a 12 volt DC source, such as vehicle power.
b) Connect the DC charger cord to the connector on the battery cover of the Side Pack Assembly. Plug the line cord into the vehicle cigarette lighter or other power source connection.
c) In mobile applications, the DC charger is used to supply vehicle power to the instrument. Therefore, it may be left connected at all times.

2.8 CHARCOAL FILTERING

When it is desired to preferentially remove the heavier hydrocarbons, such as those associated with automobile exhaust, gasoline, etc., simply remove the pickup fixture from the end of the probe and install the optional charcoal filter assembly.

This same charcoal filter assembly can be installed directly into the Readout Assembly by using the adapter provided.

2.9 MOISTURE FILTERING

Filtering of moisture in the sample is not normally required. However, when moving in and out of buildings in cold weather, excessive condensation can form in the lines and detector chamber. In this case, the charcoal filter adapter can be filled with a desiccant such as "Drierite" which will filter out the moisture contained in the sample.

SECTION 3

SUMMARIZED OPERATING PROCEDURES

3.1 GENERAL

The procedures presented in this section are intended for use by personnel generally familiar with the operation of the instrument. Section 2 presents the comprehensive detailed operating procedures.

It is assumed that, prior to start up the positions of all switches and valves are in shut down configuration as described in paragraph 3.3.

3.2 START UP

a) Move PUMP Switch to ON and check battery condition by moving the INSTR Switch to the BATT position.
b) Move INSTR Switch to ON and allow five (5) minutes for warm-up.
c) Set Alarm Level Adjust Knob on back of Readout Assembly to desired level.
d) Set CALIBRATE Switch to X10 position, use CALIBRATE Knob and set meter to read 0.
e) Move PUMP Switch to ON position then place instrument panel in vertical position and check SAMPLE FLOW RATE indication.
f) Open the H2 TANK VALVE and the H2 SUPPLY VALVE.
g) Depress Igniter Button until burner lights. Do not depress Igniter Button for more than six (6) seconds. (If burner does not ignite, let instrument run for several minutes and again attempt ignition.)
h) Use CALIBRATE Knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set CALIBRATE Switch to X1 and readjust zero on meter. To avoid false flame-out alarm indication, set meter to 1 ppm with CALIBRATE Knob and make differential readings from there.

3.3 SHUT DOWN

a) Close the H2 SUPPLY VALVE and the H2 TANK VALVE.
b) Move the INSTR Switch and PUMP Switch to OFF.
c) Instrument is now in shut down configuration.

SECTION 4

CALIBRATION

4.1 GENERAL

The OVA is capable of responding to nearly all organic compounds. For precise analyses it will be necessary to calibrate the instrument with the specific compound of interest. This is especially true for materials containing elements other than carbon and hydrogen.

The instrument is factory calibrated to a methane in air standard. However, it can be easily and rapidly calibrated to a variety of organic compounds. A GAS SELECT control is incorporated on the instrument panel which is used to set the electronic gain to a particular organic compound.

Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four (4) such adjustments all located on the electronics board. One adjustment potentiometer, R-38, is used to set the power supply voltage and is a one-time factory adjustment. The remaining three adjustments, R-31, R-32 and R-33 are used for setting the electronic amplifier gain for each of the three (3) calibrate ranges. Access to the adjustments is accomplished by removing the instrument from its case. Figure 4-1 indicates the location of the adjustments.

4.2 ELECTRONIC ADJUSTMENTS

Primary calibration of this instrument is accomplished at the factory using methane in air sample gases.

4.2.1 GAIN ADJUSTMENT

a) Place instrument in normal operation with CALIBRATE Switch set to X10 and GAS SELECT control set to 300.
b) Use the CALIBRATE ADJUST (zero) Knob and adjust the meter reading to zero.
c) Introduce a methane sample of a known concentration (near 100 ppm) and adjust trimpot R-32 on circuit board (see Figure 4-1 for location) so that meter reads equivalent to the known sample.
d) This sets the instrument gain for methane with the panel mounted gain adjustment (GAS SELECT) set at a reference number of 300.
e) Turn off H2 SUPPLY VALVE to put out flame.

4.2.2 BIAS ADJUSTMENT

a) Leave CALIBRATE Switch on X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to 4 ppm.
b) Place CALIBRATE Switch in X1 position and, using trimpot R-31 on circuit board, adjust meter reading to 4 ppm. (See Figure 4-1)
c) Move CALIBRATE Switch to X10 position again. Use CALIBRATE ADJUST (zero) Knob to adjust meter to a reading of 40 ppm.
d) Move CALIBRATE Switch to X100 position and use trimpot R-33 on circuit board to adjust meter reading to 40 ppm.
e) Move CALIBRATE Switch to X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to zero.
f) Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

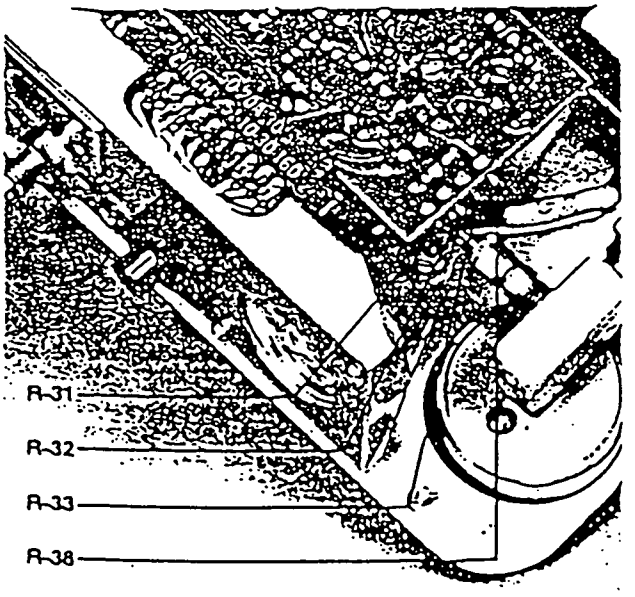


FIGURE 4-1. LOCATION OF ELECTRONIC ADJUSTMENTS
(Model OVA-118 shown; location typical to OVA-128)

4.3 CALIBRATION TO OTHER ORGANIC VAPORS

4.3.1 SETTING GAS SELECT CONTROL (Span)

Primary calibration of the instrument is accomplished using a known mixture of a specific organic vapor compound. After the instrument is in operation and the "normal background" is "zeroed out", draw a sample of the calibration gas into the instrument. The GAS SELECT Knob on the panel is then used to shift the readout meter indication to correspond to the concentration of the calibration gas mixture.

The instrument is then calibrated for the vapor mixture being used. After this adjustment, the setting on the "digidial" is read and recorded for that particular organic vapor compound. This exercise can be performed for a large variety of compounds and when desiring to read a particular compound the GAS SELECT control is turned to the predetermined setting for the compound. Calibration on any one range automatically calibrates the other two ranges.

4.3.2 USING EMPIRICAL DATA

Relative response data may be obtained, which can then be used to estimate concentrations of various vapors. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The relative response, in percent, for that test vapor would then be the concentration read/concentration of the calibrated sample X 100.

4.3.3 PREPARATION OF CALIBRATION STANDARDS

4.3.3.1 COMMERCIAL SAMPLES

Commercially available standard samples offer the most convenient and reliable calibration standards and are recommended for the most precise analyses. Always remember to obtain the cylinder with the desired sample and the "balance as air". Sample should be drawn from the cylinder into a collapsed sample bag, then drawn from the bag by the instrument to prevent a pressure or vacuum at the sample inlet.

4.3.3.2 PURE GASEOUS SAMPLES

Obtain a large collapsible sample bag, preferably polyethylene such as a 40 gallon trash can liner. Insert a tube into the bag opening and tie shut around the tube. The tubing should have a shut-off valve or plug and be suitable for connecting the OVA input tube. Determine the volume of the bag by appropriate means (i.e., wet-test meter, dimensions of the bag, etc.). Forty gallon polyethylene bags provide a volume of approximately 140-160 liters. For gas samples, flush a 10 cc hypodermic syringe with the compound to be tested and then inject a 10 cc sample through the wall of the air-filled bag. Immediately after withdrawing the needle, cover the hole with a piece of plastic tape. Allow a few minutes for the sample to completely diffuse throughout the bag. Agitation will ensure complete diffusion. Connect the outlet tube to the OVA and take a reading. To verify repeatability of sampling technique, disconnect the bag and inject a second sample of the gas into the bag without emptying. Since only 2 or 3 liters will have been removed, the overall volume change will be small and the instrument reading should now be twice that of the

original. The concentration in ppm (V/V) will be equal to sample size in cc divided by the volume of the bag in s times 1000. For example, a 10 cc gas sample when laced in a 160 liter bag will provide a sample of 63 ppm, i.e., 10 X 1000/160 equals 63 ppm.

3.3 GASEOUS AND LIQUID SAMPLES (Alternate Method)

Obtain a five (5) gallon glass bottle and determine its ume by measuring the volume of water needed to fill use of a 1000 ml graduated cylinder, obtainable from entific supply houses, is convenient). Another approach is to weigh the empty bottle, fill it with water and igh again. The difference between the two values is e weight of water. By multiplying the weight of water pounds by 0.455, you obtain the volume of the bottle n liters. Empty the water out and allow the bottle to dry. Place a one-foot piece of plastic tubing in the flask to aid mixing the vapors uniformly with the air. The volume such a bottle should be about 20 liters, which is 20,000 ml. If the volume were 20,000 ml, then a 2 ml sample of a gas placed in the bottle would be equivalent to 200 ml r 2 million ml or 100 ppm (V/V). Use of a gas tight syrge, readable in 0.01 ml, allows the preparation of mixures in the 1 - 2 ppm range, which are sufficient for the quantitative estimation of concentrations. A rubber topper is loosely fitted to the top of the bottle and the eedle of the syringe placed inside the jug neck and the topper squeezed against the needle to decrease leakage during sample introduction. Inject the sample to the bottle and withdraw the needle without removng the stopper. Put the stopper in tight and shake the ottle for a few minutes with sufficient vigor that the lastic tubing in the bottle moves around to ensure ood mixture of the vapors with the air.

For liquid samples, use of the following equation will llow the calculation of the number of microliters of organic liquid needed to be placed into the bottle to make 100 ppm (V/V) of vapor.

V1 equals V2 X Mw/244D

V1 - Volume of liquid in microliters needed to make an air mixture of 100 ppm (V/V)
V2 - Volume of bottle in liters
Mw - Molecular weight of substance
D - Density of substance

This procedure has the advantage that you can see when all of the organic liquid has vaporized and the volume can be determined readily.

For liquid samples, an alternate procedure involves the use of a diffusion dilution device such as that described by Desty, Geach and Goldup in "Gas Chromatography", R.P.W. Scott, ed., Academic Press, New York, 1961.

4.4 THEORY

Theoretical background and empirical data related to the Century Organic Vapor Analyzer is presented in 4.4.1 and 4.4.2.

4.4.1 HYDROCARBONS

In general, a hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives excellent repeatable results with all types of hydrocarbons; i.e., saturated hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

The typical relative response of various hydrocarbons to methane is as follows:

Compound	Relative Response (percent)
Methane	100 (reference)
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Toluene	120
Ethane	90

4.4.2 OTHER ORGANIC COMPOUNDS

Compounds containing oxygen, such as alcohols, ethers, aldehydes, carbolic acid and esters give a somewhat lower response than that observed for hydrocarbons. This is particularly noticeable with those compounds having a high ratio of oxygen to carbon such as found in the lower members of each series which have only one, two or three carbons. With compounds containing higher numbers of carbons, the effect of the oxygen is diminished to such an extent that the response is similar to that of the corresponding hydrocarbons.

Nitrogen-containing compounds (i.e., amines, amides and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Thus, $CHCl_3$ gives a much higher response than does CCl_4. As in the other cases, when the carbon to halogen ratio is 5:1 or greater, the response will be similar to that observed for simple hydrocarbons.

The typical relative response of various compounds to methane is as follows:

Methane	100 (calibration sample)
Ketones	
Acetone	60
Methyl ethyl ketone	80
Methyl isobutyl ketone	100
Alcohols	
Methyl alcohol	15
Ethyl	25
Isopropyl	65

Halogen compounds

Carbon tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

The OVA has negligible response to carbon monoxide and carbon dioxide which evidently, due to their structure, do not product appreciable ions in the detector flame. Thus, other organic materials may be analyzed in the presence of CO and CO_2.

SECTION 5

SAFETY CONSIDERATIONS

5.1 GENERAL

The Models OVA-108, OVA-128 and OVA-138 have been tested and certified by Factory Mutual Research Corporation (FM) as intrinsically safe for use in Class I, Division 1, Groups A, B, C & D hazardous atmospheres. Similar foreign certifications have been obtained, including BASEEFA and Cerchar approval for Group IIC, Temperature Class T4 on the Models OVA-108, OVA-128 and OVA-138, and equivalent approval from the Japanese Ministry of Labor for the Model OVA-128. Special restrictions must be strictly adhered to, to ensure the certification is not invalidated by actions of operating or service personnel.

All flame ionization hydrocarbon detectors are potentially hazardous since they burn hydrogen (H2) or H2 mixtures in the detector cell. Mixtures of H2 and air are flammable over a wide range of concentrations whether an inert gas such as nitrogen (N2) is present or not. Therefore, the recommended precautions and procedures should be followed for maximum safety. Safety considerations was a major factor in the design of the Organic Vapor Analyzer (OVA).

All connectors are of the permanent type as opposed to quick disconnect. To protect against external ignition of flammable gas mixtures, the flame detection chamber has porous metal flame arrestors on the sample input and the exhaust ports as well as on the H2 inlet connector. The standard battery pack and other circuits are internally current limited to an intrinsically safe level.

5.2 OPERATING, SERVICING AND MODIFYING

It is imperative that operation and service procedures described in this manual be carefully followed in order to maintain the intrinsic safety which is built into the OVA. No modification to the instrument is permissible. Therefore, component replacement must be accomplished with the same type parts.

5.3 ELECTRICAL PROTECTION

The 12V battery power supply circuit is current limited to an intrinsically safe level. Fuses are not utilized and all current limiting resistors and other components which are critical to the safety certification are encapsulated to prevent inadvertent replacement with components of the wrong value or specification. Under no circumstances should the encapsulation be removed.

5.4 FUEL SUPPLY & TANK

The OVA fuel tank has a volume of 75 to 85 cc which, when filled to the maximum rated pressure of 2300 PSIG, holds approximately 5/8 cubic foot of gas. The fuel used in the OVA is pure hydrogen which can be readily purchased in a highly pure form at nominal cost. The H2 tanks used in the instrument are made from stainless steel, proof-tested to 6,000 PSIG and 100% production tested to 4,000 PSIG.

5.5 H2 FLOW RESTRICTORS

Hydrogen gas gains heat when expanding and, therefore, should not be rapidly released from a high pressure tank to a low pressure environment. Flow restrictors are incorporated in the H2 refill fitting and H2 is restricted on the output side of the tank by the low flow rate control system. In addition, a special flow restrictor is incorporated in the FILL/BLEED valve of the hydrogen filling hose assembly. These precautions limit the flow rate of the H2 to prevent ignition due to self-heat from expansion.

5.6 DETECTOR CHAMBER

The OVA has a small flame ionization chamber cavity with sintered metal flame arrestors on both the input and output ports. The chamber is ruggedly constructed of teflon such that even if highly explosive mixtures of H2 and air are inadvertently created in the chamber and ignited, the chamber would NOT rupture.

5.7 H2 FILLING AND EMPTYING OPERATIONS

Precautions should be taken during H2 filling or H2 tank emptying operations to ensure that there are no sources of ignition in the immediate area. Since the instrument tank at 2300 PSIG holds only 5/8 cu. ft. of H2, the total quantity, if released to the atmosphere, would be quickly diluted to a non-flammable level. There is, however, the possibility of generating flammable mixtures in the immediate vicinity of the instrument during the filling or emptying operations if normal care is not exercised.

5.8 VENTING

The OVA case is vented to eliminate the possibility of trapping an explosive mixture of H2 and air inside the case.

SECTION 6

MAINTENANCE

6.1 GENERAL

This section describes the routine maintenance schedule recommended and provides procedures for trouble shooting malfunctions or failures in the instrument.

Appendix "A" to this manual contains the assembly drawings and associated parts list for the Side Pack Assembly and two major subassemblies; the Electronic Component Assembly and the Cylinder Assembly. These drawings and parts lists may be used for locating and identifying components. Also included in Appendix "A" is a schematic wiring diagram showing interconnecting wiring between major electronic assemblies and typical signal levels at selected points on the certified instruments. The enclosed drawings and parts lists are subject to change without notice and part replacement on any certified instrument should be verified to comply with the "no modifications permitted" requirement.

CAUTION

Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all portions of this manual relating to safety of operation, servicing and maintenance, including Section 5, be thoroughly understood. There should be no potential igniters or flame in the area when filling, emptying or purging the hydrogen system and the instrument should be turned off.

Extreme care should be exercised to ensure that required parts replacement is accomplished with the same parts specified by Century. This is especially necessary on the Models OVA-108, OVA-128 and OVA-138 in order that their certification for use in hazardous atmospheres be maintained. No modifications are permitted. Disassemble instrument only in a non-hazardous atmosphere.

6.2 ROUTINE MAINTENANCE

Note that Figure 6-1 is a flow diagram of the basic gas handling system.

6.2.1 FILTERS

6.2.1.1 PRIMARY FILTER

This filter is located behind the sample inlet connector (Fitting Assembly) on the Side Pack Assembly and is removed for cleaning by using a thin wall socket to unscrew the Fitting Assembly. The filter cup, "O" ring and loading spring will then come out as shown in the Side Pack Assembly drawing in Appendix "A". The porous stainless filter cup can then be cleaned by blowing out or washing in a solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter. Reassemble in reverse order ensuring that the "O" ring seal on the Fitting Assembly is intact.

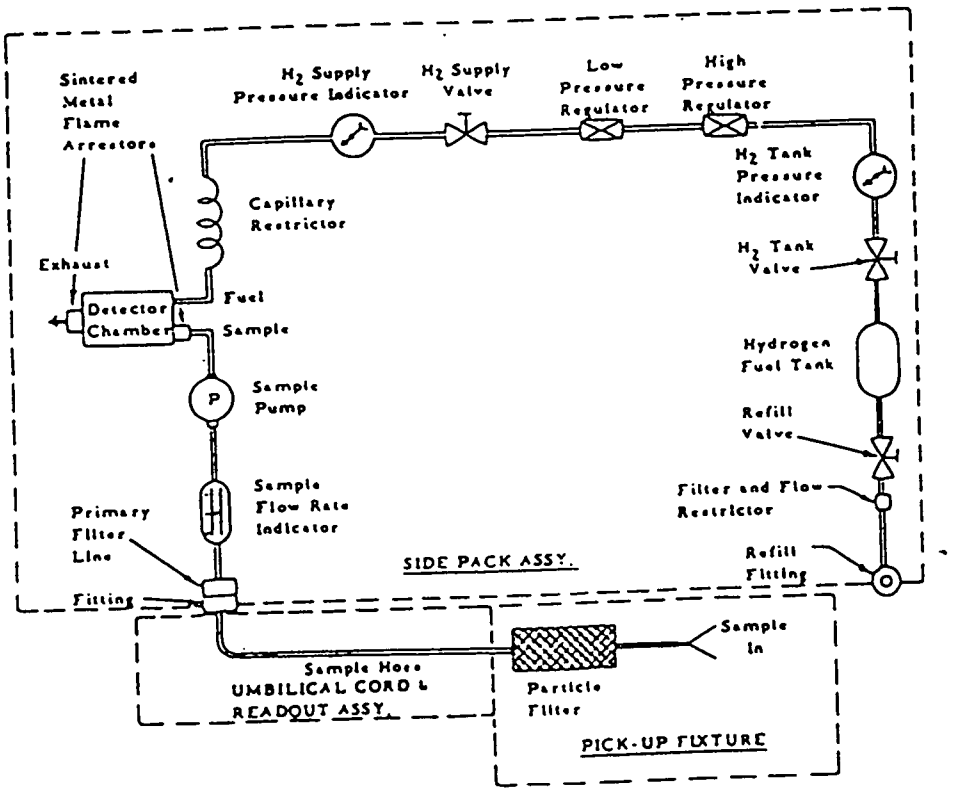


FIGURE 6-1. Flow Diagram - Gas Handling System

6.2.1.2 PARTICLE FILTERS

A particle filter is located in each pickup fixture. One of these filters must be in the sample line whenever the instrument is in use. The Models OVA-88 and OVA-138 use a disposable cellulose filter which should be changed as often as required. The Models OVA-98, OVA-108, OVA-118 and OVA-128 use a porous metal filter which can be replaced or cleaned using the cleaning procedure in paragraph 6.2.1.1.

6.2.1.3 MIXER/BURNER ASSEMBLY FILTER

Another porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. See Side Pack Assembly drawing. This filter is used as the sample mixer and inlet flame arrestor in the chamber. This filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

Access to this filter for output surface cleaning is gained by simply unscrewing the exhaust port from the Preamp Assembly without removing the instrument from the case. The OVA-108, OVA-128 and OVA-138 instruments require removal of the safety cover prior to unscrewing the exhaust port. The Filter Assembly can now be seen on the side of the chamber (Preamp Assembly) and can be scrapped or cleaned with a small wire brush.

If filter replacement is required, install a new or factory rebuilt Mixer/Burner Assembly. In several OVA models, this requires removal of the Preamp Assembly.

6.2.1.4 EXHAUST FLAME ARRESTOR

A porous metal flame arrestor is located in the exhaust port of the detector chamber (Preamp Assembly). See Side Pack Assembly drawing. It acts as a particle filter on the chamber output and restricts foreign matter from entering the chamber. This filter may be cleaned, if required, by removing the exhaust port from the Preamp Assembly. The exhaust port is removed from the bottom of the case without case removal. Note that the filter is captive to the exhaust port on the Models OVA-108, OVA-128 and OVA-138. Clean the filter with a solvent or detergent but ensure that it is dry and any solvent completely baked out at 120°F before reinstalling.

6.2.2 PICKUP FIXTURES

The pickup fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particle matter. If a solvent is used, the fixture should be subsequently cleaned with detergent and baked out at 120°F to eliminate any residual hydrocarbons from the solvent.

6.2.3 SEAL MAINTENANCE - CYLINDER ASSEMBLY

6.2.3.1 H2 TANK, H2 SUPPLY AND REFILL VALVES

After some time, the teflon washers under each valve packing nut can "cold flow" (move with pressure) and allow hydrogen to leak. Leakage can be determined by using Leak-Tec, Snoop or a soap solution around the valve stems. This leakage can usually be stopped by tightening the compression nut (adapter) as outlined below. See Side Pack Assembly and Cylinder Assembly drawings.

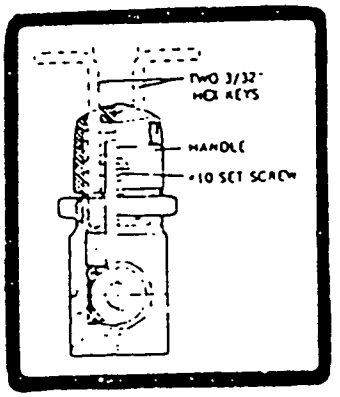
1) Remove instrument from the case by unlocking the four (4) 1/4 turn fasteners on the panel and removing the exhaust safety cover (if included), exhaust port and refill cap nut. Be sure refill valve is closed before removing refill cap nut.
2) Remove the valve knob screw and knob.
3) Loosen the panel nut with a 3/4" wrench.
4) The valve compression nut is located just under the panel. Tighten the compression nut—usually not more than 1/4 turn.

This compression is against soft material and only a small amount of force is necessary to sufficiently compress the teflon washers. If, after tightening, leakage still occurs, it would be advisable to replace the two teflon washers, as follows:

1) Drain hydrogen system slowly and to the extent necessary to work on the leaking valve(s). Observe safety precautions (see Section 5). There should be no potential igniters in the area.
2) Disconnect the capillary tube from the manifold at low pressure gauge (H2 Supply Pressure).
3) Remove all three (3) knob screws and knobs.
4) Remove the three (3) panel nuts and washers.
5) Carefully remove the tank assembly from the panel. NOTE: If OVA has GC Option installed, the GC valve assembly must be loosened or removed in order to remove the tank assembly from the panel.
6) Remove the compression nut on the valve that is not sealing properly. Remove the stem by unscrewing it from the valve body. Observe the sandwich of metal and teflon washers and note their order.
7) Visually check the Kel-F seat on the stem for cracks or foreign material. Wipe clean, if necessary, with a lint free cloth (no solvents or oils) and replace if damaged.
8) Remove the washers and replace the teflon washers (the factory procedure is a light wipe of hydrocarbon free silicone grease).
9) Replace the stem assembly in the valve body and tighten lightly.
10) Push the washers down into the compression area in the same order as noted upon removal. Replace the compression nut and tighten snugly.
11) Close the low pressure valve and fill the tank assembly. Check valves for leaks. Tighten again, if necessary, and reassemble the unit.

6.2.3.2 REFILLER VALVE PACKING ADJUSTMENT

Adjustment for the valve on the refiller can be made by loosening the set screw with a 3/32" hex key, so that the handle turns freely on the stem. Insert two (2) 3/32" hex keys through the holes provided in the handle and turn until they engage the holes in the packing adjuster. Then tighten the packing by turning the handle.



AIR SAMPLING SYSTEM MAINTENANCE

GENERAL

ntial problem associated with the OVA instru- is that leaks can develop in the air sample pump- stem. These leaks can result in either dilution or ample, causing low reading of vapor concentra- slow response time.

TESTING FOR LEAKS

OVA's a re equipped with a flow gauge, which a method to check for air leaks. Assemble the robe selected for use to the readout assembly nen position the sidepack vertically so the flow may be observed. Cover the end of the pickup th your finger and observe that the ball in the ge goes to the bottom, indicating no air flow (if as slight chatter while on bottom, this is accep- . Cover the center of the chamber exhaust port r thumb and again observe the ball going to the Another simple check is to expose the pickup to cigarette smoke or a light vapor (butane) and ve that the meter responds in approximately 1.5 - onds. It should be noted that slow meter e may also indicate a restriction in the air ing system.

LEAK ISOLATION

e of the ball to go to the bottom when the inlet is indicates a leak in the system between the pro- d the pump inlet or the inlet check valve. To isolate blem, remove parts, one at a time, and again f the air inlet. Remove the pickup probe(s) and e air inlet at the Readout Assembly. If the ball to the bottom, check that the "readout to probe" asher is in place and replace the probes, holding ack against this seal while tightening the nut. k, and if leakage is still present, it is probably in robe (pickup fixture), which should be repaired or ced.

age is indicated as being past the readout han- en the connection to the sidepack is tight, nnect the sample line at the fitting on the sidepack over this inlet with your finger. If the flow gauge ball goes to the bottom, the problem should be a leak in the umbilical cord/Readout Assembly, which should be investigated and repaired. There is also the possibility of a leaking check valve in the pump which would not show up on this test. If the leakage is not found in the umbilical cord, it is most likely in the pump check valve which should be repaired or replaced.

If the ball does not go to the bottom, the leak will be either in the flow gauge or it's connecting tubing. Visually check that the tubing is connected and if so, the flow gauge should be repaired or replaced. Check the "O" ring installation in the sample inlet connector (Fitting Assembly).

As an alternate approach, leaks on the inlet side of the pump can be detected by using alcohol on a "Q" Tip and lightly swabbing the connections one at a time or by directing organic vapor or smoke at the potential leakage points and observing the meter response or audible alarm.

Leaks (beyond the pump) are easier to locate, as any of the commercially available leak detection solutions can be used. Cover the exhaust port, which will place the exhaust system under pressure, and check each connection, one at a time. Replace the teflon tubing or retape the threaded connections with teflon joint tape. Check the igniter and Mixer/Burner Assembly where they screw into the detector, the high voltage terminal screw on the side of the Mixer/Burner and exhaust port itself. If after these checks, the flow gauge ball still will not go to the bottom with the exhaust blocked, the problem is likely a leaking exhaust check valve in the pump, which should be repaired or replaced.

6.2.5 CONTAMINATION CONTROL AND MAINTENANCE

6.2.5.1 GENERAL

On occasion, the background reading of the OVA may be relatively high under normal ambient conditions. Ambient background readings will vary somewhat depending on the geographical location where the instrument is being used. However, the background reading normally should be in the range of 3 to 5 ppm as methane. The acceptable background reading consists of 1 to 1-1/2 ppm of methane which is present in the normal air environment. In addition to the measurement of a normal methane background, there will normally be 2 to 4 ppm of equivalent methane background caused by acceptable levels of contamination in the hydrogen fuel and/or hydrogen fuel handling system resulting in a total equivalent methane reading of 3 to 5 ppm in clean air.

If the background reading goes above 5 ppm to 6 or 7 ppm, this is normally still acceptable since any measurement is additive to that background reading, i.e., 2 ppm on top of 5 or 2 ppm on top of 7 provides the same differential reading, however, the lower background is obviously desirable.

The background reading on the linear OVA's is zeroed out or nulled out—even though in reality the background still exists. The background reading on the

linear OVA's is measured by zeroing the meter with the flame out and noting the meter indication after the flame is on. However, on the logarithmic scaled OVA's the background reading is observed on the meter at all times. This is considered desirable since it assures the operator that the instrument is, in fact, operating properly. The background reading on the OVA's serves as a low level calibration point since it does represent the measurement of ambient levels of methane in the air, which are extremely stable and predictable any place in the world.

The cause for a high background reading is usually associated with contamination in the hydrogen fuel system. This will, of course, cause a background reading since this is the function of the basic detector "to measure contamination entering the detector chamber". In addition, contamination present in the hydrogen will many times leave a small unobservable deposit on the burner face which can continue to generate a background reading when the detector is in operation and the burner assembly is heated.

Another possible cause of contamination is the mixer/burner assembly when the contamination is trapped in the porous bronze sample filter. This is not a common problem and usually only happens when an unusually high level of contaminant is drawn into the assembly. Another possible cause of high background reading is contamination someplace in the air sample line to the detector. This is also uncommon but can be the source of the problem.

NOTE

OVA's that include the Chromatograph Option installed can also have an indication of high background related to saturation or contamination of the activated charcoal filter, which is in the line during chromatograph analysis, or of the column which is in the hydrogen line at all times.

6.2.5.2 ANALYSIS AND CORRECTION

Prior to analyzing the problem, the OVA should be checked for proper electronic operation. Check logarithmic instruments for proper high and low calibration points and for proper gas selector operation (see Section 4). On logarithmic OVA's, check Gas Selector by turning to 500 and observing the flame-out alarm comes on as the needle goes below 1 ppm. It should be ensured that the instrument is calibrated to methane as referenced.

If, after checking that the OVA is properly calibrated, the background is still higher than normal for ambient conditions, the following procedure should be followed to isolate the cause of the problem.

1) Let the OVA run for a period of time (15 to 30 minutes) and see if the background level decreases as a function of time. The background could go down and stay down as a result of clearing line contamination which is removable simply by the normal flow of air through the sample line.
2) Take a reading in a known, relatively clean air environment. Normally, outside air environment is clean enough to assess by comparison whether the background reading is internal to the instrument or is present in the laboratory, office or location where the instrument is being used.
3) If the OVA includes the Gas Chromatograph Option, depress the sample inject valve so that the activated charcoal is in the line and observe whether the background reading goes down and stays steady after the elution of the air peak. The reading should always go down or stay the same but never be a higher background reading with the sample valve depressed, since the charcoal filter will take out any trace elements of organic vapors in the air heavier than a C_2. If another activated charcoal filter is available, this may be attached to the end of the probe to scrub the air so that a clean air sample would be going to the detector. The external activated charcoal can be used on any instrument, with or without chromatograph, for providing a clean air sample to assess background level.
4) If background still stays up and cannot be reduced by any of the previous steps, the safety cover (if included) and the exhaust port on the detector chamber (Preamp Assembly) on the bottom of the case should be removed and the Mixer/Burner Assembly scraped or brushed with a small wire brush. (Reference paragraph 6.2.1.3.) This will remove any small quantities of contamination that are on the Mixer/Burner Assembly which could be the source of the background vapor. After cleaning the face of the burner and tube, replace the exhaust port and safety cover (if included) and reignite the OVA. If contamination on the burner face was the cause, the problem should be immediately resolved and the ambient background will drop to an acceptable level.
5) If the background is still present, place your finger over the inlet of the probe so as to reduce the flow of air to the detector chamber. Reduced flow rate may be observed either on the sample flow gauge or can normally be observed by the sound of the pump motor.
6) If the background drops immediately in response to the reduced flow of air to the chamber, this is an indication that the contamination is in the air sample line. Therefore, the various parts of the sample flow line such as pickup probes, umbilical cord to the instrument, etc., should be investigated by the process of elimination to see if the contamination can be isolated.
7) Serious contamination in the air sample line is very uncommon. However, if very large doses of very heavy compounds are sampled, there is a possibility of a residual contamination which would eventually clear itself out but may take a considerable period of time. A typical cause for the high background from the sample line is a

contaminated Mixer/Burner Assembly. See paragraph (4) above for cleaning procedure. If heavy contamination of the Mixer/Burner is still indicated by a high background, replace the Mixer/Burner Assembly. In several OVA models, this will require removal of the Preamp Assembly. The old Mixer/Burner Assembly should be either discarded or returned to the factory for cleaning and rebuilding.

8) In the event there is contamination in the pump or other internal parts of the sample flow lines which cannot be removed, the sample flow components would have to be disassembled and cleaned. This is normally a factory type operation. However, the components such as the pump can be replaced in the field along with any contaminated tubing in the sample lines.

9) High background readings on OVA's which include the Gas Chromatograph Option can be caused by other sources of contamination. If the charcoal in the charcoal filter mounted on the panel of the instrument is contaminated or saturated, contaminated air would be supplied to the detector and raise the ambient level background. To check for this, the charcoal filter cartridge can be removed from the panel and either a bypass tube put between the two connectors or the charcoal can be removed from the charcoal cartridge and the cartridge refilled with clean activated charcoal. This would determine if the charcoal was the source of the background reading. It is possible that an apparent high background reading could be due to contamination in the column that is on the instrument. This background could be caused by compounds that are slowly eluting from a column which has become contaminated. The easiest way to check for column contamination is to replace the column with a known clean column or a short empty piece of column tubing and see if the high background reading drops.

10) If all the above steps do not correct the high background problem, the cause will normally be contamination in the hydrogen fuel system.

ntamination in the hydrogen fuel system is usually e direct result of contamination in the hydrogen gas or contamination introduced during the filling ation. Filling hose contamination can be caused by ng the hose in a contaminated area.

o remove contamination from the hydrogen fuel tem, it should be purged with hydrogen. Effective ing of the hydrogen system is accomplished by nnecting the capillary tube fitting which attaches to the manifold block which has the low pressure uge (H2 Supply Pressure Gauge and H2 Supply e). This disconnects the capillary tubing from the ogen line so that hydrogen may be purged at a sonable rate from the tank assembly through the ulators, gauges and valves. After disconnecting the lary, the hydrogen tank can be filled in the normal manner. The tank valve and H2 supply valve can then be opened which will bleed the hydrogen from the tank through the H2 fuel system purging out the contamination which is in vapor form. There is the possibility that contamination has been introduced into the hydrogen fuel system which is not readily purged out by the hydrogen gas but this is unlikely. After purging with clean hydrogen, approximately two or three times, the capillary tube should be reconnected and the background again checked. Five or ten minutes should be allowed before assessing the background reading, since contaminated hydrogen may still have been trapped in the capillary tube.

If another tank assembly in a clean instrument is available, the fuel system from the clean instrument can be connected to the contaminated instrument to absolutely verify that it is or is not in the hydrogen fuel supply system. The interconnection should be made to the capillary tube of the contaminated instrument.

6.2.6 FUSE REPLACEMENT

This paragraph applies only to the standard (non-certified) OVA's. There are two (2) overload fuses incorporated in the Battery Pack Assembly, one is a 3AG-1 AMP Slo-Blo in the power line to the pump and igniter and the other a 3AG-1/4 AMP in the power line to the electronics. Both fuses follow the current limiting resistors which provide primary short circuit protection. However, in the event of an excessive overload, the fuses will open and prevent overheating of the current limiting resistors. It should be pointed out that the 1 AMP Slo-Blo fuse will blow in approximately 8 to 12 seconds if the igniter switch is kept depressed. Normal ignition should take place in not more than 6 seconds. Therefore, do not depress igniter button for more than 6 seconds. If ignition does not occur, wait 1 to 2 minutes and try again. If the required 1 AMP Slo-Blo fuse cannot be readily obtained, replace temporarily with a 3 AMP-3 AG standard fuse.

6.3 TROUBLE SHOOTING

Table 6-1 presents a summary of recommended field trouble shooting procedures. If necessary, the instrument can be easily removed from the case by unlocking the four (4) 1/4 turn fasteners on the panel face and removing the refill cap and exhaust port. The battery pack is removed by taking out the four (4) screws on the panel and disconnecting the power connector at the battery pack.

6.4 FACTORY MAINTENANCE

To ensure continuous trouble-free operation, Century recommends a periodic factory maintenance, overhaul and recalibration. The recommended schedule is every six (6) to nine (9) months. This maintenance program includes replacement of plastic seals and parts as required, pump overhaul, motor check, new batteries, sample line cleaning, H2 leak check, recalibration, replacement of plastic hose as required, and detailed examination of the unit for any other required maintenance and repair.

he recommended procedure for maintenance and beyond the scope of this manual is to send the ete instrument or subassembly to the Century y for repairs. The assemblies will be handled ex-litiously for rapid turn-around.

FIELD MAINTENANCE

hough not recommended, where field intenance beyond that described herein is con-od essential, the assembly drawings, parts lists chematics in Appendix "A" will be of assistance.

RECOMMENDED SPARES

entury does not recommend that spares be main-d for its instruments. However, if the instrument is used in a remote area or spares are desired for er reasons, the following list should be used as a ide.

RECOMMENDED SPARES

m	Description	Part No.	Recommended Quantity					
			Standard			Approved		
			88	98	118	108	128	138
	Igniter	510027-1	2	2	2			
1	Igniter	510461-1				2	2	2
	Pump Valve	510067-3 (10/pkg.)	1	1	1	1	1	1
	Pump Diaphragm (Buna-N)	510091-1	1					1
	Pump Diaphragm (Teflon)	510063-1		1	1	1	1	
	Cup, Filter (3/8 OD, SS)	510318-1 (5/pkg.)	1	1	1	1	1	1
	Mixer/Burner Assy	510557-2	1					
	Mixer/Burner Assy	510557-1		1	1			
	Mixer/Burner Assy	510513-1				1	1	1
	Wafer, Teflon, H_2 Valve	510160-1 (10/pkg.)	1	1	1	1	1	1
1	Washer, Brass, H_2 Valve	510160-2 (10/pkg.)	1	1	1	1	1	1
	Exhaust Port Assy	510425-1	1	1	1			
	Exhaust Port Assy	510530-1				1	1	1
	Battery Pack Assy	510070-1	1	1	1			
5	Battery Pack Assy	510542-1				1	1	1
	Sample Line Assy	510316-1		1	1	1	1	1
	Particle Filters	510114-1	1					1
	Particle Filters	510116-1		1	1	1	1	

TE: Unit quantity is each unless otherwise noted.

TABLE 6-1

TROUBLE	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator. Nominally 2 units on flow gauge. (See also 6 below and refer to paragrapn 6.2.4)	a) Check primary filter in sidepack and particle filters in the pickup assembly. b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove Readout Assembly, remove primary filter, etc. c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output, chamber input, etc. Note: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when trouble shooting.	Replace or clean filter if clogged. (See paragraph 6.2.1) Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required. If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace.
2) H2 flame will not light. (See also 8 below)	a) Check sample flow rate (see 1 above). b) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE Button is depressed. c) Check for rated H2 Supply Pressure. (Listed on calibration plate on pump bracket.) d) Check H2 flow rate by observing the PSI decrease in pressure on the H2 Tank Pressure gauge. The flow rate should be about 130 PSI decrease in pressure per hour. (Approximately 12 cc/min. at detector.) On instruments with GC Option, disconnect column and measure H2 flow rate with a bubble meter. e) Check all H2 plumbing joints for leaks using soap bubble solution. Also, shut off all valves and note pressure decay on H2 tank gauge. It should be less than 350 PSIG per hour. f) Check to see if H2 supply system is frozen up by taking unit into a warm area.	If sample flow rate is low, follow procedure 1 above If igniter does not light up, replace the plug. If igniter still does not light, check the battery and wiring If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap. The normal cause for H2 flow restriction would be a blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the H2 Supply Pressure by one-half or one PSI. If flow rate cannot be compensated for, replace capillary tubing. Repair leaking joint. If there is moisture in the H2 supply system and the unit must be operated in subfreezing temperatures, purge the H2 system with dry N2 and ensure the H2 gas used is dry.

check battery level by connecting to charger

If in dead zone, battery may not have enough power to light flame. (need will clean battery, more)

	g) Remove exhaust port and check for contamination. (See Figure 6-2.)	If the chamber is dirty, clean with ethyl alcohol and dry by running pump for approximately 15 minutes. If H2 fuel jet is misaligned, ensure the porous metal flame arrestor is properly seated.
	h) Check spacing between collecting electrode and burner tip. Spacing should be 0.1 to 0.15 inches.	Adjust by screwing Mixer/Burner Assembly in or out. This spacing problem should only occur after reassembling a Mixer/Burner Assembly to a Preamp Assembly.
3) H2 flame lights but will not stay lighted	a) Follow procedures 2 (a), (c), (d), (e), (g) and (h) above. Also refer to 5 below.	
4) Flame-out alarm will not go on when H2 flame is out	a) Check instrument calibration setting and GAS SELECT control setting. Refer to paragraphs 2.3.1.2 and 2.3.2.	Readjust as required to proper setting. Note that on linear OVA's the flame-out alarm is actuated when the meter reading goes below zero. On logarithmic OVA's, the alarm is actuated when the signal level goes below 1 ppm methane or equivalent.
	b) Remove exhaust port and check for leakage current path in chamber (probably moisture or dirt in chamber).	Clean contamination and/or moisture from the chamber using a swab and alcohol, dry chamber by running pump for approximately 15 minutes.
	c) If above procedures do not resolve the problem, the probable cause is a malfunction in the preamp or power board assemblies.	Return preamp chamber or power board assembly to the factory for repair.
	d) Check volume control knob is turned up.	Adjust for desired volume.
5) False flame-out alarm. (Applies to linear OVA's)	a) Flame-out alarm is actuated on linear instruments when signal goes below electronic zero (eventhough flame is still on). This can be due to inaccurate initial setting, drift or a decrease in ambient concentration. Verify if this is the problem by zeroing meter with flame out and reigniting. (See paragraph 2.3.2)	When using the X1 range, adjust meter to 1 ppm rather than zero. See paragraph 2.3.2. Be sure instrument has been zeroed to "lowest expected ambient background level".
6) Slow response time, i.e., time to obtain response after sample is applied to input. (Refer to paragraph 6.2.4)	a) Check to ensure that probe is firmly seated on the rubber seal in the readout assembly.	Reseat by holding the probe firmly against the rubber seal and then lock in position with the knurled locking nut.
	b) Check sample flow rate per procedure 1 above.	See 1 above.

7) Slow recovery time, i.e., too long a time for the reading to get back to ambient after exposure to a high concentration of organic vapor.	a) This problem is normally caused by contamination in the sample input line, requiring pumping for a long period to get the system clean of vapors again. Charcoal in the lines would be the worst type of contamination. Isolate through the process of elimination. (See 1 (b)). b) Check flame chamber for contamination.	[redacted] as required. Clean as required.
8) Ambient background reading in clean environment is too high. (Refer to paragraph 6.2.5)	a) An ambient background reading can be caused by hydrocarbons in the H2 fuel supply system. Place finger over sample probe tube restricting sample flow and if meter indication does not go down significantly the contamination is probably in the H2 fuel. b) An ambient background reading can be caused by a residue of sample, building up on the face of the sample inlet filter. If the test in 8 (a) above produces a large drop in reading, this is usually the cause. c) An ambient background reading can also be caused by hydrocarbon contamination in the sample input system. The most likely cause would be a contaminant absorbed or condensed in the sample line. Note: It should be emphasized that running the instrument tends to keep down the buildup of background vapors. Therefore, run the unit whenever possible and store it with the carrying case open in clean air.	Use a higher grade of hydrocarbon free hydrogen. Check for contaminated fittings on filling hose assembly. Remove the exhaust port (it is not necessary to remove instrument from case), use small wire brush from the tool kit or a knife blade and lightly scrub surface of sample inlet filter. Clean and/or replace the sample input lines. Normally the lines will clear up with sufficient running
9) Pump will not run	a) Check 1 AMP Slo-Blo fuse on the battery pack cover. NOTE: Certified OVA's do not have fuses.	Replace fuse. IMPORTANT. Note that fuse is a Slo-Blo type. If fuse continues to blow when igniter switch is closed, check igniter for short circuit. If igniter is not the problem, there is a short in the wiring or pump motor. Return OVA to factory or authorized repair facility
10) No power to electronics but pump runs	a) Check 1/4 AMP fuse on the battery pack cover. NOTE: Certified OVA's do not have fuses.	Replace fuse. If fuse continues to blow, there is a short in the electronics assembly. Return OVA to factory or authorized repair facility.
11) No power to pump or electronics.	a) Place battery on charger and see if power is then available. Recharge in a non-hazardous area only.	If power is available, battery pack is dead or open. Recharge battery pack. If still defective, replace battery pack. Reference paragraph 2.7

APPENDIX D

REQUESTED ANALYSIS METHODS

WARZYN

Alkalinity (H_2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for Alkalinity in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered. Results will be reported as mg/L $CaCO_3$.

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

214 groundwater samples - low level and 8 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-90]

Estimated date(s) of collection:

Estimated date(s) and method of shipment: Daily by overnight carrier

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

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

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

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

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

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

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

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

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

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

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

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

Name of sampling/shipping contact: Mike Linskens

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (+% or Conc.)
Alkalinity	2 mg/l for low level	± 2 mg/l for Conc. < 20 mg/l $CaCO_3$
	20 mg/l for high level	± 10% for Conc. > 20 mg/l

NOTE: These are minimum requirements. Report actual detection limits used based on allowable methodologies.

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

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

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanlyze samples.

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

lease return this request to the Sample Management Office as soon as possible to expedite rocessing of your request for special analytical services. Should you have any questions r eed any assistance, please call the Sample Management Office.

TOC (H^2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for TOC in waters (surface waters, groundwaters, drinking waters, leachates, etc.) samples will be unfiltered.

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

214 groundwater samples - low level and 8 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-86]

Estimated date(s) of collection: _____

Estimated date(s) and method of shipment: Daily by overnight carrier.

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

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

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

EPA Method 415.1 (combustion or oxidation).

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

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

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

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

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

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

11. Name of sampling/shipping contact: Mike Linskens

Phone: (608) 273-0440

DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
TOC	2 mg/l	Difference in duplicate results should not exceed ± 10% for concentrations >20 mg/l or 2 mg/l for concentrations less than 20 mg/l.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

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

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

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

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

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

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

TKN (H_2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for TKN in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered.

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

214 groundwater samples - low level and 8 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-87]

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: DAily by overnight carrier

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

Laboratories shall report results within 30 days after receipt of samples

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

1) EPA Method 351.2 (Colorimetric, Block Digestor, AA II)
2) EPA Method 351.3 (Calorimetric, Titrimetric, or Potentiometric) (NOTE: For Method 351.3 the micro-Kjeldahl technique is not acceptable.) Samples will be preserved in the field using H2SO4 (1ml/L) to pH<2, samples will be stored at 4°C until analysis and validation of results.

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

For all Methods: Analyze samples within 28 days after collection. Check the sample pH (wide range pH paper). If the pH>2, contact CPMS, CRL for instructions. Use nicotinic acid for the control standard. Use an organic nitrogen compound for the matrix spike. Use only the Methods specified in item 7. Metho 351.3 requires distillation separation, prior to all final ammonia measurements.
For Method 351.3: Use only the Colorimetric method for samples containing less than 1 mg N/l.
For Colorimetric Methods (351.2 and 351.3): Use at least five calibration standards (including a zero concentration standard). Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Potentiometric Method (351.3): Use at least four calibration standards. Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Titrimetric Method (351.3): Standardize the titrant each day. Include records of indicator blank.

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

Identify the test procedure and options used. Provide bench records and all records of calibration, analyses, and calculations for standards, samples blanks, any titration indicator blanks, duplicates, spikes, controls, etc. Include absorbances, peak heights, responses, concentrations, etc. for each measurement. Include digestion logs showing sample volumes and dilutions for all samples. Identify organic nitrogen compound used for matrix spikes. Records must be legible and sufficient to recalculate all concentrations and QA audit results. Provide photocopies of all instrument readouts (i.e. stripcharts, print-outs, etc). Report results as mg N/l. Identify the compound used for the matrix spike.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "tru or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

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

11. Name of sampling/shipping contact: Mike Linskens

Phone: (608) 273-0440

DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
TKN =	0.1 mg N/l	Duplicate sample results must agree within 0.1 mg/l for concentrations <1 mg/l and within 10% for concentrations > or = to 1 mg/l
NOTE: These are minimum requirements. Report the actual detection limit used based on allowable methodology options.		

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

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Control standards (Nicotinic Acid)	one per set	70 - 110% recovery
Matrix spike*	one per group of 10 or fewer samples	85 - 115% recovery
Lab duplicate	" "	± (10% or 0.1 mg N/l)
Lab blank	" "	± 0.1 mg N/l
Calibration verification Standard	" " and at the end of the set	90 - 110%
1 set of EPA QC nutrient reference samples conc. 3 and 4.	one per set	85 - 115%

*Matrix spike concentration will be greater than 30% of the sample concentration but will not exceed the highest calibration standard. Matrix spikes will be prepared from an organic nitrogen compound.

ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

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

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

$NO_3 + NO_2$ (H^2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for $NO_3 + NO_2$ in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered.

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

214 groundwater samples - low level and 6 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-85]

Estimated date(s) of collection:

Estimated date(s) and method of shipment: Daily bu overnight carrier

Number of days analysis and data required after laboratory receipt of samples:
Laboratory should report results within 30 days of receipt of samples.

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

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

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

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

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

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

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

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

11. Name of sampling/shipping contact: Mike Linskens (608) 273-0440

I. DATA REQUIREMENTS

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

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

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

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

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

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

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

SO_4 (H^2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for sulfate in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered.

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

214 groundwater samples - low level and 8 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-89]

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:
Laboratory should report the results within 30 days upon receipt of samples.

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

1. EPA Method 375.2 (Colorimetric Methylthmol Blue) - 1983 ed.
- Note: This method requires 0.75 mg/l SO_4 in Dilution Water(See Reagent Section 6.

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

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

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

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

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

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

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

11. Name of sampling/shipping contact: Mike Linskens

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Sulfate	3 mg/l	Method 375.2: Differences in duplicate sample results are to be < 3 mg/l for concentrations < 50 mg/l, and < 10% for concentration > 50 mg/l. Method 426 C: Differences in duplicate sample results are to be ≤ 2 mg/l for concentrations ≤ 20 mg/l and ≤ 10% for concentrations > 20 mg/l in aliquot tested.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

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

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85-115%
Lab Duplicate	" "	± (10% or 3 mg/l) for Method 375.2
Lab Blank (0 mg/l SO_4)	" "	± (10% or 2 mg/l) for Method 426C
Lab Blank (10 mg/l SO_4)	" "	< 5 mg/l - Method 375.2 -2 to +2mg/l-Buffer B of Method 426C or 8 to 10mg/l - Buffer A of Method 426C
Calibration Verification Standard	1 per group of 10 samples and at end of sample set	90 - 110%
1 Set of EPA QC Mineral Reference Samples	once per sample set	85-115% for each concentration.

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

II ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

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

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

Designation: D 422 – 63 (Reapproved 1972)[1]

Standard Method for PARTICLE-SIZE ANALYSIS OF SOILS[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

NOTE—Section 2 was added editorially and subsequent sections renumbered in July 1984.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-μm), or No. 200 (75-μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm.

2. Applicable Documents

2.1 *ASTM Standards:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give

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

Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

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

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

[4] *Annual Book of ASTM Standards*, Vol 14.01.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.

NH_3 (H^2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for NH_3 in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered. Results will be reported as mg/L N.

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

214 groundwater samples - low level and 6 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-84]

NH3

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:
Laboratory should report results within 30 days of receipt of samples.

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

1) EPA Method 350.1 (Automated Phenate), or

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

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

The working concentration range of Method 350.1 Auto Analyzer should be 0.1 to 10 mg/l NH3-N or lesser concentration.

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

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

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

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

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

11. Name of sampling/shipping contact: Mike Linskens

Phone: (608) 273-0440

I. DATA REQUIREMENTS

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

GENERAL STATEMENT

I. QC REQUIREMENTS _ Do not use designated field blanks for QA Audits.

a) For Method 350.1

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

b) For Method 350.3

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

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

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

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

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

Cl (H_2O)

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

SAS Number

SPECIAL ANALYTICAL SERVICES Approved for Scheduling
Client Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region/Client: Region V, Wausau NPL Site

B. RSCC Representative: Dennis Wesolowski

C. Telephone Number: 312-886-1971

D. Date of Request: _____

E. Site Name: Wausau NPL Site

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

1. General description of analytical service requested: Analysis for chloride in waters (surface waters, groundwaters, drinking waters, leachates, etc.) Samples will be unfiltered.

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

214 groundwater samples - low level and 8 surface water samples - low level

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

WDNR lead remedial investigation

[jap-750-97]
[jap-750-88]

timated date(s) of collection:

mated date(s) and method of shipment: Daily bu overnight carrier

mber of days analysis and data required after laboratory receipt of samples:

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

alytical protocol required (attach copy if other than a protocol currently used in program):

1. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA II), 1983 ed.

NOTE: A Region V CRL Auto-Analyzer manifold (0-20 mg/l) is attached for modification of Method 325.2 and to correct errors in Method 325.2.

or

2. ASTM D 512C-81 (Colorimetric, Manual Ferricyanide).

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

pecial technical instruction (if outside protocol requirements, specify compound ames, CAS numbers, detection limits, etc.): Prepare all standards , reagents, blanks, etc. with ASTM Type II reagent water or equivalent, calibration standards will be prepared daily from stock solutions. Use working concentration range or standard curve between 0-20 mg/l or less. Calibration curves must contain at least 5 points (including a zero concentration standard). Dilute and reanalyze any samples with concentrations greater than highest standard. Remove any large amounts of turbidity prior to sample analysis (see Section 7.1 of Method 325.2).
Use only the specified methods. No others are allowed.

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

Bench records tabulating the order of calibration standards, verification and control standards, samples, blanks, matrix spikes, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of worksheets used to calculate results. A photocopy of instrument readouts, i.e. stripcharts, printer tapes, etc., must be included with all results. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

h (use additional sheets or attach supplementary information, as needed):

me of sampling/shipping contact: Mike Linskens

Phone: (608) 273-0440

DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
chloride	0.5 mg/l	Differences in duplicate sample results are to be < or = to 0.5 mg/l for concentrations ≤ 5 mg/l and < or = to 10% for concentrations exceeding 5 mg/l.
NOTE: These are minimum requirements. Report actual detection limit used based on allowable methodology options.		Report chloride concentrations to the nearest 0.1 mg/l between 0 and 20 mg/l.

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

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike*	1 per group of 10 or fewer samples	85 - 115% Recovery
Lab duplicate	" "	+ or -(10% or 0.5 mg/l)
Lab blank	" "	< 0.5 mg/l
Calibration verification standard	" "	90 - 110% Recovery
1 set of EPA QC Mineral Reference samples - 2 concentration levels.	1 per sample set	85 - 115% Recovery

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

ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

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

Return this request to the Sample Management Office as soon as possible to expedite ssing of your request for special analytical services. Should you

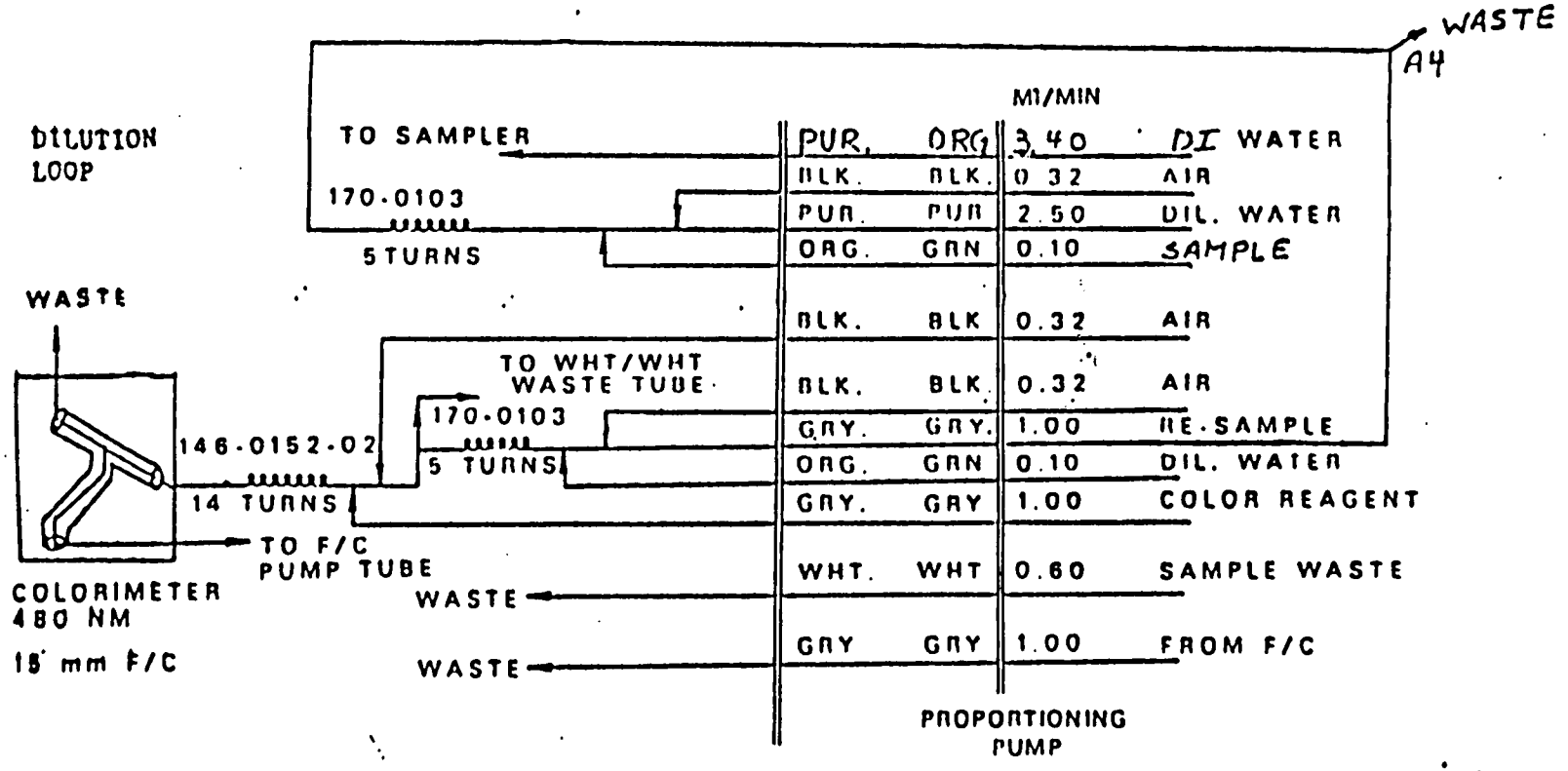


FIGURE 1
CHLORIDE MANIFOLD AA 11 0-200 mg Cl/l

To analyze samples in the range of
0 - 20 mg Cl/l, bypass the dilution loop.

Calcium + Magnesium, Iron
Sodium, Potassium

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

SAS Number _____

SPECIAL ANALYTICAL SERVICES
Regional Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region and Site Name: Region V - Wausau NPL Site

B. Regional Representative: Dennis Weslowski

C. Telephone Number: ( )

D. Date of Request:

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

1. General description of analytical service requested: Calcium and magnesium iron, sodium and potassium of groundwater samples is to be determined.

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

Analysis is to be performed on 214 groundwater samples

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

WDNR - lead remedial investigation

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: delivered daily by Federal Express.

6. Approximate number of days results required after lab receipt of samples: Laboratory should report results within 30 days after receipt of samples.

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

Calcium (dissolved) EPA Method 215.1

Magnesium (dissolved) EPA Method 242.1

Iron (dissolved) EPA Method 2361

Sodium (dissolved) EPA Method 273.1

Potassium (dissolved) EPA Method: 258.1

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

Samples will be preserved with 5 ml/l 1:1 HNO_3. Groundwater samples will be filtered (in-field) through a .45 micron filter prior to analysis.

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

Copies of aall bench records for lab duplicates, matrix spikes, blanks, continuing calibration standards, calibration verification standard, and saamples with resulting concentrations will be provided with copies of any worksheets used to calculate results.

Calcium + Magnesium; Iron
Sodium, Potassium

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

Name of sampling/shipping contact: Brian Hegge

Phone: (608) 273-0440

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

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Calcium	0.01 mg/l	10% or 0.01 mg/l for conc. < 1.0
Magnesium	0.001 mg/l	10% or 0.001 mg/l for conc. < .10
Iron	0.03 mg/l	10% or 0.01 mg/l for conc. < 1.0
Sodium	0.002 mg/l	10% or 0.01 mg/l for conc. < 1.0
Potassium	0.01 mg/l	10% or 0.01 mg/l for conc. < 1.0

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Lab blank	2 for sets ≤ 10 1 per 10 for sets > 10	< detection limit
Lab duplicate	2 for sets ≤ 10 1 per 10 for sets > 10	10% or detection limit
Matrix spike*	2 for sets ≤ 10 1 per 10 for sets > 10	85 - 115% recovery
Continuing calibration standard	1 per 10 samples and end of set	85 - 115% recovery
Calibration verification STD (EPA QC reference sample)	1 per set	85 - 115% recovery

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

III. *Action Required if Limits are Exceeded:

Reanalyze. Contact Chuck Elly (312) 353-9087.

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

SAS Number _____

SPECIAL ANALYTICAL SERVICES
Regional Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region and Site Name: Region V Wausau NPL Site

B. Regional Representative: Dennis Wesslowski

C. Telephone Number: ( )

D. Date of Request:

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

1. General description of analytical service requested: Analysis for volatiles by GC/MS in surface water and groundwater with low sensitivity limits.

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

Analysis will be performed on 6 surface water samples and 214 groundwater samples to be considered low concentration.

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

MDNR lead remedial investigation

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: delivered daily by Federal Express.

6. Approximate number of days results required after lab receipt of samples: Laboratory will report results within 15 days of receipt of samples. 7 days for analysis

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

Organic analysis IFB WA85-J664

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

1. Exceptions to Organic IFB - Attachment 1
2. Required low sensitivity limits - Table 7.
3. Requirements for determining sensitivity limits: easily recognizable spectra for all compounds using 1.5 ug/l for VOA's.
4. Initial calibrations: %RSD for RFs should be <40 for each VOA before beginning analysis.
5. Continuing calibration: run daily calibration standard before running analysis. %D should be <25 for all compounds in VOAs. If some are greater than 25%, they should be reinjected. If still out, rerun 3 point curve.
6. If dilution of samples is required, results for both original and diluted sample should be reported.

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

All deliverables included in the IFB are required including instrument sensitivity determinations. The lab will notify the Region prior to diluting any sample. If Regional approval is given to dilute, all the data will be submitted; data dilution will be reported on separate OADS forms.

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

Name of sampling/shipping contact: Brian Hegge

Phone: (608) 273-0440

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

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
As listed (Table D-1)	As listed (Table D-1)	See Attachment 1

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Organics as in IFB	As in IFB	Attachment 1

III. *Action Required if Limits are Exceeded:

Reanalyze. Contact Chuck Elly (312) 353-9087 or Dennis Wesslowsk.

[jam-26-1]

TABLE D-1

(All Units are Micrograms/Liter)

PARAMETER	CAS #	METHOD DETECTION LIMIT IN REAGENT WATER
Benzene	71-43-2	1.5
Bromodichloromethane	75-27-4	1.5
Bromoform	75-25-2	1.5
Bromomethane	74-83-9	10
Carbon Tetrachloride	56-23-5	1.5
Chlorobenzene	108-90-7	1.5
Chloroethane	75-00-3	1.5
2-Chloroethyl Vinyl Ether	110-75-8	1.5
Chloroform	67-66-3	1.5
Chloromethane	74-87-3	10
Dibromochloromethane	124-48-1	1.5
1,1-Dichloroethane	75-34-3	1.5
1,2-Dichloroethane	107-06-2	1.5
1,1-Dichloroethene	75-35-4	1.5
trans,1,2-Dichloroethene	156-60-5	1.5
1,2-Dichloropropane	78-87-5	1.5
cis-1,3-Dichloropropropene	10061-01-5	2
trans-1,3-Dichloropropene	10061-02-6	1
Ethyl Benzene	100-41-4	1.5
Methylene Chloride (*)	75-09-2	1
1,1,2,2-Tetrachloroethane	79-34-5	1.5
Tetrachloroethene	127-18-4	1.5
Toluene (*)	108-88-3	1.5
1,1,1-Trichloroethane	71-55-6	1.5
1,1,2-Trichloroethane	79-00-5	1.5
Trichloroethene	79-01-6	1.5
Vinyl Chloride	75-01-4	10
Acrylein	107-02-8	100
Acetone (*)	67-64-1	10
Acrylonitrile	107-13-1	50
Carbon Disulfide	75-15-0	3
2-Butanone	78-93-3	10
Vinyl Acetate	108-05-4	10
4-Methyl-2-Pentancne	108-10-1	(3)
2-Hexanone	519-78-6	10
Styrene	100-42-5	1
m-Xylene	108-38-3	2
o-Xylene**	95-47-6	
p-Xylene**	106-42-3	2.5

*Common Laboratory Solvent - Blank Limit is 5x Method Detection Limit
**The o-Xylene and p-Xylene are reported as a total of the two

[cmj-400-54a]

ATTACHMENT 1

VOA - Increase sample volume up to 20 ml to meet quantitation limits.

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

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

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

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

All RFs must be ≥0.05.

NOTE: The IFB limits for the RPDs for the matrix spike/matrix spike duplicate results apply for all of the organics analyses.
For corrective action when surrogates are outside the SAS required recovery limits, see the IFB for re-extraction/re-analysis requirements.
*The surrogate and matrix spike amounts listed are the concentrations in the liter of the sample.

KDF/cmj/KDF
[cmj-400-54]

Total Organic Carbon

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

SAS Number _____

SPECIAL ANALYTICAL SERVICES
Regional Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region and Site Name: Region V - Wausau NPL Site

B. Regional Representative: Dennis Wesolowski

C. Telephone Number: (312) 886-1971

D. Date of Request: _____

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

1. General description of analytical service requested: Determination of total organic carbon in soil samples.

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

15 soil (concentrations unknown)

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

Superfund Remedial

4. Estimated date(s) of collection: _____

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

6. Approximate number of days results required after lab receipt of samples: Laboratory should report results within 30 days after receipt of samples.

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

Procedures will follow those described in:

Methods of Soil Analysis, Part 2, Second Edition

American Society of Agronomy, 1982 (See Attached)

Analysis wil be performed by wet combustion (Section 29-2.3) after pretreatment for inorganic carbon removal (Section 29-3.3.2).

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

Procedures are to be followed as described. Expected carbonate content ranges from 5 to >50% by weight.

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

Report all raw laboratory data and calculations. Report mass of soil tested on a dry weight basis and the volume and normality of soil used to neutralize carbonates. Report method of determining CO_2 generated by wet combustion and mass or titrant volumes as normality as appropriate.

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

See attached methods description

Name of sampling/shipping contact: Brian Hegge

Phone: (608) 273-0440

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

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Total Organic Carbon	0.05% (w/w)	10% or +- 0.05%

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Laboratory Blank	2 per sample set	10% RPD
Laboratory Duplicate	1 per 10 and least 2	10% RPD

III. *Action Required if Limits are Exceeded:

Reanalyze. Contact Chuck Elly (312) 353-9087.

[jam-26-5]

AGRONOMY

A Series of Monographs

The American Society of Agronomy (ASA) and Academic Press published the first six books in this series. Subsequent books were published by ASA alone, but in 1978 the associated societies, ASA, Crop Science Society of America (CSSA), and Soil Science Society of America (SSSA), published Agronomy 19. The books numbered 1 to 6 on the list below are available from Academic Press, Inc., 111 Fifth Avenue, New York, NY 10003; those numbered 7 to 22 are available from ASA, 677 S. Segoe Road, Madison, WI 53711.

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1. C. EDMUND MARSHALL: The Colloid Chemical of the Silicate Minerals, 1949
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3. K. D. JACOB: Fertilizer Technology and Resources in the United States, 1953
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METHODS OF SOIL ANALYSIS

Part 2

Chemical and Microbiological Properties

Second Edition

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Number 9 (Part 2) in the series

AGRONOMY

American Society of Agronomy, Inc.
Soil Science Society of America, Inc.
Publisher
Madison, Wisconsin USA

1982

gravimetry, an automated CO_2 analyzer (LECO no. 761-100) based on thermal conductivity measurements of the effluent gases is applicable to soil analysis (Tabatabai & Bremner, 1970). This system allows a single operator to analyze total C in 15 to 20 samples/hour. Alternatively, a titrimetric method was developed to allow estimation of both total C and ^{14}C in soil samples amended with ^{14}C compounds (Cheng & Farrow, 1976). A bypass valve and a 125-ml gas washing bottle (e.g., Corning 31760) are used in place of the CO_2 absorption bulb of Fig. 29-1. All CO_2 released by combustion is trapped in 50 ml of 0.5*N* NaOH followed by removal of 1 aliquot for liquid scintillation counting to quantify $^{14}CO_2$ and a second aliquot for titration with standard HCl to determine total C. The total C data obtained were comparable with a wet combustion procedure.

29-2.2.5 OTHER INSTRUMENTAL METHODS

The following section describes three additional commercial instruments for determining total C in soils. They were chosen to illustrate the principles involved in instrumenting total C analysis. The inclusion of the following three instruments does not imply that they are superior or inferior to others currently being marketed. As with all instruments, various evaluation procedures should be used to determine if the instrument selected is compatible with the types of samples requiring analysis.

29-2.2.5.1 Perkin-Elmer 240. The Perkin-Elmer 240 (Perkin-Elmer Corp., Instrument Division, Norwalk, Conn.) simultaneously measures C, H, and N using the principles employed in the traditional Pregl and Dumas procedures. A sample contained in a Pt boat is oxidized with O_2 at ~1,000°C for 2 min in a combustion tube in the absence of carrier gas (He) flow. After combustion, He flow is initiated and the CO_2, H_2O, and N_2 gases produced by combustion are passed over CuO to convert CO to CO_2 and Ag mesh (silver vanadate on Ag wool) to remove S and halogen gases. The gases then flow into a tube maintained at 650°C and packed with Cu granules between end plugs of Ag wool, where quantitative reduction of N oxides to N_2 occurs. The gases are brought to constant pressure and volume in a gas mixing chamber and then allowed to expand into the analyzer portion of the instrument. The analyzer consists of three thermal conductivity (TC) detectors connected in series and separated by two traps. The sequence of TC detectors and traps enabling quantification of H, C, and N is as follows:

1) TC detector 1 (output equals total gas composition).
2) Magnesium perchlorate trap to remove H_2O.
3) TC detector 2 (decrease in output from detector 1 is proportional to H content).
4) Soda asbestos plus $Mg(ClO_4)_2$ trap to remove CO_2.
5) TC detector 3 (decrease in output from detector 2 is proportional to C content).
6) The remaining gases in the sample are N_2.

All operations within the instrument are automatic.

29-2.2.5.2 Dohrman DC-50. The Dohrman DC-50 (Dohrman, Santa Clara, Calif.) is designed to analyze liquid samples, although an alternative sample injection boat system allows analysis of suspensions (i.e., finely ground soil suspended in water or another suitable dispersant). The system involves injecting a 30-μl sample into a sample boat containing CoO, followed by vaporization of H_2O at 90°C and combustion of organic and inorganic C at 850°C. Purified He is used as the carrier gas to sweep the CO_2 formed through a column (350°C) containing alumina coated with Ni where H_2 is introduced to reduce the CO_2 to CH_4. After removal of H_2O by a $CaSO_4$ column, the CH_4 is determined by a flame ionization detector. The peak area is integrated automatically, and the results (milligrams of C per liter) are displayed on a digital read-out. The instrument has a linear response range of approximately 1 to 2,000 mg of C/liter. The instrument was designed for injection of liquid samples and thus should be more amenable to total C analysis of soil extracts than intact soils. Ultrapure He, air, and H_2 must be employed with the instrument. Various aspects of this instrument have been described by Takahashi et al. (1972).

29-2.2.5.3 Coleman Model 33. Coleman Model 33 (Coleman Instruments Division, Perkin-Elmer Corp., Oak Brook, Ill.) is an automated version of the medium temperature resistance furnace method described in section 29-2.2.3 and determines both C and H. Compressed O_2 is purified by $Mg(ClO_4)_2$ and COSORB traps before entry into a combustion tube. A sample placed in the combustion tube is heated to ~1,000°C by a resistance furnace, and the gases formed are passed over CuO, platinized asbestos, silver vanadate, and Ag gauze. Scrubbers are used to remove interfering gases (e.g., N). Two traps in series containing COSORB and $Mg(ClO_4)_2$ retain CO_2 and H_2O, respectively. Both C and H traps are removed from the instrument and weighed manually.

29-2.3 Total Carbon by Wet Combustion

29-2.3.1 INTRODUCTION

The wet combustion analysis of soils by chromic acid digestion has long been a standard method for determining total C, giving results in good agreement with dry combustion. The main advantages for wet combustion are that the cost of apparatus is but a small fraction of the cost for dry combustion equipment and that the parts needed to assemble the apparatus are standard equipment in most laboratories. The chief disadvantage of the earlier wet combustion procedures (e.g., Heck, 1929) is that they use macro equipment, which is tedious to assemble and disassemble, and which occupies considerable bench space more or less permanently. Wet combustion is also used when the special manometric Van Slyke-Neil apparatus (Van Slyke & Folch, 1940; Bremner, 1949) is employed to estimate total C in soils.

The wet combustion method of Allison (1960), described here, embodies important refinements from published procedures, such as a simple

and effective digestion acid mixture (Clark & Ogg, 1942), a simple purification and absorption train assembled on a small panel (McCready and Hassid, 1942), and a more rapid procedure than formerly used (Heck, 1929; Jackson, 1958, p. 211). The significant features of this apparatus (Fig. 29-2) are as follows: (i) it can be assembled from simple parts and requires no ground-glass connections, (ii) its small internal volume precludes the necessity for preaeration under most laboratory conditions, (iii) it requires only a short period of aeration following digestion, and (iv) the entire assembly (F-K) occupies only a small area. This method is satisfactory for salt-affected soils high in Cl^- and also for the dry residues of soil extracts rich in organic matter. A rapid treatment to remove carbonates described in section 29-3.3.2 permits determination of organic C on the residue of a pretreated calcareous soil. The following description of wet combustion methodology was presented by Allison et al. (1965).

29-2.3.2 PRINCIPLES

The soil sample is digested in a 60:40 mixture of H_2SO_4 and H_3PO_4 containing $K_2Cr_2O_7$. The boiling temperature of this mixture, 210°C, is high enough to ensure complete oxidation of carbonaceous matter, yet low enough to prevent excessive fuming in the condenser. The CO_2 evolved is absorbed by a suitable absorbent and weighed, although it may be absorbed in a standard base and titrated.

A combination of fuming H_2SO_4, H_3PO_4, HIO_3 (added as KIO_3), and CrO_3 has been used for determining C in organic compounds (Van Slyke & Folch, 1940) and in soil (McCready & Hassid, 1942). The reputed advantages of this oxidation mixture are that it vigorously attacks and dehydrates resistant forms of C, thereby reducing boiling time for complete oxidation, and that it facilitates conversion of CO to CO_2. Carbon monoxide is often produced when readily oxidizable carbohydrates are present in the sample. Extensive comparisons of the Van Slyke-Folch and the 60:40 H_2SO_4-H_3PO_4 oxidizing mixtures on many soils indicate that the two mixtures are equally effective in converting total soil C to CO_2. The more rapid digestion with the Van Slyke-Folch mixture, resulting in a saving of 3 or 4 min per determination, is not sufficient advantage to offset the difficulties of preparing and maintaining a digestion acid that contains fuming H_2SO_4. Moreover, it was found that the need for HIO_3 in the digestion mixture does not exist, which indicates that soil organic matter contains little or no active carbohydrate capable of producing CO during digestion (Allison, 1960).

Salt-affected soils frequently contain sufficent Cl^- to give errors by wet combustion analysis whether the CO_2 is determined titrimetrically (Clark & Ogg, 1942) or gravimetrically (Allison, 1960). When soil high in Cl^- is heated in a digestion mixture containing $Cr_2O_7^{2-}$, chromyl chloride (CrO_2Cl_2) is formed by the following reaction before boiling begins:

$$6H_2SO_4 + K_2Cr_2O_7 + 4KCl = 2CrO_2Cl_2 + 6KHSO_4 + 3H_2O. \quad [4]$$

The reddish CrO_2Cl_2 decomposes at about 190°C, releasing free Cl_2, with a

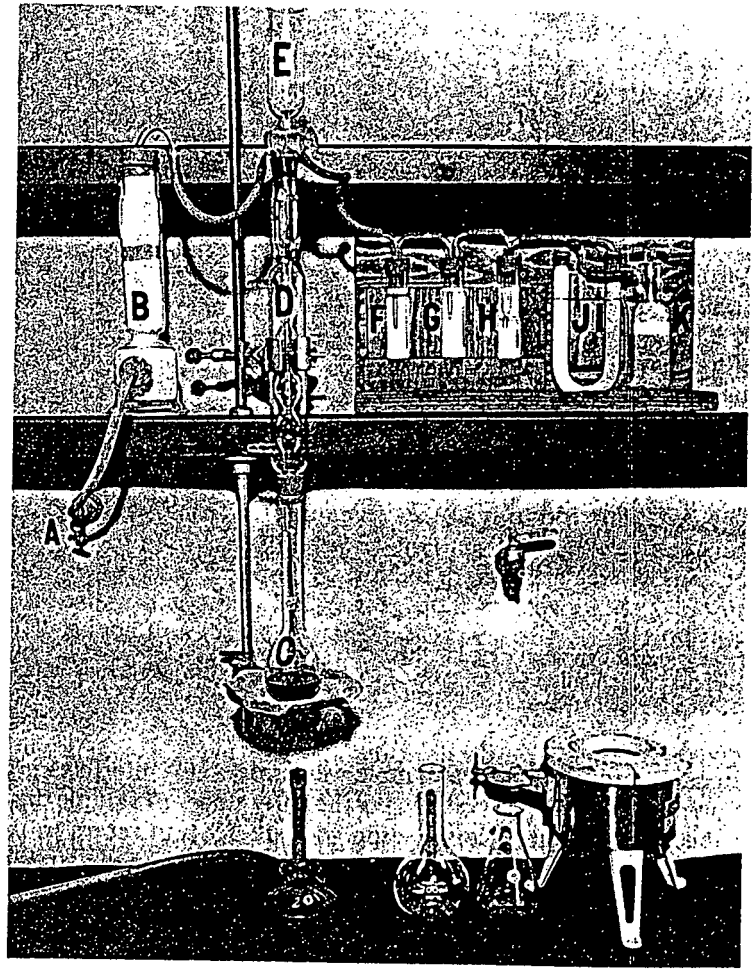

Fig. 29-2. Photograph of apparatus for the determination of C by the Allison (1960) method. For legend, see section 29-2.3.3.1.

color change to pale green. Any Cl_2 and traces of undecomposed CrO_2Cl_2 that pass the purification train are retained in the CO_2 absorption bulb to give a positive error.

In the methods described, Cl_2 interference is prevented by including two traps in the purifying train, one containing KI and one containing Ag_2SO_4 (traps F and G in Fig. 29-2). The use of Ag_2SO_4 alone gives protection up to about 0.2% Cl^- (Allison, 1960), but its protective value becomes questionable at higher Cl^- concentrations. Since KI has a very high capacity to absorb free Cl_2 by the reaction

$$2KI + Cl_2 = 2KCl + 2I_2, \quad [5]$$

the use of a KI trap is recommended for soils high in Cl^-. With both traps in

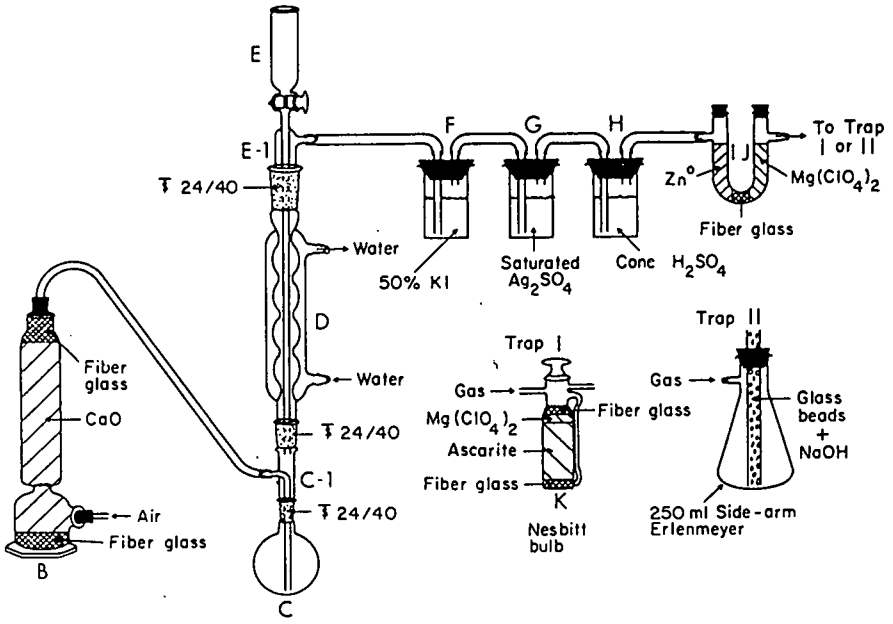

Fig. 29-3. Diagram of apparatus used to determine C by the Allison (1960) method. Trap I or II is used for determination of CO_2 evolved by gravimetric or titrimetric techniques, respectively. (Diagram is not drawn to scale.)

the system, Cl^- up to 5% of the sample weight does not interfere, provided proper precautions are observed during the early stages of sample digestion. Use of the Ag_2SO_4 trap in conjunction with the KI trap serves to indicate when the latter is exhausted. For soils containing trace or low amounts of Cl^-, the carrier stream may flow directly into the Ag_2SO_4 trap.

29-2.3.3 METHOD

29-2.3.3.1 Special Apparatus. The traditional apparatus is shown in Fig. 29-2, and a modified version is presented in Fig. 29-3. Assemble the apparatus in Fig. 29-2 from the following parts: (A) Hoke needle valve: (B) 25-cm high soda-lime tower; (C) 100-ml Kjeldahl flasks to fit a no. 2 stopper; (D) Allihn four-bulb condenser, fitted with a no. 2 stopper at the delivery end; (E) 60-ml open-top separatory funnel; (F-H) 25 by 90 mm shell vials with no. 4 stoppers; (I and J) 15-cm long $CaCl_2$ U-tube; and (K) Nesbitt absorption bulb. Use neoprene stoppers and gum rubber tubing for all connections. Coat all rubber tube connections lightly with silicone lubricant.

Items C through E can be replaced with ground-glass joint glassware if desired (Fig. 29-3). All joints are standard-taper 24/40. The following parts are needed: (C) 100-ml round-bottom flask (Corning 4320); (C-1) distilling adapter tube (Corning 9421), which contains inlet tube for bubbling CO_2-free air into digestion acid mixture; (D) Allihn condenser, ~300-mm jacket

length (Corning 2480); (E-1) distilling tube with suction side arm (Corning 9420) (side arm is connected to purifying traps); (E) graduated separatory funnel (Corning 6382 A). A heating mantle and rheostat are used to heat the 100-ml digestion flask.

Provide a CO_2-free carrier stream by releasing air from an air pressure line through valve A and passing it through soda-lime tower B. Connect B in a glass tube 4 mm o.d. that extends downward through condenser D and dips about 1 cm below the surface of the oxidizing acid in digestion flask C. Shorten the stem of funnel E to a length of about 9 cm, and reduce the tip opening of the stem to a diameter of about 2 mm. Adjust the position of the funnel E to extend into D at least 5 cm below the stopper to avoid contact between oxidizing acid and stopper. Lubricate stopcock E with the digestion acid mixture or with syrupy H_3PO_4. Regular stopcock lubricant *should not* be used on stopcocks.

Assemble the purifying traps, F to J, inclusive, on a panel 19 cm high by 36 cm long, with attached base as shown in Fig. 29-2. Fit the vials of traps F, G, and H with no. 4 stoppers that have approximately 6 cm of the bottom cut off to provide a tight seal with the vials. Reduce the tip openings of the inflow tubes in F and G, but do not make them smaller than 1 mm in diam, or sealing may occur. Fill traps F and G approximately two-thirds full with 50% KI solution and saturated Ag_2SO_4, respectively. Adjust the inflow tubes so that they extend into the solutions not more than 3.8 cm for trap F and 1.3 cm for trap G; otherwise back pressure may develop and cause leaks in the system.

Fill trap H not more than one-third full with concentrated H_2SO_4. Prepare the inflow tube for H from the barrel of a 5-ml pipette with the tip extending not more than 1.3 cm into the acid (note that trap H connects directly to trap I). Place a fiberglass disc in the bottom of the U-tube; and fill the right side, trap I, with 30-mesh granular Zn for absorbing any acid fumes that escape past H. Fill the left side, trap J, with anhydrous $Mg(ClO_4)_2$, which absorbs water from the carrier stream containing evolved CO_2 before it enters K.

Fill the Nesbitt absorption bulb K with any good, self-indicating absorbent having a high capacity for absorbing CO_2. Indicarb and Mikhobite are excellent for this purpose. When filled as shown in Fig. 29-2, the bulb contains successively a 3-cm layer of 8- to 14-mesh absorbent, a 2-cm layer of 14- to 20-mesh absorbent, and a 1-cm overlayer of anhydrous $Mg(ClO_4)_2$, with a wad of glass wool above and below the column.

29-2.3.3.2 Reagents.

1. Digestion acid mixture: Pour 600 ml of conc sulfuric acid (H_2SO_4) into 400 ml of 85% phosphoric acid (H_3PO_4), cool the mixture, and store it in a glass-stoppered bottle. Keep the bottle well stoppered to prevent absorption of water vapor.
2. Potassium dichromate ($K_2Cr_2O_7$), reagent grade.
3. Potassium iodide (KI) solution, 50%: Dissolve 100 g of KI in 100 ml of water.

4. Silver sulfate (Ag_2SO_4) solution, saturated.
5. Carbon dioxide (CO_2) absorbent, self-indicating, 7- to 14- and 14- to 20-mesh size: Suitable materials are Mikhobite (G. Frederick Smith Chemical Co., Columbus, Ohio), Caroxite or Indicarb (Fisher Scientific, Pittsburgh, Pa.), or Ascarite (Arthur H. Thomas Co., Philadelphia).
6. Soda lime, 8- to 14-mesh size.
7. Granular Zn, <30-mesh size.
8. Anhydrous magnesium perchlorate [$Mg(ClO_4)_2$] (Anhydrone, Dehydrite, or equivalent).

29-2.3.3.3 **Procedure.** Place a finely ground soil sample containing 20 to 40 mg of C (usually 0.5 to 3 g of oven-dry soil) into digestion flask C, and add about 1 g of $K_2Cr_2O_7$. Wash down the neck of the flask with 3 ml of distilled water, and connect the flask to condenser D. Weigh the Nesbitt bulb (sections 29-2.2.3.4 and 29-2.3.3.4), attach it to the system, and immediately open the valve at the top of the bulb. Pour 25 ml of the digestion acid mixture into funnel E above the condenser, and cover the funnel with a small beaker. Open stopcock E, allow the acid to flow through D into flask C, and close the stopcock immediately to prevent loss of CO_2. Adjust the air delivery tube that passes through D into C so that its tip extends not more than 1 cm into the acid during digestion.

At this point, turn on the cooling water. Adjust the carrier stream to a flow rate of about 2 bubbles/sec, and maintain this rate during digestion. Apply a flame 5 to 6 cm high, and bring the sample to boiling in 3 or 4 min. If Cl^- is high, heat the mixture slowly at first, and bring it to boiling in about 5 min. Continue gentle boiling, avoiding excessive frothing, for a total heating period of 10 min. Reduce the rate of heating if visible white fumes of SO_3 occur above the second bulb of D during digestion.

Remove the flame at the end of the digestion period, and aerate the system for 10 min at the rate of 6 to 8 bubbles/sec. When aeration is completed, shut off the air stream, and disconnect the digestion flask from the condenser. Close the stopcock on the Nesbitt bulb, and disconnect it from the system. Brush the bulb with a camel's hair brush to remove any lint and dust, and weigh it immediately. Make a blank determination, using the identical procedure, but without sample. Add four to five glass beads to the blank to prevent bumping. The calculation is as follows:

$$\text{Total C, \%} = \frac{(\text{g } CO_2 \text{ (sample)}] - [\text{g } CO_2 \text{ (blank)}]}{\text{g water-free soil}} \times 0.2727 \times 100. \quad [6]$$

29-2.3.3.4 **Comments.** Soil samples should be ground to pass through a sieve with openings 0.5 mm or smaller in diam. This is necessary to reduce errors due to the presence of occasional fragments of carbonate minerals in a predominantly noncalcareous matrix.

A single analysis, involving all operation from weighing the sample to calculation of results, requires 25 min. By using two sets of apparatus, one may analyze two samples concurrently, thereby reducing the overall time required to 15 min per determination, provided the digestion phase of one sample coincides with the aeration phase of the other.

Because CO_2 absorption bulbs change weight on standing overnight or for longer periods, it is necessary to bring the bulb to constant weight by the following procedure before beginning C or blank determinations. Without being weighed, the bulb should be connected to the system, all reagents (but no soil) should be added to the digestion flask, and the apparatus should be operated as directed for sample determinations. After aeration, the bulb should be detached and weighed, and this weight should be used as the initial (constant) weight of the bulb. See section 29-2.2.3.4 for additional comments on care and use of CO_2 absorption bulbs.

Blank determinations have ranged from 0.8 to 1.2 mg of CO_2, for which an average value of 1.0 mg has been used. If blanks are found to be high, preaeration may be necessary. The system may be preaerated by placing the digestion flask (containing all materials except the digestion acid) in position for digestion, disconnecting the rubber tube between D and F, opening valve A, and directing a stream of CO_2-free air (about 10 bubbles/sec) into C and through D for 2 min (spattering of the contents in C must be avoided). The air flow is then readjusted to about 2 bubbles/sec, D is connected to F, and the analysis is performed as directed.

The H_2SO_4 in trap H should be renewed at the beginning of each day's operation or more often if frothing occurs. The KI solution in trap F has a high capacity for absorbing Cl^-, and the need for its renewal is indicated by the first trace of an AgCl precipitate in trap G.

The Nesbitt absorption bulb, when filled as described, weighs about 125 g and will absorb about 10 g of CO_2, equivalent to about 100 determinations averaging 100 mg of CO_2 each.

When the apparatus is idle overnight or for longer periods and the Nesbitt bulb is detached, the tube connecting J and K should be clamped off to prevent entrance of water vapor into the desiccant in trap J.

A titrimetric procedure for CO_2 determination is readily adaptable to the above procedure (Fig. 29-3). Replace the Nesbitt bulb with a 250-ml sidearm Erlenmeyer (filtering flask) fitted with a no. 6 1/2 stopper containing a 22 cm by 14 mm diam glass tube. This bubble tower should extend to within 0.5 cm of the flask bottom and should be filled with glass beads. Through the glass tube, 25 ml of 1*N* KOH should be added, and the soil sample should be oxidized as described previously. Tropolene O can be added to the KOH to ensure that sufficient alkalinity remains after trapping the CO_2 evolved. After oxidation, the KOH is washed from the bubble tower with distilled water, treated with 5 ml of saturated $BaCl_2$ and several drops of phenolphthalein, and titrated with standard HCl. The data are calculated from

$$\text{Total C, \%} = \frac{\text{ml (blank)} - \text{ml (sample)}}{\text{g soil}} \times N_{HCl} \times 0.6. \quad [7]$$

29-3.2 Organic Carbon as Calculated from Total Carbon Determinations

Methods previously described for total C are basic for many of the procedures used to determine organic C in soils. However, soils may contain both organic and inorganic C, and thus total C analysis procedures recover both forms of C. In noncalcareous soils and soils not recently limed, the total C can be considered to be organic C. With calcareous or recently limed soils, organic C may be estimated as the difference between total C and inorganic C concentrations.

29-3.2.1 ORGANIC CARBON IN NONCALCAREOUS SOILS

Prepare soil samples, and conduct a total C determination by dry or wet combustion using titrimetric, gravimetric, volumetric, infrared, or thermal conductivity techniques to quantitate evolved CO_2 as described in section 29-2. Report the total C determined as percent organic C in the sample (i.e., total C = organic C).

29-3.2.2 ORGANIC CARBON IN CALCAREOUS SOILS

Prepare soil samples, and conduct a total C determination on the sample by dry or wet combustion techniques as described in section 29-2. Determine inorganic C on a separate sample by one of the quantitative methods described in section 11-2. Calculate the percent organic C in the sample from the relationship

$$\% \text{ organic C} = \% \text{ total C} - \% \text{ inorganic C.} \quad [8]$$

29-3.3 Wet and Dry Combustion Techniques for Organic Carbon in Calcareous Soils

In contrast to noncalcareous soils, inorganic C must be removed from calcareous or recently limed soils before the analysis if wet or dry combustion techniques are used to directly measure the organic C present.

Inorganic C is conveniently removed before wet combustion by pretreating the sample contained in a digestion flask with a mixture of dilute H_2SO_4 and $FeSO_4$. The $FeSO_4$ is added to the mixture to minimize oxidation and decarboxylation of organic matter by added H_2SO_4 or by MnO_2 present in soil (Allison, 1960). After pretreatment, the digestion flask containing soil is transferred to the combustion train, and a total C determination is carried out as described in section 29-2.3.

Inorganic C removal is generally more difficult before determination of organic C by dry combustion techniques. Treatment of soil at room temperature with H_2SO_3 followed by heating to remove excess H_2SO_3 is normally used to decompose inorganic C compounds (Piper, 1942; Bremner, 1949); however, several difficulties are apparent with the procedure. Little destruction of organic matter occurs during room temperature

treatment of samples with H_2SO_3, but some decarboxylation is possible as the sample is heated (Bremner, 1949). It is difficult to decide when all inorganic C has been removed and when H_2SO_3 treatment should be discontinued. It is doubtful that dolomite is completely decomposed by the relatively mild H_2SO_3 treatment employed (Allison, 1965). Nommik (1971) suggested that inorganic C may be effectively removed from soil samples by treatment with a metaphosphoric acid solution for 30 min at room temperature and 30 min at 130°C. However, Nommik's procedure has not been evaluated with a variety of soils.

29-3.3.1 TEST FOR PRESENCE OF INORGANIC CARBON

Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4*N* HCl dropwise to the wetted sample, and observe any effervescence. Allow sufficient time for dolomite to react (~5 min). If inorganic C is absent from the soil, proceed with organic C (total C) analysis as per section 29-2. If inorganic C is present or the test is not definitive, proceed as described below.

29-3.3.2 PRETREATMENT PRIOR TO WET COMBUSTION

29-3.3.2.1 Special Apparatus. See the special apparatus listed in section 29-2.3.3.1.

29-3.3.2.2 Reagents.

1. Digestion reagent for carbonates (H_2SO_4-$FeSO_4$): Dissolve 57 ml of conc sulfuric acid (H_2SO_4) and 92 g of ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) in 600 ml of deionized water, cool, and dilute to 1 liter.
2. Potassium dichromate ($K_2Cr_2O_7$), reagent grade, pulverized.
3. Other reagents as described in section 29-2.3.3.2.

29-3.3.2.3 Procedure. Prepare soil samples as described in section 29-2.3.3.3. Transfer a sample of known water content and containing 20 to 40 mg of C (but not more than 2 g of soil) to a 100-ml Kjeldahl digestion flask. Using 3 ml of the H_2SO_4-$FeSO_4$ digestion acid, wash down any soil that adheres to the neck of the flask. Place the flask in a rack or beaker, and allow the sample to digest at room temperature with occasional turning of the flask for at least 20 min or until effervescence appears to cease. Then hold the flask upright over a flame 1 cm high, and boil the contents slowly for 1.5 min to destroy any remaining carbonate. Rotate the flask continuously during boiling to avoid excessive frothing. Allow the sample to cool.

Insert a long-stemmed funnel into the flask, and add 2 g of pulverized $K_2Cr_2O_7$. Immediately connect the flask to the reflux condenser (Fig. 29-2), and proceed with the determination of organic C as directed in section 29-2.3.3.3 beginning with the third sentence.

Report the C present in the pretreated sample as percent organic C.

29-3.3.2.4 Comments. The 3 ml of 2*N* H_2SO_4-5% $FeSO_4$ used in this procedure replaces the 3 ml of distilled water used in the total C procedure

described in section 29-2.3.3.3. Three milliliters of this reagent adds 6 meq H^+, which will neutralize 0.3 g of $CaCO_3$ (i.e., 15% $CaCO_3$ in a 2-g soil sample). An appreciable excess of acidity must be present to ensure complete decomposition of carbonates. Rather than using >3 ml of the 2*N* reagent for soils containing more than ~10% $CaCO_3$ equivalent, it is preferable to use 3 ml of a 3 or even a 4*N* H_2SO_4–5% $FeSO_4$ reagent.

29-3.3.3 PRETREATMENT PRIOR TO DRY COMBUSTION

29-3.3.3.1 Special Apparatus. See the special apparatus listed in sections 29-2.2.3.1 and 29-2.2.4.1.

29-3.3.3.2 Reagents.

1. Sulfurous acid (H_2SO_3), approximately 5%: Bubble SO_2 through distilled water until a saturated solution is obtained. Keep the bottle well stoppered to prevent rapid loss of SO_2.
2. Sodium hydroxide (NaOH), pellets.

29-3.3.3.3 Procedure. Transfer a soil sample that passes through a 100- or 140-mesh sieve (section 29-2.2.3.4) and of known water content to a nonporous combustion boat that has been previously ignited and cooled. Treat the sample with an excess of a 5% H_2SO_3 solution. After several hours, remove the water and excess H_2SO_3 by leaving the boat overnight in an evacuated desiccator containing NaOH pellets. Repeat the treatment until CO_2 evolution ceases on addition of H_2SO_3.

Proceed with the determination of organic C by one of the dry combustion methods (section 29-2.2.3 or 29-2.2.4). Report the C present in the pretreated samples as percent organic C.

29-3.4 Organic Carbon in Soil Extracts

29-3.4.1 SPECIAL APPARATUS

See the special apparatus listed in section 29-2.3.3.1.

29-3.4.2 REAGENTS

See the reagents listed in section 29-2.3.3.2.

29-3.4.3 PROCEDURE

Place an aliquot of the extract (10 to 50 ml, depending on the organic C content) in a 100-ml Kjeldahl digestion flask, and add 1 ml of the H_2SO_4–$FeSO_4$ reagent. Immerse the bulb of the flask in boiling water, and direct a stream of dry, dust-free air onto the surface of the liquid in the flask. Reduce the volume of solution in the flask to 3 ml or less. Add five or six glass beads and 1 g of $K_2Cr_2O_7$ to the flask, and proceed with the determination of organic C as directed in section 29-2.3.3.3.

29-3.4.4 COMMENTS

Drying of extracts is best accomplished in 100-ml flasks of the Kjeldahl type. A 2-liter beaker conveniently holds four flasks.

29-3.5 Rapid Dichromate Oxidation Techniques

29-3.5.1 INTRODUCTION AND PRINCIPLES

Schollenberber (1927) first proposed that the organic matter in soil may be oxidized by treatment with a hot mixture of $K_2Cr_2O_7$ and H_2SO_4 according to Eq. [9].

$$2\,Cr_2O_7^{2-} + 3\,C^0 + 16\,H^+ = 4\,Cr^{3+} + 3\,CO_2 + 8\,H_2O. \qquad [9]$$

After the reaction, the excess $Cr_2O_7^{2-}$ is titrated with $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, and the $Cr_2O_7^{2-}$ reduced during the reaction with soil is assumed to be equivalent to the organic C present in the sample. It must be emphasized that all methods based on analysis of $Cr_2O_7^{2-}$ remaining or Cr^{3+} formed assume that C in soil organic matter has an average valence of zero. Although most dichromate oxidation procedures described since the original Schollenberger method have involved chromic acid solutions or mixtures of concentrated H_2SO_4 and aqueous $K_2Cr_2O_7$ solutions (Table 29-3), the use of other oxidants has been proposed. Degtijareff (1930) suggested that a mixture of H_2O_2 and chromic acid be used to oxidize organic matter. However, Walkley and Black (1934) conclusively established that the addition of H_2O_2 to chromic acid procedures gave fictitiously high values for organic C because H_2O_2 reduces $Cr_2O_7^{2-}$ in acid solution. Tinsley (1950) and Kalembasa and Jenkinson (1973) proposed that the chromic acid mixture used to oxidize organic C compounds be 9 and 4.5*N*, respectively, with respect to H_3PO_4. There is no evidence, however, to suggest that oxidation mixtures

Table 29-3. Digestion reagents used in various rapid dichromate methods for organic C determinations.

Method	Digestion reagent concentration		
	$K_2Cr_2O_7$†	H_2SO_4	H_3PO_4
		N	
Schollenberger (1927)	0.35	36	–
Tyurin (1931)	0.40	18	–
Walkley & Black (1934)	0.33	25	–
Anne (1945)	0.16	22	–
Tinsley (1950)	0.40	15	9
Mebius (1960)	0.267	20	–
Kalembasa & Jenkinson (1973)	0.20	18	5
Nelson & Sommers (1975)	0.40	21.6	–
Modified Mebius (described here)	0.20	21.6	–

† Based on $Cr_2O_7^{2-} + 14H^+ = 2Cr^{3+} + 7H_2O + 6e^-$ half reaction.

Grain Size

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

SAS Number _____

SPECIAL ANALYTICAL SERVICES
Regional Request

__X__ Regional Transmittal _____ Telephone Request

A. EPA Region and Site Name: Region V - Wausau NPL Site

B. Regional Representative: Dennis Wesslowski

C. Telephone Number: ( )

D. Date of Request:

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

1. General description of analytical service requested: Determination of grain size in soil samples.

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

30 soils - concentrations unknown

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

Superfund Remedial

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Shipped as a group by overnight carrier

6. Approximate number of days results required after lab receipt of samples: Laboratory should report results within 30 days after receipt of samples.

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

Particle Size Analysis of Soils, ASTM Method D422

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

Use only the method specified above. Obtain approval of CPMS, CRL, prior to use of any other method.

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

Submit all raw data including container tare weights, hydrometer readings (along with any correction factor associated with the hydrometer used) and liquid temperatures. Provide reportables as described in sections 17 and 18 of Method D422.

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

Name of sampling/shipping contact: Brian Hegge

Phone: (608) 273-0440

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

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Percentage Finer Than	2%	Duplicates within 10%

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Lab Duplicate	2 for sets ≤ 10 1 per 10 for sets >10	10%

III. *Action Required if Limits are Exceeded:

Reanalyze. Contact Chuck Elly (312) 353-9087.

[jam-26-4]

results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1½-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
¾-in. (19.0-mm)	No. 140 (106-μm)
⅜-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-μm)
¾-in. (19.0-mm)	No. 50 (300-μm)
⅜-in. (9.5-mm)	No. 100 (150-μm)
No. 4 (4.75-mm)	No. 200 (75-μm)
No. 8 (2.36-mm)	

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly reverse or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve, while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion p
10 sieve shall be approximately 1
soils and approximately 65 g fo
soils.

5.2 Provision is made in Sectio
D 421 for weighing of the air-dry s
purpose of tests, the separation of
No. 10 sieve by dry-sieving and wa
weighing of the washed and drie
tained on the No. 10 sieve. Fro
masses the percentages retained ar
No. 10 sieve can be calculated i
with 12.1.

NOTE 8—A check on the mass value
oughness of pulverization of the clods
by weighing the portion passing the N
adding this value to the mass of the wa
dried portion retained on the No. 10 sie

SIEVE ANALYSIS OF PORTION ON NO. 10 (2.00-mm) SIE

6. Procedure

6.1 Separate the portion retained
10 (2.00-mm) sieve into a series of fr
the 3-in. (75-mm), 2-in. (50-mm),
mm), 1-in. (25.0-mm), ¾-in. (19.0
(9.5-mm), No. 4 (4.75-mm), and N
or as many as may be needed deper
sample, or upon the specifications f
rial under test.

6.2 Conduct the sieving operatio
of a lateral and vertical motion o
accompanied by a jarring action in o
the sample moving continuously over
of the sieve. In no case turn or mani
ments in the sample through the siev
Continue sieving until not more thar
of the residue on a sieve passes that s
1 min of sieving. When mechanical
used, test the thoroughness of sievin
the hand method of sieving as describ

6.3 Determine the mass of each fr
balance conforming to the requireme
At the end of weighing, the sum of
retained on all the sieves used sho
closely the original mass of the quanti

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cyclinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hy-

drometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 2½ psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the
of the suspension by inserting the
into the suspension.

11. Sieve Analysis

11.1 After taking the final hydr
ing, transfer the suspension to a No.
sieve and wash with tap water ur
water is clear. Transfer the materia
200 sieve to a suitable container, dr
at 230 ± 9°F (110 ± 5°C) and n
analysis of the portion retained, us
sieves as desired, or required for the
upon the specification of the materi

CALCULATIONS AND REP

12. Sieve Analysis Values for
Coarser than the No. 10 (2.00-r

12.1 Calculate the percentage pas
10 sieve by dividing the mass passin
sieve by the mass of soil originally
No. 10 sieve, and multiplying the re
To obtain the mass passing the N
subtract the mass retained on the
from the original mass.

12.2 To secure the total mass of
the No. 4 (4.75-mm) sieve, add to
the material passing the No. 10 sieve
the fraction passing the No. 4 sieve
on the No. 10 sieve. To secure the t
soil passing the ⅜-in. (9.5-mm) sieve
total mass of soil passing the No.
mass of the fraction passing the ⅜-i
retained on the No. 4 sieve. For the
sieves, continue the calculations in
manner.

12.3 To determine the total perce
ing for each sieve, divide the total m
(see 12.2) by the total mass of samp
tiply the result by 100.

13. Hygroscopic Moisture Correctio

13.1 The hydroscopic moisture co
tor, is the ratio between the mass o
dried sample and the air-dry mass be
It is a number less than one, except
is no hygroscopic moisture.

14. Percentages of Soil in Suspensio

14.1 Calculate the oven-dry mass
in the hydrometer analysis by mult
air-dry mass by the hygroscopic mois

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. Accord-

ing to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,

n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)),

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of soil particles, and

G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation
gradation of the sample tested.
those contained in the sample
testing, the report shall so state
maximum size.

18.2 For materials tested
definite specifications, the f
such specifications shall be
tions smaller than the No. 1
from the graph.

18.3 For materials for wh
definite specifications is not
the soil is composed almost
passing the No. 4 (4.75-m
read from the graph may be

(1) Gravel, passing 3-in. and re
No. 4 sieve
(2) Sand, passing No. 4 sieve
tained on No. 200 sieve
(a) Coarse sand, passing No
and retained on No. 1
(b) Medium sand, passing
sieve and retained on
sieve
(c) Fine sand, passing No.
and retained on No. 2
(3) Silt size, 0.074 to 0.005 mm

have been retained on the
ıt been removed. This mass
percentage retained on the
ius total percentage passing
e total sample represented
sed (as calculated in 14.2
l by 100.
.t the total mass passing the
gether the fractional masse
.eves, including the No. 1
is sum from the mass of the
ılated in 14.2).
ct the total masses passing
ves, in a manner similar t

the total percentages pass
ɔtal mass passing (as calc
: total mass of sample (a
ınd multiply the result b

ydrometer analysis is pe
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not made on a portion o
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ıall include the following
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of sand and gravel part

ounded or angular.
—hard and durable, soft, or

ıvity, if unusually high or

lty in dispersing the fraction
2.00-mm) sieve, indicating
and amount of dispersing

sion device used and the
ɔn period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve . . . %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve . . . . %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve . . %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve . . . %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve . . . %
(3) Silt size, 0.074 to 0.005 mm . . . . %
(4) Clay size, smaller than 0.005 mm
  Colloids, smaller than 0.001 mm

18.4 For materials for which co
definite specifications is not indica
the soil contains material retained
sieve sufficient to require a sieve a
portion, the results may be repor
(Note 17):

SIEVE ANALYSIS

Sieve Size	
3-in.	
2-in.	
1½-in.	
1-in.	
¾-in.	
⅜-in.	
No. 4 (4.75-mm)	
No. 10 (2.00-mm)	
No. 40 (425-μm)	
No. 200 (75-μm)	

HYDROMETER ANALYSI

0.074 mm	
0.005 mm	
0.001 mm	

NOTE 17—No. 8 (2.36-mm) and
sieves may be substituted for No. 10 a

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve . . %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve . . . . . %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve . . %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve . . . . %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve . . . . . %
(3) Silt size, 0.074 to 0.005 mm . . . . %
(4) Clay size, smaller than 0.005 mm . . %
  Colloids, smaller than 0.001 mm %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	. . . .
2-in.	. . . . .
1½-in.	. . . . .
1-in.	. . . . .
¼-in.	. . . . .
⅜-in.	. . . . .
No. 4 (4.75-mm)	. . . . .
No. 10 (2.00-mm)	. . . .
No. 40 (425-μm)	. . . . .
No. 200 (75-μm)	. . . . .

HYDROMETER ANALYSIS

0.074 mm	. . . . .
0.005 mm	. . . . .
0.001 mm	. . . . .

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

TABLE 1 Values of Correction Factor, a, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5

TABLE 2 Continued

Hydrometer 151H		Hydrometer 152 H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation

$$L = L_1 + \tfrac{1}{2}\,[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,
L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm^3, and
A = cross-sectional area of sedimentation cylinder, cm^2.

Values used in calculating the values in Table 2 are as follows:

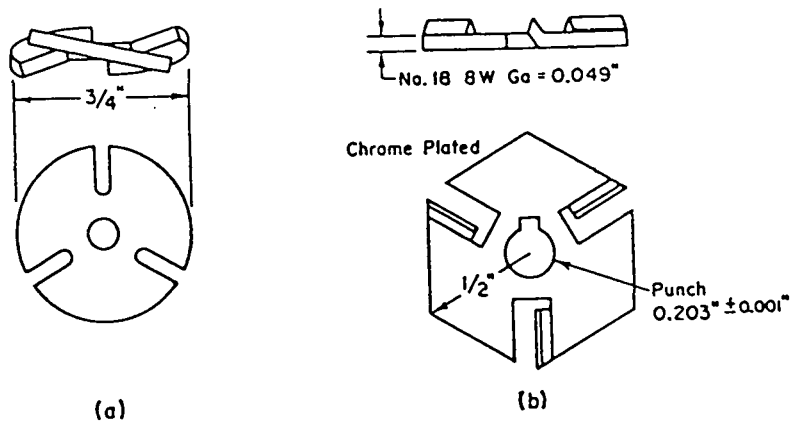
For both hydrometers, 151H and 152H:
L_2 = 14.0 cm
V_B = 67.0 cm^3
A = 27.8 cm^2

For hydrometer 151H:
L_1 = 10.5 cm for a reading of 1.000
= 2.3 cm for a reading of 1.031

For hydrometer 152H:
L_1 = 10.5 cm for a reading of 0 g/litre
= 2.3 cm for a reading of 50 g/litre

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

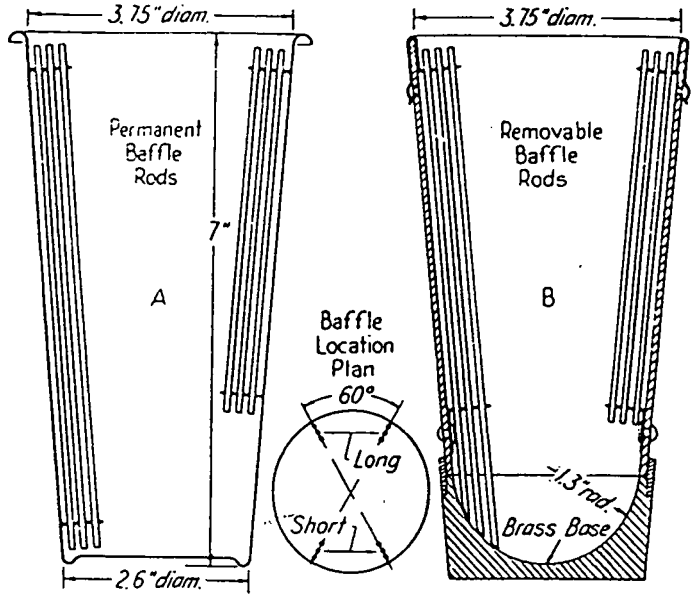
Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149



Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

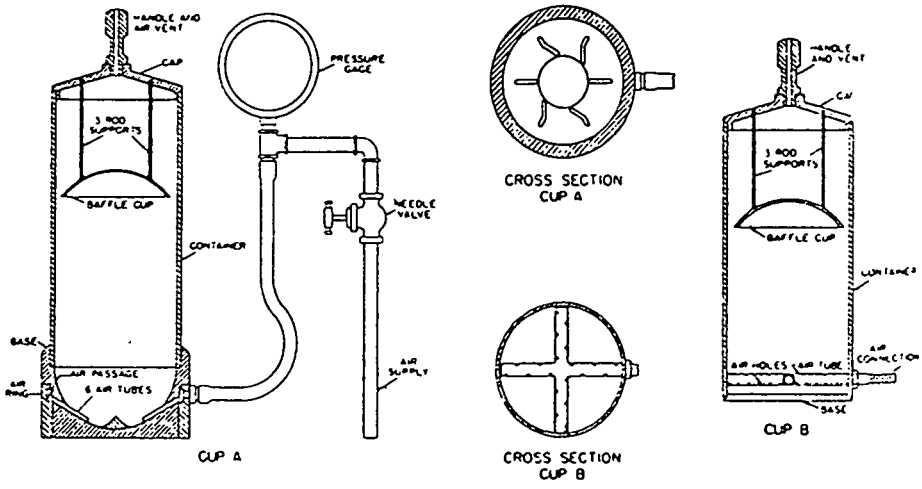
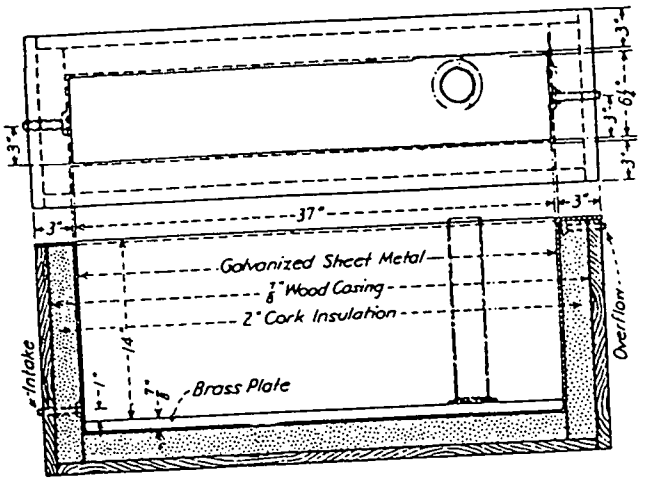


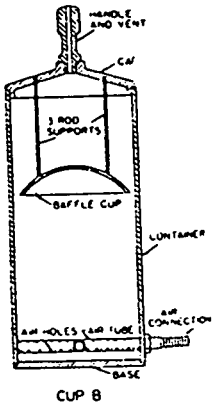
FIG. 3 Air-Jet Dispersion Cups of Apparatus B



Metric Equivalents

in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath



CUP B

APPENDIX E

OPERATION AND MAINTENANCE OF MOUNT SOPRIS, 1000-C NATURAL GAMMA RAY LOGGING TOOL

WARZYN

PORTABLE BOREHOLE LOGGER .

MODEL 1000-C

Operation and Maintenance Manual

Serial Number 011 to Present

September 1977

MOUNT SOPRIS INSTRUMENT COMPANY

P. O. Box 449 Industrial Park
Delta, Colorado, U.S.A. 81416
Phone (303) 874-4852 TWX 910-929-6833

ADDENDUM

Changes for Model 1000-BP

1-10.0 Change the following items to read as shown below:

10.	120 vac Charging cable	1000-BP-120
10A.	240 vac Charging cable	1000-BP-240
11.	12 vdc Charging cable	A-500K 0107

2-6.1 Change paragraph to read as follows:

Connect the proper charging cable for the voltage source available to the BATTERY CHARGE connector (Fig. 1-10). If the 12 vdc cable is used, the red lead connects to the positive (+) terminal of the source and the black lead to the negative (-). Two charging cords are provided for charging from an a.c. source, one for 120 volts and the other for 240 volts. Select the proper cable and connect the free end as follows: Black to the a.c. HOT, white to the RETURN, and green to EARTH GROUND.

NOTE: If the polarity of the 12 vdc cable is reversed, no damage will result but the batteries will not be charged.

TABLE OF CONTENTS

SECTION I

GENERAL INFORMATION

1-1. INTRODUCTION

The Model 1000-C is a complete, fully portable, lightweight (27.3 Kg-60 lbs) backpack mountable, borehole logging unit.

Using the standard probe (G375/A), it is possible to record up to three different logs in one trip in the hole: (1) Gross count, dead-time corrected gamma radiation, (2) Self Potential, and (3) Single Point Resistance. Other Nuclear Pulse counting tools available include gamma-gamma and neutron-neutron.

The recorder is a dual pen, servo-driven type, equipped with a bi-directional chart drive allowing multiple logs and re-runs without resetting the chart paper.

The power requirements for the Model 1000-C are provided by internal nickel cadmium batteries which are recharged by a built-in battery charger when connected to an A.C. or D.C. source. Battery life will provide a minimum of eight (8) hours of logging.

The hand cranked winch has two speeds: a 1:1 ratio for going down hole, and a 4:1 ratio for coming back out. The winch comes with 305 M (1000 ft.) of steel armored logging cable.

The Model 1000-C, when handled reasonably and maintained properly, will provide many years of reliable logging.

1-2.0 GENERAL SPECIFICATIONS

The Model 1000-C logger comes equipped with one combination probe, 305 M (1000') of cable, recorder-instrument assembly, shipping containers, spare parts and consumables, and all necessary cables and hardware required to log a borehole. A backpack frame with necessary fittings is optionally available.

1-2.1 Shipping Weight: 36.4 Kg. (80 lbs.)

1-2.2 Net Weight (With 305 M of cable and backpack frame): 27.3 kg. (60 lbs.)

1-2.3 Shipping Dimensions: Probe/Accessory Case = 33cm x 124.5cm x 10.2cm (13" x 49" x 4")

Winch/Recorder Case = 58.4cm x 48.3cm x 43.2cm (23" x 19" x 17")

1-2.4 Operating Temperature Range (limited by pens): -25°C (-13°F) to 55°C (131°F)

1-3.0 WINCH

1-3.1 Cable Capacity: 305 M (1000')

1-3.2 Cable Type: U. S. Steel type no. 1N10SB (or equivalent) galvanized steel armored logging cable. Single center conductor, D. C. resistance approximately 24Ω/305M. Tensil strength is 408 Kg. (900 lbs.).

1-3.3 Cable Head: Tapered cone and sleeve strain relief. Cable center conductor is water sealed with a Mecca underwater connector. Connection to probe is through center contact and outer screw threads (3/4 - 16). Outside diameter is 2.86 cm (1.125").

1-3.4 Hand Crank Gearing: Direct drive (1:1 ratio) or step down (4:1 ratio) selected with easily removable crank.

1-4.0 RECORDER INSTRUMENT ASSEMBLY

1-4.1 Depth Odometer: 5 digit bi-directional, resettable, mechanical odometer geared directly to the measuring wheel. Resolution is 0.1 M (0.1' on English models).

1-4.2 Measuring System: Hard anodized grooved aluminum wheel. The measuring wheel rotates 3 1/3 revolutions per meter (3' on English models).

1-4.3 Servo Amplifier (Recorder): Two pen, non-overlapping, servo-driven, rectilinear recorder.

1-4.4 Recorder Response Time: Time to full scale (step function in) is ~1 second.

1-4.5 Pens: Disposable felt tip cartridge available in red and black.

1-4.6 Pen lifter: Both pens operate simultaneously with front panel lever.

1-4.7 Chart Drive: Bi-directional, geared (through multi-tooth clutch) to measuring wheel. Turned on and off with front panel shift lever.

1-4.8 Chart Paper: 10 cm wide grid Z-fold paper with 1 cm x 1 cm major divisions and 0.5 cm intermediate lines. Each box contains two packets (7.6 m - 25' each) of paper. The last 61 cm (2') are marked with a red line on the right-hand margin.

1-4.9 Sensitivity: 100 mV. (F. S.)

1-5.0 GAMMA CIRCUIT

1-5.1 Ranges: 5 cps/div. (50 cps full scale) to 5 Kcps/div. (50 Kcps full scale) in 1-2-5 ratio steps.

1-5.2 Internal Calibration Source: 5.000Khz. square wave crystal oscillator with a temperature coefficient of ±0.01%/°C maximum.

1-5.3 Temperature Coefficient (total circuit): Each unit individually aligned for ±0.02%/°C maximum from -25°C to +75°C.

1-5.4 Time Constant: 2 pole Bessel function active filter. Full scale response time is ≈7 seconds (step function in).

1-5.5 Deadtime Correction: Realtime analog correction adjusted to<1 microsecond as calculated by the Energy Research and Development Administration in their test pits at Grand Junction, Colorado.

1-5.6 Input Sensitivity: +380 mV ±10 mV.

1-5.7 Downhole Power: 30 vdc ±1v at 50 ma. maximum as measured at the slip rings (with combination probe connected).

1-6.0 SELF POTENTIAL

1-6.1 Ranges: 2mV./div. (20mV full scale) to 100 mV/div. (1v. full scale) in 1-2-5 ratio steps.

1-6.2 Bias Range: 0 to 500 mV + or - as selected with POLARITY switch.

1-6.3 Time Constant: 2 pole Bessel function active filter. Full scale response time is ≈1 second (step function in).

1-6.4 Input Impedance: >2 megohms.

1-7.0 SINGLE POINT RESISTANCE

1-7.1 Range: 2Ω/div. (20 ohms full scale) to 100Ω/div. (1000 ohms full scale) in 1-2-5 ratio steps.

1-7.2 Displacement: 0 to 500 ohms maximum.

1-7.3 Time Constant: 2 pole Bessel function active filter. Full scale response time is ~1 second (step function in).

1-7.4 Current Generator Output: Constant current (varies with range switch setting--20mA max.) at 25 hz to 30 hz (selected by internal switch).

1-7.5 Resistance Voltmeter Input Impedance: >500 K ohms.

1-8.0 BATTERIES & BATTERY CHARGER

1-8.1 Battery Type: General Electric type GCW3.5SB (or equivalent) Nickel Cadmium batteries (10 each).

1-8.2 Battery Rating: 1.2vdc at 3.5 Ah.

1-8.3 Battery Life: 8 hours continuous operation minimum. Varies with log(s) being run. Maximum drain is during SP-R log.

1-8.4 Battery Charger Input: 12 to 14 vdc at 1.0 A maximum, 120 vac at 0.10 A max., and 220 vac at 0.05 A maximum (A. C. input is 50 to 400 hz.).

1-8.5 Battery Charger Output: Dual, constant current outputs. 350 mA each.

1-8.6 Charging time to full charge: 12 to 14 hours.

1-9.0 PROBES

1-9.1 G375/A Standard Combination Probe

1-9.1.1 Dimensions: 1.1 m (43.5") long by 3.18 cm (1.25") maximum outside diameter including neoprene insulating sheath. The stainless steel housing is 2.86 cm (1.125") diameter.

1-9.1.2 Weight: 2.95 Kg (6.5 lbs.).

1-9.1.3 Construction: Stainless steel (type 303) housing with lead electrode for SP and R logs. O-ring sealed at all joints.

1-9.1.4 Power Requirement: 20 vdc (at cable head) minimum at 35 mA. Maximum voltage in is 30 vdc, 24 volts is nominal.

1-9.1.5 Scintillation Crystal: 38.1mm (1.50") long by 12.7mm (0.5") diameter sodium iodide, thalium activated scintillation crystal in ruggedized, sealed, aluminum mount.

1-9.1.6 Electrical Connection: Insulated center spring loaded contact and mechanical threaded attachment to cable head. Center contact is positive.

1-9.1.7 Output: 6.5v 1 microsecond wide positive pulse superimposed on the positive supply line (center conductor).

1-9.1.8 Deadtime: Constant 5.0 microseconds.

1-9.1.9 K-factor: Average K-factor at 10 cm intervals is 4.8×10^{-6} (2.5×10^{-5} at 0.5' intervals).

1-9.2 G375/A-1.0 1" O.D. Combination Probe

1-9.2.1 Dimensions: 1.1m (43.5") long by 2.54cm (1.0") outside diameter stainless steel housing (for very small diameter holes). The probe must be covered with a neoprene sheath or electrical tape to run the SP and R logs.

1-9.2.2 Weight 2.7 Kg (6 lbs.).

1-9.2.3 K-factor: Average K-factor at 10 cm intervals is 5.2×10^{-6} (2.6×10^{-5} at 0.5' intervals).

All other characteristics are the same as Model G375/A.

1-9.3 G375/AS Stratigraphic Combination Probe

1-9.3.1 Dimensions: 1.02m (40.2") long by 41.1 cm (1.62") maximum outside diameter including neoprene sheath (housing is 38.1cm (1.50") diameter.

1-9.3.2 Weight: 4.4 Kg (9.7 lbs.).

1-9.3.3 Scintillation Crystal: 76.2mm (3.0") long by 22.2 mm (0.875") diameter sodium iodide, thalium activated scintillation crystal in ruggedized, sealed aluminum mount.

All other characteristics are the same as Model G375/A.

1-10.0 CONSUMABLES AND SPARE PARTS

Item	Description	Part No.	Quantity*
1.	Chart Paper	BP-10	3 Boxes
2.	Pen Cartridge-Black (Center Nib, long)	120-430	5 Each
3.	Pen Cartridge-Black (Nib on Right, long)	130-430	5 Each
4.	Pen Cartridge-Red (Offset Right, long)	130-430	5 Each
5.	Adjustment Screwdriver	R-3324	1 Each
6.	Rubber Cable Wiper Balls	RB-1.875	2 Each
7.	Surface Electrode Assembly	B-500K 0125	1 Each
8.	Mecca Sockets	2670-6	3 Each
9.	Mecca Boot	2458-1	1 Each
10.	A. C. Charging Cable	A-500K 0124	1 Each
11.	12vdc Charging Cable	B-500K 0108	1 Each
12.	Cablehead Protector Cap	B-2000 0108	1 Each
13.	Probe Top Protector Plug	B-2000 0107	1 Each
14.	Hand Crank for Winch		1 Each
15.	O-Ring (for cable head)	2-115	1 Each
16.	Nylon Accessory Storage Bag		1 Each
17.	Recorder Protective Cover		1 Each
18.	Operation and Maintenance Manual (1000-C)		1 Each
19.	Winch-Recorder Shipping Case		1 Each
20.	Probe-Accessory Shipping Case		1 Each
21.	Standard Combination Probe	G375/A	1 Each
22.	Backpack Frame		Optional
23.	1" O.D. Combination Probe	G375A-1.0	Optional
24.	Stratigraphic Combination Probe	G375/AS	Optional
25.	Filtered Combination Probe	G375F/A	Optional

When logging in extremely remote areas, you may wish to consult Mount Sopris for a list of spare parts (including both electronic and mechanical components to allow repairs to be made in the field.

*NOTE: This quantity refers to the number of pieces shipped with each unit initially.

SECTION II

OPERATING INSTRUCTIONS

2-1.0 Incoming Inspection

The Model 1000-C should be unpacked and inspected as soon as possible. Check externally for broken knobs, bent shafts or levers, damaged connectors, etc. Also check for scratches, dents, or any gross misalignment of cases, shafts, plates, etc.

A quick incoming test may be performed as follows:

2-1.1 Chart Drive and Depth Measuring System

Shift the CHART DRIVE lever (fig. 1-13) to ON. CAUTION: do not force the lever. If necessary, rotate the CHART ADVANCE wheel (fig. 1-22) slightly while pushing the CHART DRIVE lever to ON. Reset the DEPTH odometer (fig. 1-12) to zero with the RESET wheel (fig. 1-11) Note the position of one of the pens on the chart, and rotate the MEASURING WHEEL (fig. 1-23) counterclockwise exactly 10 revolutions. The chart will move 3 cm and the DEPTH odometer will read 9997.0 meters (9991.0' on English models). Rotating the MEASURING WHEEL clockwise exactly 10 revolutions brings the chart and DEPTH odometer back to zero.

2-1.2 Recorder-Instrument Circuitry

Connect one of the combination probes to the cable head as shown in figure 4. Connect the surface electrode to the front panel connector (fig. 1-10) and short this electrode to the electrode on the probe (fig. 4). Turn the CPS/DIV. switch (fig. 1-1) to CAL, gamma DISPLACEMENT switch (fig. 1-3) to zero, and the LOG SELECTOR (fig. 1-9) to γ. The right-hand pen will go completely off scale to the right, and the left pen will slowly go to full scale ($\sim$7 seconds). Depress the left ZERO button (fig. 1-16) and see that the left ZERO control (fig. 1-15) adjusts the pen to the left and right of zero (left-hand margin). Set the left pen to zero. Turn the CPS/Div. switch to 10 (100 cps full scale). The recorder will indicate the level of background radiation. Turn the LOG SELECTOR to POWER OFF and set the following controls as indicated: POLARITY switch (fig. 1-5) to "+", BIAS control (fig. 1-6) to zero, MV/DIV. switch (fig. 1-4) to 10, Ω/DIV. switch (fig. 1-7) to 10, and resistance DISPLACEMENT control (fig. 1-8) to zero.

Turn the LOG SELECTOR to SP-R. The left pen (SP) will read near the left margin, and the right pen (R) reads up scale about 8 cm. If the right pen goes off scale to the right, check the connection between the surface electrode and probe electrode. Depress the right ZERO button (fig. 1-19) and see that the right ZERO control (fig. 1-18) will adjust the pen both left and right of the center of the chart. The zero line for the right pen is the center of the chart. Set the right pen on "zero". Turning the BIAS control clockwise will cause the SP pen to go up scale (to the right) on "+" POLARITY and down scale on "-" POLARITY.

Turning the resistance DISPLACEMENT control clockwise causes the R pen to go down scale.

Should any of the aforementioned tests fail, check to see if the LOW BATTERY indicator (fig. 1-14) is on (LOG SELECTOR on γ or SP-R); if it is, recharge the batteries (refer to sec. 1-8). Otherwise, refer to section 3-9. It is normal for the LOW BATTERY indicator (fig. 1-14) to flash as the LOG SELECTOR is switched from POWER OFF to γ or SP-R.

2-1.3 Storage

The logger should be stored in a temperate, dry area if possible. The temperature range for storage is -40° C (-40° F) to 70° C (158° F). All protective covers should be in place and the units stored in their shipping containers. The batteries should be given a full charge (12 to 14 hrs.) prior to storage and re-charged (at least 8 hours) every six months.

2-2.0 OPERATING CONTROLS

Refer to Fig. 1	Control	Function
1.	CPS/DIV:	Gamma range switch, selects the number of counts per second per division (1cm) to be recorded on the chart. In the CAL. position, the internal calibration oscillator is connected to the input of the gamma circuitry and causes the pen to go full scale (right-hand margin).
2.	Gamma CAL.:	20 turn screwdriver adjustment to set the gamma pen on full scale with the CPS/DIV. switch in the CAL. position.
3.	Gamma DISPLACEMENT:	Displaces the gamma pen down scale (to the left). The amount of displacement is equal to the CPS/DIV. switch setting (100/div. maximum) multiplied by the gamma DISPLACEMENT switch setting i.e., if the CPS/DIV. switch is on 20 and the DISPLACEMENT switch is on 15, the pen is displaced 300 cps (1½ times full scale) to the left. CAUTION: This switch must be kept on zero when the CPS/DIV. switch is on 200 or above.
4.	MV/DIV.:	Self Potential range switch. Selects the number of millivolts per division (1cm) to be recorded. Full scale for the SP pen is assumed to be at the center of the chart.
5.	BIAS POLARITY:	Selects the polarity of the bias signal fed to the SP circuit.
6.	BIAS:	Controls the amount of bias signal fed to the SP circuit. The BIAS range is 0 to 500 mv.
7.	/DIV.:	Resistance range switch. Selects the number of ohms per division (1cm) to be recorded. Zero for the R pen is assumed to be the center of the chart; full scale is the right-hand margin.
8.	Resistance DISPLACEMENT:	Controls the number of ohms and R pen is displaced to the left. The range is 0 to 500 ohms.

Refer to Fig. 1	Control	Function
9.	LOG SELECTOR:	Controls the power to all circuitry and selects gamma or SP-R operation. This switch must be in POWER OFF to allow charging.
10.	BATTERY CHARGE-SURFACE ELECTRODE:	A connector which provides connections for all battery charging and the surface electrode.
11.	RESET:	Allows resetting DEPTH odometer to zero.
12.	DEPTH:	Displays the amount of cable which has been spooled off the winch drum from the time the odometer was reset.
13.	CHART DRIVE:	Controls multi-tooth clutch to engage or disengage chart. The chart will move at a ratio of 1 meter of borehole to 1 centimeter of chart. CAUTION: Do not force the CHART DRIVE lever to ON. If necessary, rotate the CHART ADVANCE wheel slightly (to line up a multi-tooth clutch) while pushing the lever to ON.
14.	LOW BATTERY:	Indicator will come on when batteries are low. A maximum of one hour of use remains after the indicator comes on.
15 & 18.	ZERO control:	Adjusts associated pen to zero. Zero for the left pen is the left-hand margin; zero for the right pen is the center of the chart.
16. & 19.	ZERO button:	When depressed, it shorts the input of the associated servo amplifier to ground (causing the pen to read zero).
17. & 20.	SERVO GAIN:	Adjusts the amount of gain in the feedback loop of the servo amplifier.
21.	PEN LIFTER:	Mechanically raises and lowers both pens.
22.	CHART ADVANCE:	Thumbwheel to manually move the chart. CAUTION: The CHART DRIVE must be OFF.

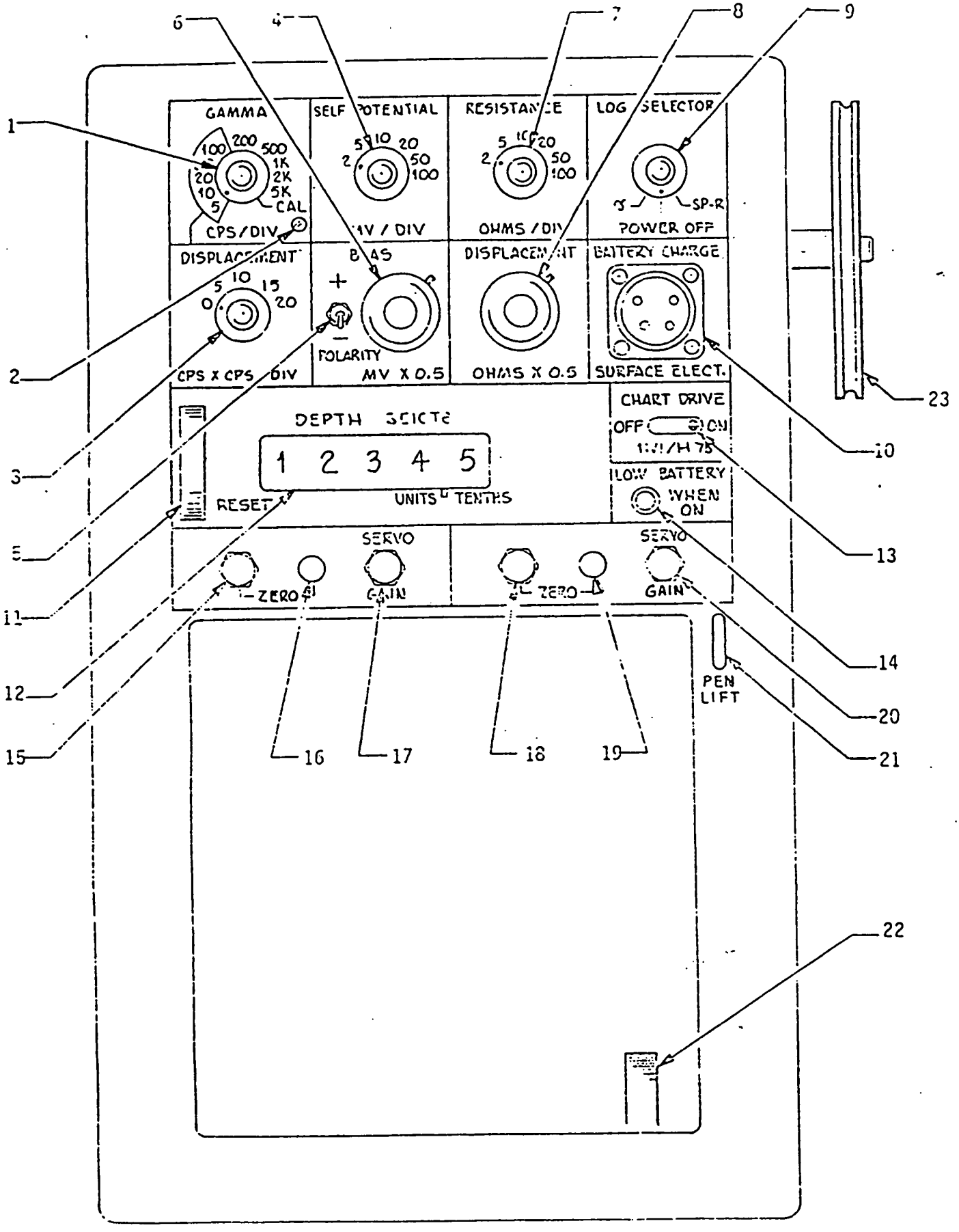


Fig. 1 Recorder Front Panel

2-3.0 Replacing Chart Paper & Pens

2-3.1 Loading Chart Paper (refer to figure 3)

It may be easier, but not necessary, to remove chart pens before loading recorder with new chart paper.

Shift the CHART DRIVE lever (refer to figure 1) to "OFF" and open the side door. Rotate chart paper thumb wheel clockwise while pushing up, from the inside, on the front end of the Teflon paper holder. The rotating sprockets will catch the paper holder and lift it up and off of the front sprocket roller. The paper holder may rub along the left side of the opening in the front panel.

The bottom end of the paper holder remains inside the recorder and around the rear sprocket roller. Do not attempt to completely remove paper holder from recorder.

Load a new packet of chart paper into recorder and thread paper up and over the front roller. Rotate thumb wheel clockwise and feed the chart paper until it engages the rear roller. Be sure the paper is flat and properly aligned on sprockets before lowering the front end of the paper holder. Maneuver paper holder down over the front roller until it catches on sprockets. The thumb wheel may then be rotated in a counter-clockwise direction. It may be necessary to hold the pens up to clear the paper holder. The paper holder will then snap into place.

Use thumb wheel to feed chart paper through recorder and into take-up magazine. Make note of which way chart paper unfolds, so that you can get paper refolding into original position.

CAUTION:

Take care not to bend the paper holder. The stretching required for the paper holder to snap on and off of roller is normal. When the paper holder is in its normal operating position, it should fit slightly loose. If the paper wrinkles or tears, or if the paper holder has a tendency to lift up during operation, the paper holder has become bent, and must be resored to its original shape.

2-3.3 Pens (Refer to Fig.1)

Turn LOG SELECTOR to POWER OFF. The pens can then be removed by grasping the front of the pen cartridge and pulling straight out. Replace with a fresh cartridge in the reverse order. When not in use, the protective caps should be kept on the pen tips to prevent the pens from drying out.

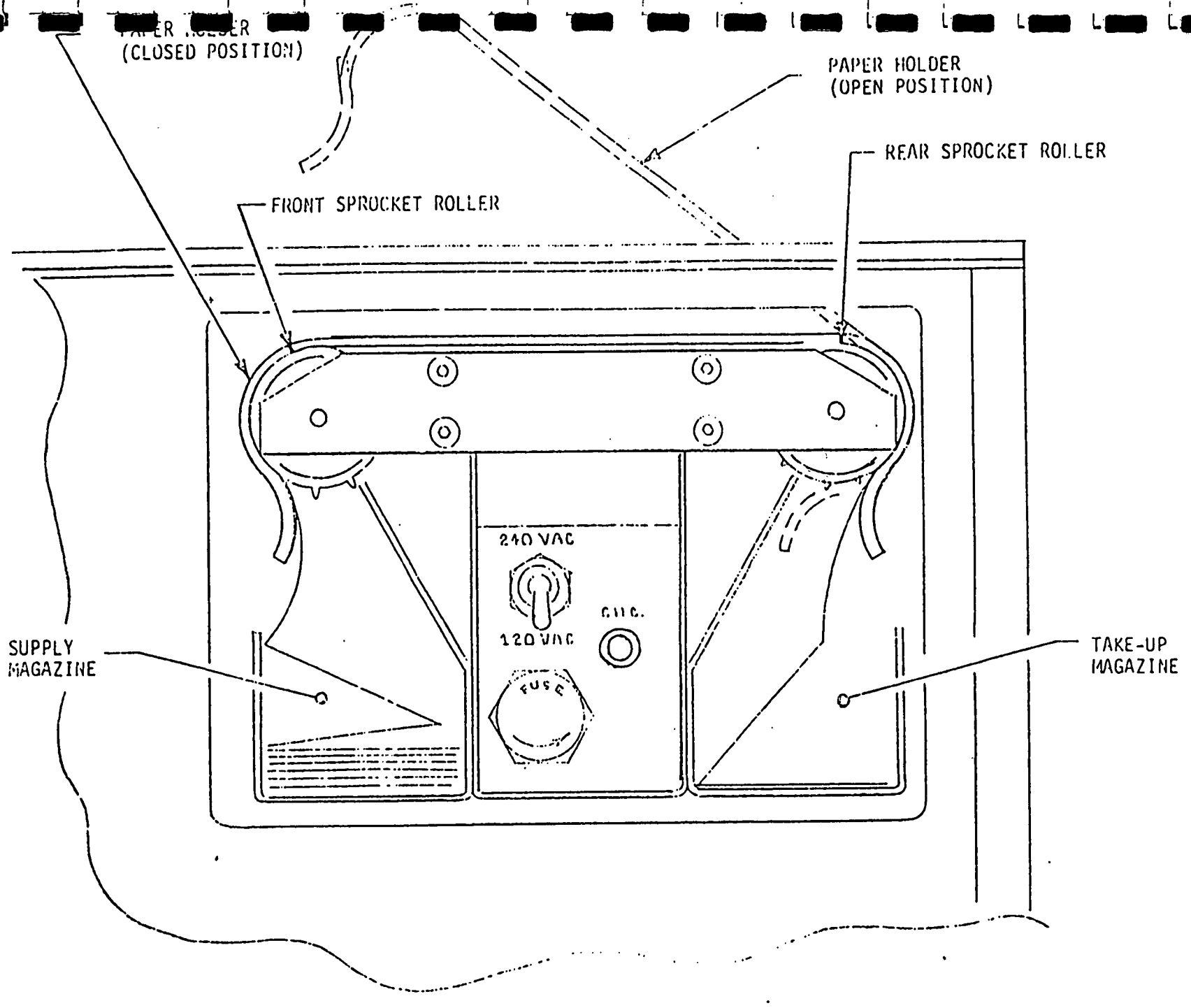


Fig. 3 CHART TRANSPORT ASSEMBLY (A)

2-4.0 Logging a Borehole

INTRODUCTION

2-4.1 The main objective for the operator of a logging unit is to produce an accurate, informative, and easy-to-read log. This means the pens make maximum use of the available chart space (full chart for Gamma and half chart each for SP and R), and do not overlap or criss-cross so much that they confuse the log. To do this requires selecting proper scales and setting bias or displacement controls properly (there is no substitute for experience). Using black pens for SP and R and a red pen for Gamma also helps keep the record straight. Since the two pens are non-overlapping, they may collide while logging SP and R. It is almost impossible to prevent this, so (if they do not separate naturally within a meter or so) the pens must be separated, using the SP BIAS control and/or R DISPLACEMENT control. Turning the R DISPLACEMENT control clockwise will cause the R pen to move to the left. Turning the SP BIAS control clockwise, with the POLARITY switch on "+", will move the SP pen to the right. With the POLARITY switch on "-" the pen will move to the left when the SP BIAS control is turned clockwise. A sample of a good log is illustrated in figure 5A and a poor log in 5B.

All three logs (Gamma, SP and R) can be run in one trip in the borehole by logging SP and R down, and Gamma on the way out. However, because of the added difficulty with this method (the hole cannot be pre-viewed on the way down to set up and SP and R controls, extra chart paper must be fed into the take-up magazine because the paper will run backwards while going down hole, etc.) it is recommended only when logging in poorly consolidated material, or when time is the ruling factor.

Prior to going to the drill site, check to make sure you have: fully charged batteries, a charging cable, spare pens (in both styles and colors), the winch crank, probe(s), extra chart paper, cable wiper balls, mud electrode, Gamma calibration screwdriver, electrical tape, and silicone grease (for O-Ring on cable head).

The following list is intended as a guideline to log a typical borehole, using the two trip method. (The probe is lowered to the bottom of the hole and Gamma is logged while coming out; the probe is then lowered a second time to log SP and R.

2-4.2 Clear the area around the borehole to give you a relatively clean, dry place to work.

2-4.3 Release the BRAKE and unspool a couple of meters of cable to give you enough slack to connect the probe and set up the tripod as shown in figure 2.

2-4.4 Place the logger on the borehole. Depending on the site, you may have to use scrap lumber, rocks, etc. to make the unit as level as possible.

2-4.5 Shift the CHART DRIVE lever to OFF. Check to see if there is enough paper in the supply magazine to log the hole.

2-4.6 Remove the protector plug from the probe and check to see that the threads and area around the contact are clean. Remove the knurled cable head protector (clean the threads and contact if dirty), apply a generous amount of silicone grease to the O-ring, and screw the probe to the cable head HAND TIGHT ONLY--do not use wrenches. The probe must be insulated with electrical tape as shown in figure 4.

2-4.7 Bury the mud plug by digging a small hole, putting the mud plug in, and filling the hole with dirt and fresh water. The mud pit will provide a good ground on freshly drilled holes. It may be necessary to use salt water to obtain a good ground in extremely arid areas. This must be noted on the log heading, as it usually reverses the polarity of the SP log.

2-4.8 Load the chart paper (refer to section II, paragraph 3) and put your log heading on it. A typical log heading might contain the unit serial number, probe serial number, probe K factor, type of logs run and their scales, hole number and location, operator's name, and the date.

2-4.9 The first log to run is Gamma, because it is usually considered most important, and depending on conditions in the borehole, one run may be all you can get. For clarity, the Gamma should be run with a red pen (SP and R in black). Remove the protective cap from the gamma pen and switch the LOG SELECTOR to γ . The right-hand pen will automatically go off scale to the right to allow full use of the chart for gamma.

2-4.10 The SERVO GAIN control (one for each channel) should be set as high as possible (clockwise) without having the recorder pens oscillate. If a recorder pen starts to oscillate, reduce the gain (counterclockwise) just to the point where the oscillation stops.

2-4.11 To check the gamma calibration, turn the CPS/DIV. switch to CAL. and alternately check for zero on the left-hand margin and full scale on the right-hand margin. Depress the left-hand ZERO button and adjust the ZERO control so the pen rests on the left-hand margin. Release the button, and the pen will come to rest near the right-hand margin. Adjust the pen for full scale with the CAL. screwdriver adjustment. Depress the ZERO button and again check for zero and then full scale to insure accurate calibration. The calibration procedure routinely should be checked before each hole.

2-4.12 Make certain the brake is set, and lower the probe into the borehole. Position the logger as necessary to center the cable in the hole. Slip the winch crank on the 1:1 ratio shaft, release the brake and crank the top of the cable head back up to ground level. Set the brake.

2-4.13 Reset the DEPTH odometer by rotating the depth RESET wheel upward, and then set in the distance from the top of the cable head, (ground level) to the reference point by manually rotating the measuring wheel. The gamma log and SP-R logs will be displaced by the distance between the center of the scintillation crystal to the center of the electrode (See Fig. 4). If this distance is considered significant, it may be compensated for by sliding the pens in or out slightly as the case may be. A good range for γ exploration is 20 cps/DIV.

2-4.14 You are now ready to lower the probe to the bottom of the hole. CAUTION: do not allow the probe to freewheel down the hole. Holding the winch crank, release the brake and crank the probe to the bottom of the hole. The bottom will be detected by the sudden loss of weight. The approximate depth can be obtained from the drilling crew.

2-4.15 When you hit bottom, take the slack out of the cable and set the brake. Move the winch crank to the 4:1 ratio shaft.

2-4.16 Adjust the chart with the CHART ADVANCE wheel so the pen will cross a major division on the chart (every 1 cm) at the same time the depth odometer indicates a whole number of meters (or at 3' intervals on English models). This makes reading the chart easier because each centimeter line will be an even depth reading i.e., every meter 273.0, 272.0, 271.0, (or 273.0, 270.0, --- 6.0, 3.0, 0.0 on English models). Shift the CHART DRIVE to ON. CAUTION: Do not force the CHART DRIVE lever. If necessary, rotate the CHART ADVANCE wheel very slightly (to line up a multi-tooth clutch) while shifting the CHART DRIVE lever to ON.

2-4.17 Check to be sure a good cable wiper ball is in place. If not, rotate the old one (or install a new ball) and cut a slice for the cable to go through.

2-4.18 You are now ready to make the first log (gamma) in the hole. Lower the pen, release the brake, and begin to crank the probe back out. A good speed for general logging is 4M/min. (15'/min.). This can be approximated by making one revolution of the crank every 2 seconds. As you come out of the hole, an attempt should be made to keep the cable as neat and level across the winch drum as possible.

2-4.19 If a gamma anomaly is encountered, the gamma pen will go off scale to the right. Continue to log until the pen comes back on scale and is reading the normal background gain. Stop cranking, raise the pen, and, while observing the pen, crank back down through the anomaly, selecting a scale which will keep the pen on scale in the upper half of the chart. Make a note of this range setting on the chart. When the pen is reading background (below the anomaly on the new scale), put the pen down and crank back up through the anomaly until the pen again returns to background. A re-run should be logged at 1 to 2 m/min (3 to 6'/min.). When the pen has returned to background, raise the pen, switch back to the original gamma range, lower the pen and continue logging the hole. A sample of a re-run is shown in figure 5c.

2-4.20 Upon reaching the top of the hole: 1. raise the pen, 2. set the brake, 3. switch the LOG SELECTOR to POWER OFF, 4. change the left pen to black (with the nib on the right) and remove the protective caps from both pens, 5. move the winch crank to the 1:1 ratio shaft, 6. switch the LOG SELECTOR to SP-R, 7. set the MV/DIV. and ohms/DIV. switch to 100, and the BIAS and DISPLACEMENT controls to zero. DO NOT shift the CHART DRIVE to OFF; the bi-directional chart drive will automatically follow the probe back downhole for the second run (SP and R).

2-4.21 Check the zero on both pens. The SP pen zero is on the left margin and the R pen zero is at the center of the chart. The scales and bias/displacement setting should be set on the trip downhole. The R pen will be off scale to the right until the water level is reached (when it comes back on scale). At this time you should start alternately lowering the OHMS/DIV. switch, to make the pen more active (larger fluctuations), and adjusting the DISPLACEMENT control to center the fluctuations on the right-hand half of the chart. The MV/DIV. switch and BIAS control are set in a similar manner; the POLARITY switch changes the polarity of the bias voltage to allow logging positive or negative SP. The SP log should cover the left-hand half of the chart and the R log the right-hand half.

2-4.22 You can now crank the probe back down the hole, setting the SP and R controls as you go. If the pens come into contact with each other, allow a few seconds for them to come back on their own, then if necessary, adjust the BIAS and/or the DISPLACEMENT controls. Turning the DISPLACEMENT control clockwise moves the R pen to the left; turning the BIAS control clockwise moves the SP pen to the right on "+" polarity and to the left on "-" polarity.

2-4.23 When you come out of the water the SP and R pens will rapidly go off scale. Raise the pens and turn the LOG SELECTOR to POWER OFF. The probe should be cranked out of the hole as fast as possible, to reduce time in the hole and the chance of getting stuck.

2-4.25 Retrieve the mud plug and wipe excess mud from it and the winch assembly. After storing the cable head and pulley assembly, tighten the cable slightly to prevent it from loosening on the drum and becoming tangled during shipping.

2-4.26 Make sure the LOG SELECTOR is in the POWER OFF position and the winch brake is on before storing or transporting the logger.

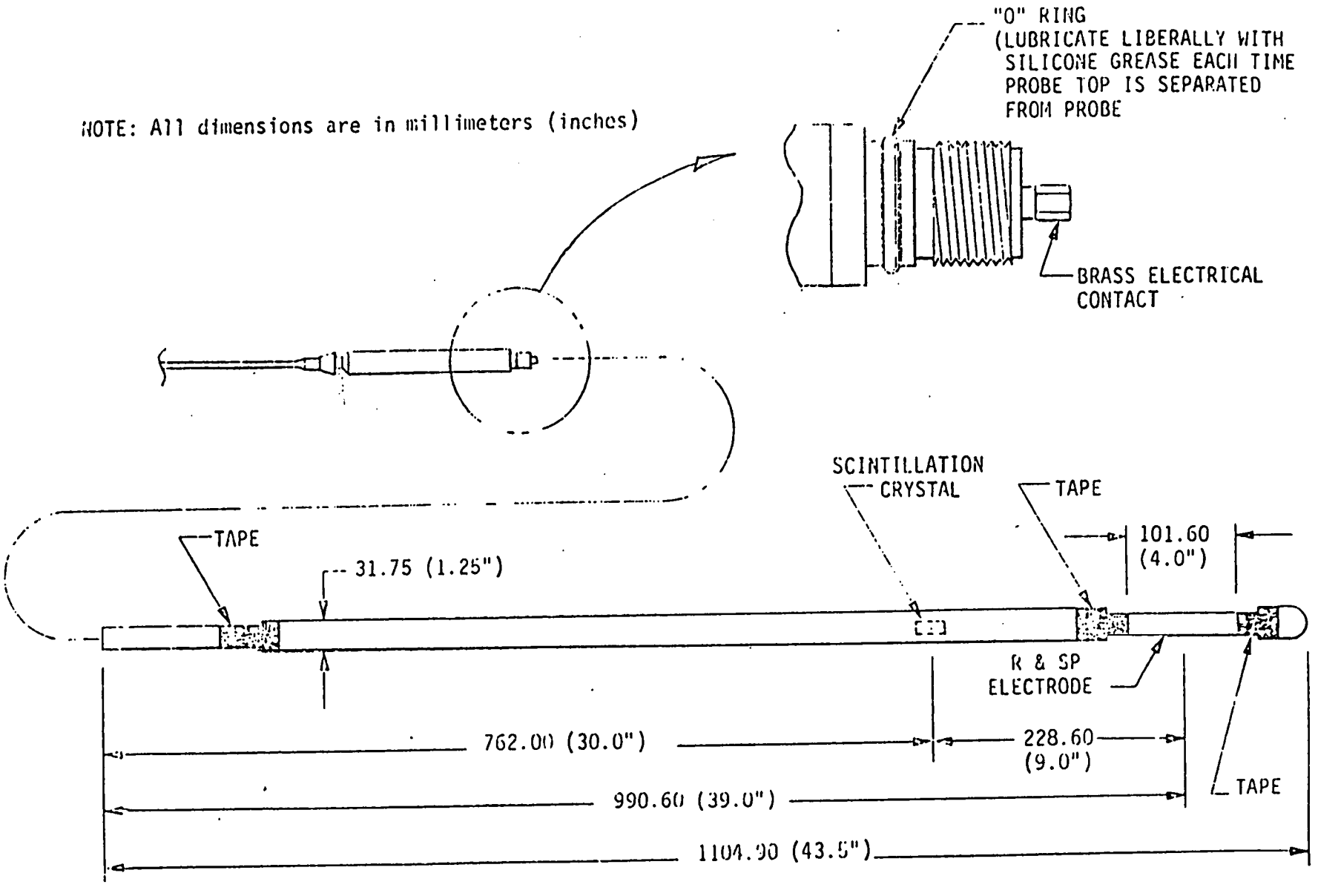
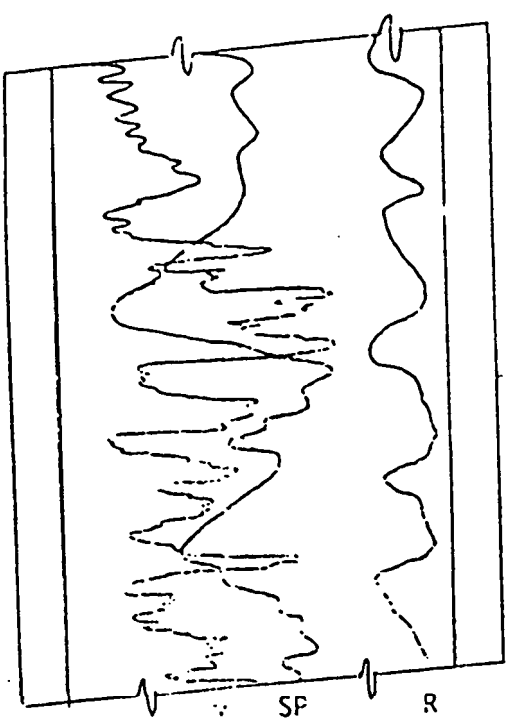
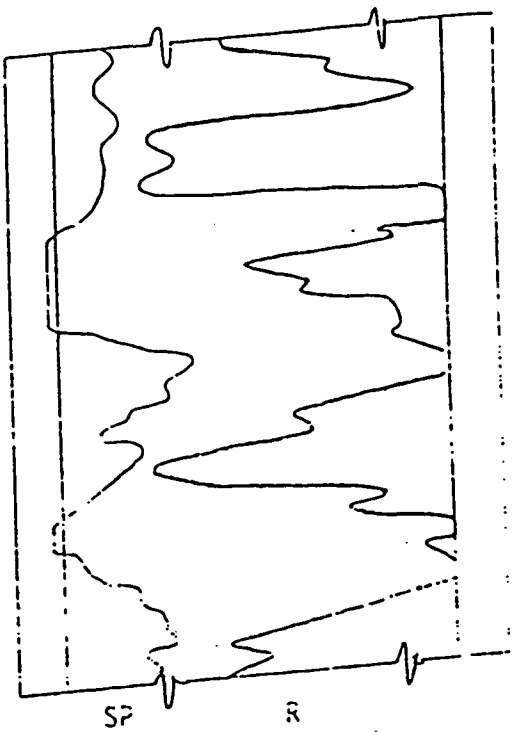


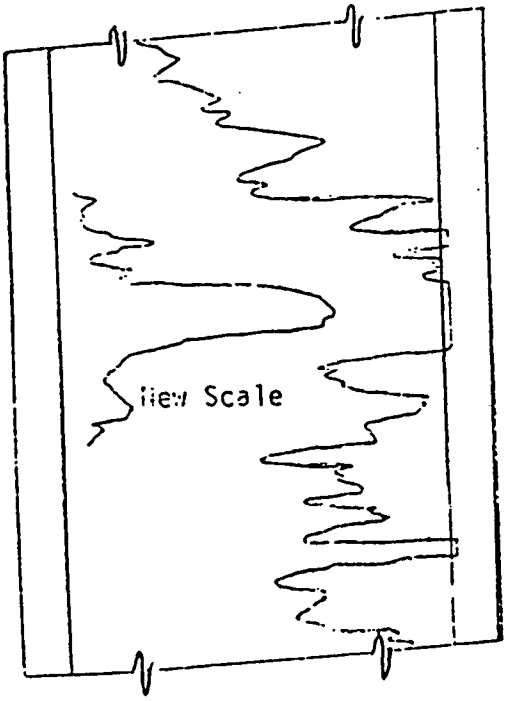
Fig. 4 PROBE DIMENSIONS



Example of a
good log
Fig. 5A



Incorrect Biasing
on the SP.
Range too high
on the R
Fig. 5 B



Re-Run
Fig. 5 C

Figure 5
Sample Logs

2-5.0 Operating Precautions

2-5.1 Keep the cable head threads and brass contact clean and the O-ring coated with silicone grease.

2-5.2 The probe top threads and area around the center connector must be kept free from dirt and grit.

2-5.3 If you become stuck in the hole DO NOT force the winch. If a moderate amount of pull from the winch will not free the probe a cable gripper and a jack or lever arm will be required.

2-5.4 When the CPS/DIV. switch is on 200 or above, the gamma DISPLACEMENT switch must be on 0 or the batteries will prematurely discharge.

2-5.5 DO NOT allow the probe to freewheel to the bottom of the hole, as damage may result to the photomultiplier tube and/or scintillation crystal.

2-5.6 In logging holes near the length of the cable, do not unspool cable completely. A minimum of 5 full wraps must be kept on the winch drum.

2-5.7 Keep the cable neat and evenly wound across the winch drum to prevent kinks and possible short circuits.

2-5.8 Do not allow the cable to kink. This causes premature wear, and could cause a short between the center conductor and the steel armor.

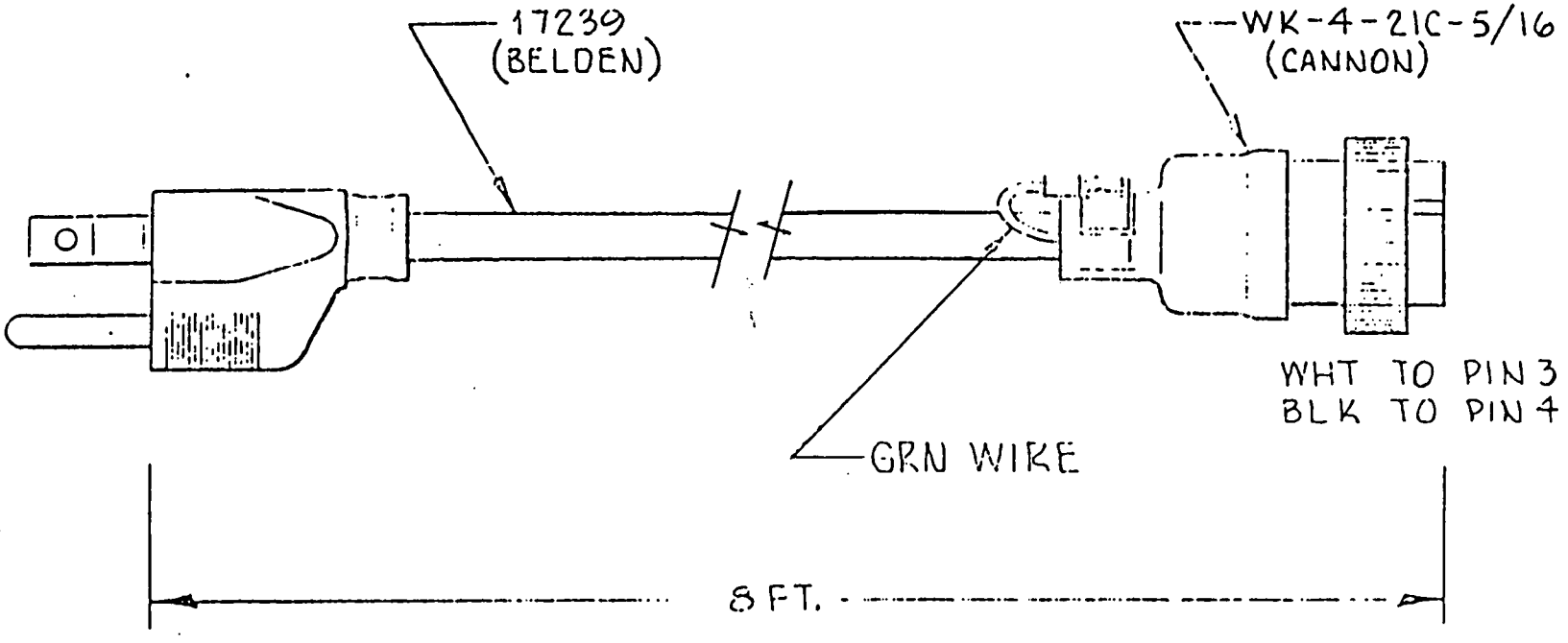
2-5.9 Handle probe with care. Avoid rapid changes in temperature and sharp blows, especially in a lateral direction.

2-5.10 LOG SELECTOR must be in POWER OFF when changing or removing probes.

2-6.0 Battery Life & Re-charging

The life of a nickel cadmium battery is largely dependent on temperature; however, there are many other factors involved (discharge rate, type of charger, etc.). The cells used in the Model 1000-C nominally give 1000 complete charge-discharge cycles. The charging system used is of the constant current type, and can be left on for an indefinite period of time with no danger of overcharging. A sensor in the charger switches automatically for 120 or 240 volt a.c. operation. It is recommended that the unit be left on charge when it is not in use, and recharged at least every six months when in storage.

2-6.1 Connect the proper charging cable, for the voltage source available, to the BATTERY CHARGE connector. If the 12 vdc cable is used, the red lead connects to the positive (+) terminal of the source, and the black lead to the negative (-). If the a.c. cord is to be used, MAKE CERTAIN the CHARGING VOLTAGE SELECTOR SWITCH (located inside the chart access door) is in the PROPER POSITION. If used on 240 v. it will be necessary to cut the cord near the a.c. plug and install an appropriate connector. Refer to figure 6 for the proper connections. Connect the cable to the power source and observe the charge light (located inside the chart access door) come on to indicate proper charging. Should the charging indicator fail to light, check the connections to the power source, make sure the power source is active, and check the fuse (a.c. operation only) located inside the chart access door. NOTE: If the polarity of the 12 vdc cable is reversed, no damage will result, but the batteries will not charge.



Pin 4----------Black----------120 v. or 240 v. (Hot)
Pin 3----------White----------120 v. or 240 v. (Return)
Conn. Shell----Green----------Earth Ground

Pin 1----Black-------Common
Pin 2----Red---------+12 to 14 v.d.c.

Fig. 6 Battery Charge Inputs

WATER AND CASING FACTOR TABLES

January 5, 1982

For Probe Model Numbers: G375/A, HLP-2375, ALP-4979

(Probes with 2SHA6/0.5-NaI(tl) PMT/Crystal assemblies and 0.065" SS wall thickness)

TABLE 1 - WATER FACTORS

Hole Diameter (Inches)	Correction Factor
2.25	1.027
4.5	1.099
6.5	1.167
8.5	1.238

TABLE 2 - CASING FACTORS (4.5" dia. hole)

Casing Wall Thickness (Inches)	Correction Factor
0.0625	1.196
0.125	1.329
0.1875	1.524
0.25	1.691
0.375	2.021

These tables supersede all previous water and casing factor tables.

APPENDIX F

FIELD SCREENING OF VOLATILE ORGNAICS

FIELD ANALYSIS OF VOLATILE ORGANICS

Scope and Application: This method covers the determination of the following organic compounds in water and soil gas.

Compounds:

- Benzene
- Bromodichloromethane
- Bromoform
- Chloroform
- Chlorodibromethane
- 1,1-Dichloroethane
- 1,2-Dichloroethane
- *1,1-Dichloroethene
- *1,2-Dichloroethene
- Ethyl benzene
- Methylene Chloride
- *Tetrachlorethene
- *Toluene
- 1,1,1-Trichloroethane
- *Trichloroethene

*Target Compounds

Method: Headspace - Gas Chromatographic/Photoionization and Hall Electrolytic Conductivity Detection

Reference: EPA Test Methods 601 and 602 with modifications

Detection Limits: Headspace (water): 1.0 - 50 ug/L; Soil Gas: 5-10 ng injected)

Quality Control:

1. Each analytical run should begin wtih a target headspace standard curve consisting of 50, 10, 5 ug/L and a blank. Every eleventh analysis threafter and the last sample analyzed should also be standards. Continuous calibration standards should be within 30% of the original standards or a new standard must be prepared and samples analyzed since the last check standard reanalyzed.

2. After the initial 3-points calibration with target headspace standards is done, the following 1-point calibration will be performed:

   2.1 Run a 1-point, 50 ug/L non-target headspace standards.
   2.2 Direct inject 5 ul of a 5 ug/ml target standard (25 ng) for a 1-point soil gas curve.
   2.3 Direct inject 5ul of 5ug/ml non-target standards (25 ng) for a 1-point soil gas curve.

3. A minimum of 10% duplicate samples should be analyzed. If less than 10 samples are analzyed, a duplicate sample should still be analyzed. Duplicates should be within 15%.

4. New stock standards should be prepared monthly in the laboratory. New secondary standards should be prepared weekly in the laboratory and brought to the field location while maintaining a temperature of approximately 4°C (iced).

Sample Collection and Handling: Water samples are to be collected in 40 mL vials with open screw-caps and teflon faced silicone septa. They should be collected so that no headspace remains in the bottle. Soil gas samples are to be collected in 250 mL glass bulbs. Sample should be collected in a manner to ensure the complete purging of the bulb. All samples should be protected from sunlight and transported to the field lab as soon as possible.

Reagents and Apparatus:

1. Open screw cap 40 mL vial (Pierce #13075 or equivalent). Detergent washed, distilled water rinsed and dried at 105°C before use.

2. Septum - Teflon-faced silicone (Pierce #12722 or equivalent). Detergent washed, distilled water rinsed and dried at 105°C before use.

3. 250 mL gas sampling bulbs.

4. Gas chromatograph - Varian 3400 equipped with PID and Hall detectors in series.

5. Column 1 - 8 ft x 1/8 in. stainless steel, packed with 1% SP 1000 on Carbopack B (60/80 mesh).

6. Dual-channel Integrator/Recorder.

7. Syringes -
   1 and 5 mL gas tight, fitted with shut-off valves and 22 gauge needle.
   10, 100, and 1,000 m/L gas tight.

8. 30 m/L Serum type reaction vials (hypo) with teflon lined septa and seals.

9. Balance - ±0.0001 g - (Cahn TA4200).

10. Balance - ±0.01 g - (Sartorius, 1202 MP).

11. Reagent water - organic free water or cold tap water which has been shown to be organic-free at the method detection limits.

12. 25 mL TC graduated cylinders.

13. Constant temperature water bath - 50°C.

14. Volumetric flasks - assorted.

15. Pipettes - assorted.

16. Certified gas standard solutions - 200 mg/L (Supelco).

Standard Preparation:

1. Stock Standard Solution: The stock standard solution is prepared at 5,000 mg/L methanol from pure standard materials (exceptions: 2-chloroethylvinylether is prepared at 100 g/L; 1,2-DCB, 1,3-DCB and 1,4-DCB are prepared at 25 g/L; Bromoform is prepared at 10 g/L). Correction for purities of less than 99% are made. Gas standards are purchased as a certified solution at 200 mg/L.

   1.1 Add about 20 mL of methanol to a 25 mL volumetric flask. Allow the flask to stand unstoppered until the methanol on the neck of the flask has dried.

   1.2 Tare the flask on the analytical balance.

   1.3 Using a 100 uL syringe add the reference material to the flask. Make sure the drops fall directly into the methanol without contacting the neck of the flask.

   1.4 Determine the amount of reference material added. Rinse the syringe with methanol, tare the flask, and add the next standard.

   1.5 After all the reference materials are added, fill to volume with methanol, cap, and invert to mix.

Secondary Standard Solution: Prepare secondary standards (target and non-target compounds) according to the following scheme.

Standard	Amount	Final Volume	Concentration
5000 ug/ml	1 ml	10 ml	500 ug/ml
500 ug/ml	1 ml	10 ml	50 ug/ml
50 ug/ml	1 ml	10 ml	5 ug/ml
10 ug/ml	1 ml	10 ml	1 ug/ml

Dilute to volume with methanol.

Calibration:

Water Samples (Headspace):

1.0 Working Headspace Calibration Standards: Prepare working calibaration standards (target and non-target compounds) according to the following scheme:

Secondary Standard	Amount	Final Volume	Cocentration
500 ug/ml	20 ul	200 ml	50 ug/l
50 ug/ml	40 ul	200 ml	10 ug/l
50 ug/ml	20 ul	200 ml	5 ug/l
10 ug/ml	20 ul	200 ml	1 ug/l

Fill a 200 ml volumetric flask with reagent water to the mark. Directly inject the secondary standards into the water with an appropriate microliter syringe.

Invert each working standard 3 times, discard the first 10 ml in the neck of the volumetric and transfer aliquots of the freshly prepared working standards to 40 ml VOC vials, (no headspace) and capped.

2.0 Calibaration Procedure

2.1 Target Headspace Standards

2.1.1 Remove and discard 10 mL from a freshly prepared standard and place the vial now having 10 mL of headspace in a 50°C water bath insuring the water level in the bath is sufficient to equal the water level in the vial.

2.1.2 Allow time for equilibration of temperature (10 minutes).

2.1.3 Remove 5 mL of headspace for injection onto the gas chromatograph.

2.1.4 Construct a minimum 3-point standard curve of peak area response versus concentration for each of the compounds of interest.

2.1.5 A continuing calibaration check is performed after each set of 10 samples and as the last sample of the day. If the response for any of the target compounds varies from the expected response by more than 30%, a new calibration curve must be prepared.

2.2 Non-Target Headspace Standards

Run a 1-point, 50 ug/L headspace standard of non-target compounds.

Soil Gas Samples:

1.0 Working Soil Gas Calibration Standards: prepare working soil gas calibration standards (target and non-target compounds) according to the following scheme:

Secondary Standard	Amount	Final Volume	Concentration
500 ug/ml	1 ml	10 ml	50 ug/ml
50 ug/ml	2 ml	10 ml	10 ug/ml
10 ug/ml	5 ml	10 ml	5 ug/ml

Dilute to volume with methanol

2.0 Calibration Procedures:

2.1 Inject 5.0 uL of each of the working standard solutions into the gas chromatograph.

Construct a minimum 3-point standard curve of peak area response versus total nanograms injected for each of the compounds of interest.

A continuing calibration check is performed after each set of 10 samples and as the last sample of the day. If the response for any of the compounds varies from the expected response by more than ±30%, a new calibration curve must be prepared.

Sample Analysis

1.0 Water Samples:

1.1 Water samples are received in 40 mL VOC vials. Remove 10 mL of the sample from the vial.

1.2 The vials are placed in a 50°C water bath and allowed to equilibrate for 10 minutes.

1.3 Remove 5 mL of headspace for injection into the gas chromatograph.

1.4 If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller aliquot of headspace can be taken from a freshly prepared sample.

2.0 Soil Gas Samples:

2.1 Soil gas samples will be received in 250 mL glass bulbs. When received, they are allowed to equilibrate to the ambient air temperature.

2.2 Remove 5 mL of sample through the sampling septum and inject onto the gas chromatograph.

2.3 If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller aliquot is taken from the same sample.

Chromatographic Conditions

Column:

8 ft x 1/8 inch stainless steel, packed with 1% SP-1000 on Carbopack B (60/80 mesh).

Carrier Gas

Helium - Ultra High Purity Grade (Linde)
35 mL/min

Detectors (in series)

1. Photoionization 10.2eV

   Sensitivity - Range 11 x Attenuation 8
   Temperature - 240°C

2. Hall 700A

   Mode - Halogen
   Reactor Temperature - 1000°C
   Solvent Flow - 0.8 mL/min Methanol
   Hydrogen Flow - 60 mL/min

Injector

Temperature - 220°C

Oven*

Initial - 60° C-0 minutes
Rate - 20° C/minute
Final - 220° C/and held for 7 minutes

* Conditions listed can be varied as needed for changing applications. Relative retention times are found on Tables 1 and 2 using these conditions.

Target Headspace

Calculations:

1. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.

2. Prepare external standard calibration curves for each compound using at least three data points and linear regression analysis.

3. Calculate the concentration found in the samples from the calibration curves using the following equations:

$$ug/L = A \times DF$$

where: A = Amount of compound found in the analysis in ug/L (from linear regression). DF = Dilution factor.

Other VOC Headspace Calculations:

1. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.

2. Calculate the concentration of each parameter found in the samples using the following equation.

$$ug/L = \frac{R(samp)}{R(std)} \times C(std) \times DF$$

where: R(Samp) = Response of parameter in sample
R(Std) = Response of parameter in standard
C(Std) = Concentration of standard in ug/L
DF = Dilution factor

Soil Gas Bulb Calculations:

1. Review of the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.

2. Calculate the concentration of each parameter found in the samples using the following equation.

$$ng/L = \frac{R(samp) \times ng(std) \times DF \times 1000}{R(std) \times VL}$$

where: R(samp) = Response of parameter in sample
R(std) = Response of parameter in standard
ng(std) = ng (10^{-9} grams) of standard injected
VL = Volume of aliquot taken from bulb (in mL)
DF = Dilution factor

Data Reporting:

1. All results, standards conditions, and notes will be recorded in a bound field notebook.

2. All data generated by field G.C. will be considered as tentatively identified, with all concentrations being estimated.

3. All raw field data will be forwarded to Warzyn Engineering Inc. analytical laboratory for final review and archiving. A final summary report will be prepared with pertinent copies of the field notebook, and chromatograms included (see attached report form).

4. Analysis will be rejected for re-analyzed if:
   - duplicates are outside the 15% acceptable range.
   - continuing calibration varies greater than 30% of true value.

[jpl-600-22b]

TABLE 1

Target VOC Detection Limits
for Water Headspace

Compound	Replicates	Mean (1)	Standard Deviation	Method Detection Limit(2)
Toluene	7	2.49	0.208	0.65 ug/L
1,1-Dichloroethene	7	2.32	0.364	1.14 ug/L
Trans-1,2-Dichloroethene	7	2.08	0.332	1.04 ug/L
Trichloroethene	7	1.84	0.294	0.92 ug/L
Tetrachloroethene	7	2.38	0.310	0.97 ug/L

(1) Mean value for spike at 3 ug/L.
(2) Calculated D.L. according to Appendix A of EPA Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

TABLE 2

Other VOC's Detection Limits
for Water Headspace

Compound	Replicates	Detection Limit
Benzene	3	2.0 ug/L
Ethyl Benzene	3	2.0 ug/L
1,1,1-Trichloroethene	3	1.0 ug/L
1,1-Dichloroethane	3	2.0 ug/L
Chloroform	3	2.0 ug/L
Methylene Chloride	3	6.0 ug/L
1,2-Dichloroethane	3	5.0 ug/L
Bromodichloromethane	3	5.0 ug/L
Chlorodibromomethane	3	25 ug/L
Bromoform	3	50 ug/L

TABLE 3

Soil Gas Detection Limits

Compound	Replicates	Mean (ng)	Standard Deviation	Detection Limit (ng) (1)
Toluene	3	4.16	0.044	5.0
1,1-Dichloroethene	3	5.06	0.086	5.0
Trans-1,2-Dichloroethene	3	4.95	0.021	5.0
Trichloroethene	3	4.91	0.032	5.0
Tetrachloroethene	3	4.21	0.219	5.0
1,1,1-Trichloroethane	3	3.90	0.147	5.0
Benzene	3	7.57	0.085	5.0
Ethyl Benzene	3	6.08	0.301	5.0
1,1-Dichloroethane	3	3.99	0.216	5.0
Chloroform	3	4.02	0.132	5.0
Methylene Chloride	3	3.65	0.788	5.0
1,2-Dichloroethane	3	2.92	0.099	5.0
Bromodichloromethane	3	4.92	0.093	5.0
Chlorodibromomethane	3	8.77	0.180	10.0
Bromoform	3	8.95	0.118	10.0

(1) Detection limit is total nanograms injected into G.C. column.

TABLE 4

Volatile Retention Order

Photo Ionization Detector

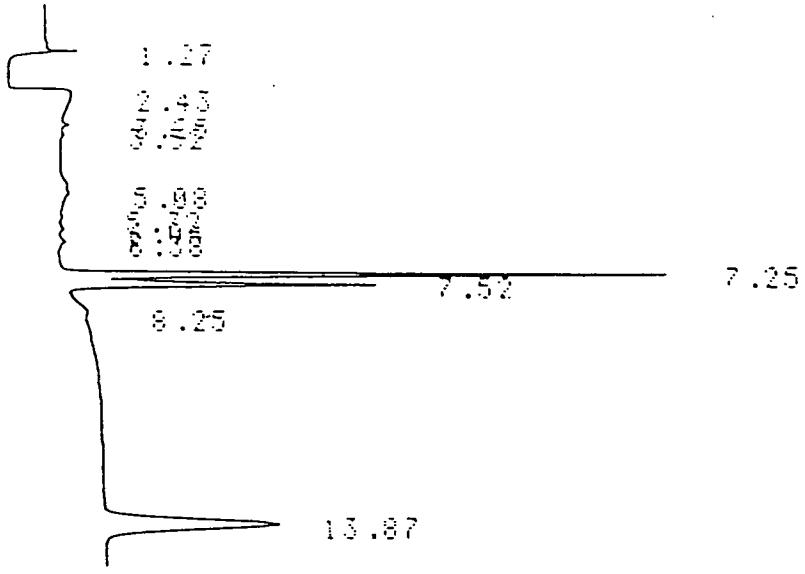
Parameter	Retention Time
1,1-Dichloroethene	4.24
Trans-1,2-Dichloroethene	4.98
Trichloroethene	7.23
Benzene	7.39
Tetrachloroethene	9.86
Toluene	10.81
Ethyl Benzene	13.84

Hall Detector

Parameter	Retention Time
Methylene chloride	3.26
1,1-Dichloroethene	4.29
1,1-Dichlorothane	4.77
Trans-1,2-Dichloroethene	5.04
Chloroform	5.21
1,2-Dichloroethane	5.49
1,1,1-Trichloroethane	6.12
Bromodichloromethane	6.40
Trichloroethene	7.29
Chlorodibromomethane	7.58
Bromoform	8.76
Tetrachloroethene	9.91

Non-targeted compounds PID detector

CH. 1 C.S 5.00 ATT 4 OFFS 10 07/29/87 08:24



D-2000 07/29/87 08:24

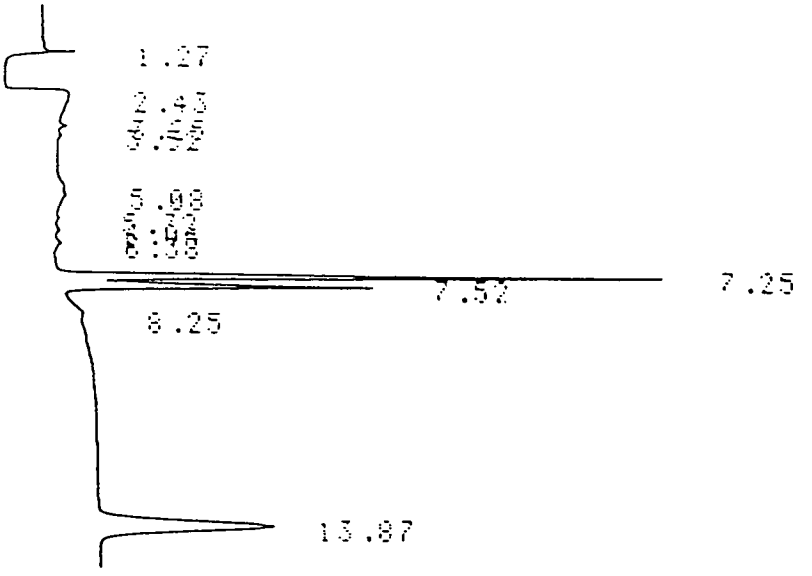
METHOD: PID TAG: 34 CH: 1

FILE: 1 CALC-METHOD: EXT-STD TABLE: 1 CONC: AREA

NO.	RT	NAME	AREA	CONC	BC
1	1.27		2463	0.002	BB
2	2.43		29535	0.029	BV
3	3.25		4060	0.004	VV
4	3.52		1398	0.001	VB
5	5.08		2370	0.002	BB
6	5.72		182	0.000	BB
7	6.04		335	0.000	BB
8	6.38		610	0.000	BB
9	7.25	BENZENE	47354	0.047	BV
10	7.52		30138	0.030	VB
11	8.25		1234	0.001	BB
12	13.87	ETHBEN	40199	0.040	BB
TOTAL			159898	0.159	

Non-targeted compounds PID detector

CH. 1 0.5 5.00 ATT 4 OFFS 10 07/29/87 08:24



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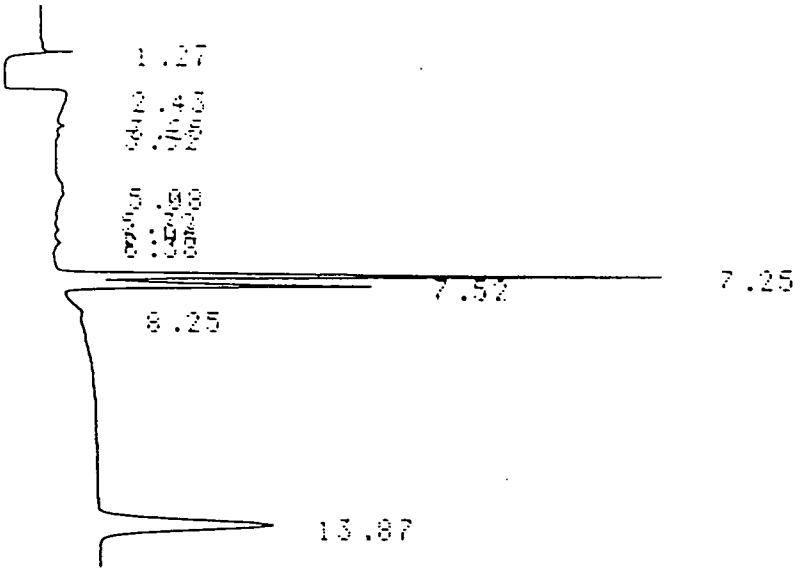
METHOD: PID TAG: 34 CH: 1

FILE: 1 CALC-METHOD: EXT-STD TABLE: 1 CONC: AREA

NO.	RT	NAME	AREA	CONC	BC
1	1.27		2463	0.002	BB
2	2.43		29535	0.029	BV
3	3.25		4080	0.004	VV
4	3.52		1398	0.001	VB
5	5.08		2370	0.002	BB
6	5.72		182	0.000	BB
7	6.04		335	0.000	BB
8	6.38		610	0.000	BB
9	7.25	BENZENE	47354	0.047	BV
10	7.52		30138	0.030	VB
11	8.25		1234	0.001	BB
12	13.87	ETHBEN	40199	0.040	BB
TOTAL			159898	0.159	

Non-targeted compounds PID detector

CH. 1 C.S 5.00 ATT 4 OFFS 10 07/29/87 08:24



D-2000 07/29/87 08:24

METHOD: PID TAG: 34 CH: 1

FILE: 1 CALC-METHOD: EXT-STD TABLE: 1 CONC: AREA

NO.	RT	NAME	AREA	CONC	BC
1	1.27		2463	0.002	BB
2	2.43		29535	0.029	BV
3	3.25		4080	0.004	VV
4	3.52		1398	0.001	VB
5	5.08		2370	0.002	BB
6	5.72		182	0.000	BB
7	6.04		335	0.000	BB
8	6.38		610	0.000	BB
9	7.25	BENZENE	47354	0.047	BV
10	7.52		30138	0.030	VB
11	8.25		1234	0.001	BB
12	13.87	ETHBEN	40199	0.040	BB
TOTAL			159898	0.159	

Non-targeted compounds Hall detector

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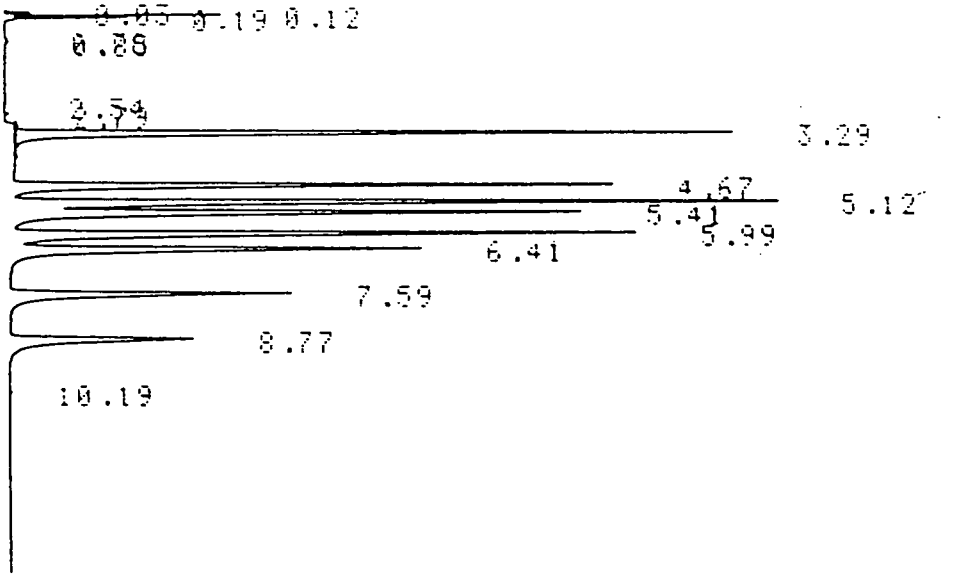

D-2000 07/29/87 08:24

METHOD: HALL TAG: 39 CH: 2

FILE: 1 CALC-METHOD: EXT-STD TABLE: 2 CONC: AREA

NO.	RT	NAME	AREA	CONC	BC
2	0.12		38839	0.038	BV
3	0.19		29790	0.029	VB
8	3.29	MECL2	489426	0.489	BB
9	4.67	11DCA	390539	0.390	BV
10	5.12	CHCL3	537809	0.537	VV
11	5.41	12DCA	474154	0.474	VV
12	5.99	111TCA	474926	0.474	VV
13	6.41	BRCL2M	348516	0.348	VB
14	7.59	CLBR2M	231536	0.231	BB
15	8.77	BROMO	177886	0.177	BB
TOTAL			3193351	3.193	

APPENDIX G

VOC - WARZYN STANDARD OPERATING PROCEDURE

WARZYN

PURGEABLE HALOCARBONS AND AROMATICS

Scope and Application: This method covers the determination of the following 35 organic compounds in water and soils:

Compounds:

Acetone*	Dibromochloromethane	Ethyl benzene
Benzene	1,2-Dichlorobenzene	Methylene chloride
Bromodichloromethane	1,3-Dichlorobenzene	1,1,2,2-Tetrachloroethane
Bromoform	1,4-Dichlorobenzene	Tetrachloroethene
Bromomethane	Dichlorodifluoromethane	Tetrahydrofuran (THF)*
Carbon tetrachloride	1,1-Dichloroethane	Toluene
Chlorobenzene	1,2-Dichloroethane	1,1,1-Trichloroethane
Chloroethane	1,1-Dichloroethene	1,1,2-Trichloroethane
2-Chloroethylvinyl ether	1,2-Dichloroethene	Trichloroethene
Chloroform	1,2-Dichloropropane	Trichlorofluoromethane*
Chloromethane	cis-1,3-Dichloropropene	Vinyl chloride
	trans-1,3-Dichloropropene	Xylenes

* Analyzed upon request.

The following sets of compounds are known not to be resolved on the 1% SP 1000 column and will be reported as the sum of the unresolved set.

- Vinyl chloride and Dichlorodifluoromethane
- cis-1,3-Dichloropropene, 1,1,2-Trichloroethane and Chlorodibromomethane
- Tetrachloroethene and 1,1,2,2-Tetrachloroethane

Note: Second column confirmation is performed upon client request.

Method: Purge and Trap - Gas Chromatography.

Reference: "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", July, 1982, Mthods 601, Purgeable Halocarbons, and 602, Purgeable Aromatics

Detection Limits: Waters 1.0 - 20 ug/L (See Table 4)
Low Level Soils - 10-200 ug/kg
Medium/High Level Soils - 50-20000 ug/kg

Quality Control:

1. Each analytical run should begin with a standard curve consisting of a 50, 25, 10 and 1 ug/L standard and a blank. Every sixth analysis thereafter and the last sample analyzed should also be one of the above standards. Ongoing calibration standards should be within 15% of the original standards or a new standard curve must be prepared and samples since the last check standard, reanalyzed.

2. Surrogate compounds 2-bromo-1-chloropropane for the halocarbons α,α,α-trifluorotoluene for the aromatics are added at 25 ug/L to each sample to monitor the performance of the method. Surrogate recoveries are calculated on an external standard basis. If a surrogate recovery falls outside of the acceptable control limits (see Table 5), the sample must be reanalyzed. If the surrogate recovery still falls outside of the control limits, the sample should be diluted until an acceptable surrogate recovery is obtained.

3. A minimum of 10% duplicate and 10% spiked samples should be analyzed. If less than 10 samples are analyzed, a duplicate and a spiked sample should still be analyzed. Duplicates should be within 15% and results are averaged. Recoveries for spiked samples should fall within the limits shown in Tables 3 and 6. For samples processed as a set where spiked sample recoveries fall outside of the established limits, data for the affected parameters should be asterisked and reported as suspect.

4. Table 4 presents verification of method detection limits calculated from seven control spikes at 5 times the detection limits.

5. New stock standards should be prepared monthly. New secondary standards should be prepared weekly. New working standards should be prepared daily.

Optimum Range: Up to 1,000 times the detection limit.

Sample Collection, Preservation and Handling:

Samples are to be collected in duplicate in 40 mL glass vials with open screw-caps and teflon faced silicone septa. They should be collected so that no headspace remains in the bottle. Samples should be preserved by adding one crystal of sodium thiosulfate, except for those which are known to contain non-chlorinated compounds which are preserved with the addition of three drops of 1:1 HCl. Samples should be refrigerated at 4°C and analyzed within 14 days of collection.

Reagents and Apparatus:

1. Open screw cap 40 mL vial (Pierce #13075 or equivalent). Detergent wash, deionized water rinse and dry at 105°C before use.

2. Septum - Teflon-faced silicone (Pierce # 12722 or equivalent). Detergent wash, deionized water rinse and dry at 105°C before use.

3. Purge and trap device - Tekmar LSC-2 equipped with ALS multiple sampler option.

4. Purge tubes - 5 mL and 25 mL fritted

5. Gas chromatograph - Varian 3400 equipped with a PID and Hall detectors in series.

6. Column 1 - 8 ft. x 1/8 inch stainless steel, packed with 1% SP-1000 on Carbopack B (60/80 mesh).

7. Column 2 - 8 ft. x 1/8 inch stainless steel packed with n-octane on Porasil - C (100/120 mesh) for confirmation.

8. Glass Syringes:

   5 mL Leurlock tip
   1 mL Hamilton
   100 uL Hamilton
   10 uL Hamilton

9. Balance - ± 0.0001 g - (Cahn TA4200)

10. Balance - ± 0.01 g - (Sartorius, 1202 MP)

11. Reagent water - organic free water or cold tap water which has been shown to be organic-free at the method detection limits.

12. Methanol - B & J Brand

13. Stainless steel tubes - 1/16" x 3" and 1/16" x 6"

14. 3 - 1/16" connectors

15. 1/16" Teflon tubing - 2 pieces 2 - 3 ft.

16. Stirring hot plate

17. Ring stand with clamps

18. Stir bars - 3/8" - 1/2" teflon coated

19. Beakers - assorted

20. Volumetric flasks - assorted

21. Pipettes - assorted

Reagent Preparation:

1. Stock Standard Solution: The stock standard solution is prepared at 5000 mg/L from pure standard materials (exceptions-Acetone, THF, and 2-chloroethylvinyl ether are prepared at 100 g/L; 1,2-DCB, 1,3-DCB and 1,4, DCB are prepared at 25 g/L; Bromoform is prepared at 10 g/L). Correction for purities of less than 99% should be made. Gas standards are purchased as a certified solution.

   1.1 Add about 20 mL of methanol to a 25 mL volumetric flask. Allow the flask to stand unstoppered until the methanol on the neck of the flask has dried.

1.2 Tare the flask on the analytical balance.

1.3 Using a 100 uL syringe add the reference material to the flask. Make sure the drops fall directly into the methanol without contacting the neck of the flask.

1.4 Determine the amount of reference material added and tare the flask. Rinse the syringe with methanol and add the next standard.

1.5 After all the reference materials are added, fill to volume with methanol and invert to mix.

2. The stock gas standard (200 ug/mL of chloromethane, bromomethane, vinyl chloride and chloroethane prepared in methanol) is purchased as a certified solution in sealed ampules.

3. Working Standard Solutions:

3.1 Prepare a secondary standard mix at 500 ug/mL by diluting 1 mL of the stock standard to 10 mL with methanol.

3.2 Prepare working standards at 1.0, 10.0, 25.0, and 50.0 ug/mL from the secondary standard.

3.3. Prepare working gas standards at 1.0 to 25 ug/mL from the purchased solution.

4. Surrogate Standard Solution:

4.1 2-Bromo-1-chloropropane and α,α,α-trifluorotoluene are used as the surrogate standards.

4.2 The stock surrogate standard is prepared the same as steps 1.1-1.5 above.

4.3 Dilute to obtain a final surrogate standard concentration of 25.0 ug/mL.

Procedures:

Waters:

1. Adjust the purge flow rate on the LSC-2 Automatic Liquid Sampler (ALS) to 40 mL/min. (Attach a bubble meter to a sample line on the ALS autosampler and adjust the purge pressure and purge flow controllers as needed).

2. Set the LSC-2 for the following cycle:

Purge - 8 minutes
Desorb - 4 minutes at 180°C
Bake - 8 minutes at 180°C
Transfer lines - 100°C

3. Remove the plunger from a 5 mL syringe. While holding a finger over the tip, rinse and fill the syringe with reagent water. Replace the barrel and adjust to 5.0 mL mark making sure that all air bubbles are removed. Spike through the valve bore with 5 uL of the standard mixture.

4. Attach syringe to the syringe valve on the purging device. Open the valve and inject the sample into the purging chamber. Close the syringe valve on the purging device.

5. Continue filling the remaining purge vessels of the ALS auto sampler using the same technique as above. Make sure to add 5 uL of the surrogate to samples and blanks.

6. Index the ALS to the sample number before the first sample, then advance the switch to auto to begin purging.

7. When analysis is completed remove the purge tube from the ALS empty the contents, flush with DI water and rinse with reagent water.

Low Soils:

1. The LSC-2 and ALS purge and trap parameters are the same as for the water samples.

2. Remove the purging vessel from the ALS. Attach teflon tubing to both the purge and sample lines. Connect the 6" stainless steel probe to the tubing from the purge line with a 1/16" union. Connect the 3" stainless steel probe to the tubing from the sample line with a 1/16" union.

3. Set up a water bath by placing a 150 or 250 mL beaker half full of water on a stirring hot plate. Heat the bath to 60°C. A ring stand with clamps will hold the modified purge vessel (40 mL VOC vial) in the water bath.

4. For standards (100, 50, 25, 10 and 0 ppb) add 5 mL of reagent water to a 40 mL VOC vial containing a 3/8" - 1/2" teflon coated stir bar. Spike with 5 uL of the standard mixture. Cap and insert the 6" purge probe through the septum to the bottom of the vial. Insert the 3" sample probe through the septum about 1 inch. Place the vial in the water bath and adjust the height so that the stir bar stirs smoothly. Start the purge cycle by advancing to start on the LSC-2.

5. When purging is completed remove the probes and place them in an empty 40 mL VOC vial until the next analysis. (Back pressure causes the sample to flow back into the purge line.)

6. For samples, weigh out 5.0 g into a 40 mL VOC vial containing a stir bar. Add 5.0 mL of reagent water and 5 uL of the surrogate mix. Cap and place probes in the vial and continue in the same manner as in step #4 above.

7. When purging is completed, remove the probes and clean by rinsing with deionized water and wiping with a Kimwipe. Place in an empty VOC vial.

8. See Table 3 for Q.C. limits for spike recoveries.

Medium/High Soils:

This VOC screening procedure is used for moderate to highly contaminated soil samples. These samples may contain high concentrations of volatiles and odor can be detected upon opening the sampling containers. Any soil samples that contain large amounts of gasoline or fuel oil, volatile interferences, or samples where screening procedures (i.e. HNu, OVA, etc.) indicate high contamination levels, should be analyzed by this procedure. This procedure can also be used for screening of samples where lower detection limits are not critical (see Table 6 for Detection Limits and Q.C. Limits for spike recoveries).

Procedure:

1. Weigh out 5.0 g of sample in a 20x150mm screw cap centrifuge tube.

2. Add 5.0 mL of methanol to the sample.

3. Spike with the appropriate level of surrogate or spike mix.

4. Cap with a teflon lined screw cap and mix the sample gently for 1 minute.

5. Place the tubes in a centrifuge for 2 min at 1200 rpm's.

6. Remove the tubes from the centrifuge and transfer a portion of the methanol layer into a small vial with a disposable pipet.

7. Inject 100 uL or less (depending on the concentration of the interferences) of the extract into 5 mL of tap water and load into a purging vessel on the LSC-2 purge and trap.

8. Proceed with analysis, (loading, timing, etc) as stated for water samples.*

* Note: A procedural blank should also be run with each set of samples. Prepare by spiking 100 uL of the methanol used for the extraction into 5 mL of tap water and running as above. Subtract any contaminant volatiles found in the methanol from the final concentrations of the samples.

Notes:

1. Purge tubes should be acid cleaned periodically when lime or other deposits become evident.

2. Stir bars should be cleaned with hot water and soap, rinsed with DI water then rinsed with methanol.

3. All soapy or foamy samples should be diluted to prevent contamination of the sample lines and the purge and trap.

4. An 8 ft x 1/8 in n-octane on Porasil C (100/120) mesh column is used for confirmation, if necessary.

Gas Chromatographic Conditions:

Column:

8 ft. x 1/8 inch stainless steel, packed with 1% SP-1000 on Carbopack B (60/80 mesh).

Carrier Gas:

Helium - Ultra High Purity Grade (Linde)
35 mL/min

Detectors (in series):

1. Photo Ionization 10.2eV

   Sensitivity - Range 11 X Attenuation 8
   Temperature - 240°C

2. Hall 700A/ECD

   Mode - Halogen
   Reactor temperature - 1000°C
   Solvent Flow - 0.8mL/min Methanol
   Hydrogen Flow - 60 mL/min

Injector:

Temperature - 200°C

Oven:

Initial - 45°C/4 minutes
Rate - 8°C/minute
Final - 220°C/20 minutes

Relative retention times are found on Tables 1 and 2.

Second Column Confirmation:

Second column confirmation is performed upon client request when routine first run samples have detected amounts of coeluting parameters. An eight foot by 1/8" n-octane on Porasil C (100/120 mesh) column is used for confirmation. When confirmation is performed, helium carrier gas is 40 mL/min flow rate, column temperature is held at 50°C for three minutes then programed at 6°C per minute to 170°C and held for four minutes.

Calculations:

1. Determine that the surrogate standards are within the quality control limits; if not, reanalyze the sample.

2. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.

3. Prepare external standard calibration curves of ug vs. response for each parameter using at least three data points and linear regression analysis.

4. Calculate the concentration found in the samples from the calibration curves using the following equations:

   Waters: $ug/L = \frac{A}{Vs} \times 1000$

   where: A = Amount of parameter found in analysis (in ug)
   Vs = Volume of sample (in mL)

   Note: Vs should be actual volume of sample taken before any dilutions.

   Soils: $ug/kg = \frac{A}{Ws} \times 1000$

   where: A = Amount of parameter found in the analysis (in ug)
   Ws = Weight of sample (in g)

5. If the calculated result does not fall within the limits of external standard curve, the sample must be diluted and reanalyzed.

TABLE 1

PHOTO IONIZATION DETECTOR

Parameter	1% SP-1000 on Carbopack B (60/80 mesh) Retention Time (Minutes)
Acetone	8.30
1,1 Dichloroethene	10.5
THF	11.9
1,2-Dichloroethene	12.6
trans,1,3-Dichloropropene	17.6
Trichloroethene	18.2
Benzene	18.7
cis-1,3-Dichloropropene	18.9
2-Chloroethylvinyl ether	19.9
Tetrachloroethene	23.7
Toluene	24.9
α,α,α-Trifluorotoluene	25.6
Chlorobenzene	26.1
Ethylbenzene	28.1
M-Xylene	32.6
O&P-Xylene	33.6
1,3-Dichlorobenzene	37.9
1,2-Dichlorobenzene	39.0
1,4-Dichlorobenzene	39.7

TABLE 2

HALL DETECTOR

Parameter	1% SP-1000 on Carbopack B (60/80 mesh) Retention Time (Minutes)
Chloromethane	1.82
Bromomethane	3.02
Vinyl Chloride	3.88
Chloroethane	5.13
Methylene Chloride	7.63
1,1-Dichloroethene	10.5
1,1-Dichloroethane	11.9
1,2-Dichloroethene	12.6
Chloroform	13.2
1,2-Dichloroethane	15.2
1,1,1-Trichloroethane	15.6
Carbon Tetrachloride	15.6
Bromodichloromethane	16.1
1,2-Dichloropropane	17.4
trans-1,3-Dichloropropene	17.7
Trichloroethene	18.2
Dibromochloromethane	19.0
1,1,2-Trichloroethane	19.0
cis-1,3-Dichloropropene	19.0
2-Chloroethyvinyl ether	19.9
Bromoform	21.6
1,1,2,2-Tetrachloroethane	23.7
Tetrachloroethene	23.7
α,α,α-Trifluorotoluene	25.6
Chlorobenzene	26.1
1,3-Dichlorobenzene	37.9
1,2-Dichlorobenzene	39.0
1,4-Dichlorobenzene	39.7

TABLE 3

SPIKES OF FIELD WATER SAMPLES

	Number of Analyses	Average % Recovery	% Relative Standard Deviation	3 Standard Deviations Acceptable Recoveries(1)
Acetone	* (8)	*(94.4)	*(16.3)	
Benzene	25	90.6	6.05	72.4-109
Bromodichloromethane	25	91.2	4.38	78.1-104
Bromoform	25	72.9	9.50	44.4-101
Bromomethane	25	95.9	11.8	60.5-131
Carbon tetrachloride	25	89.8	8.29	64.9-115
Chlorobenzene	25	87.7	7.87	64.1-111
Chloroethane	21	96.2	6.82	75.7-117
2-Chloroethylvinyl ether	20	104	16.2	55.4-153
Chloroform	25	95.6	3.04	86.5-105
Chloromethane	20	100.4	6.99	79.4-121
Dibromochloromethane	15	80.7	6.10	62.4-99.0
1,2-Dichlorobenzene	25	76.6	7.74	53.4-99.8
1,3-Dichlorobenzene	25	81.0	9.17	53.5-108
1,4-Dichlorobenzene	25	80.8	8.03	56.7-105
Dichlorodifluoromethane	*	*	*	
1,1-Dichloroethane	25	98.7	4.38	85.6-112
1,2-Dichloroethane	25	85.3	5.50	68.8-102
1,1-Dichloroethene	25	94.6	5.91	76.9-112
1,2-Dichloroethene	23	93.9	5.46	77.5-110
1,2-Dichloropropane	25	93.7	4.66	79.7-108
cis-1,3-Dichloropropene	*	*	*	
trans-1,3-Dichloropropene	*	*	*	
Ethyl benzene	25	86.2	9.36	58.1-114
Methylene chloride	25	94.1	10.3	63.2-125
1,1,2,2-Tetrachloroethane	*	*	*	
Tetrachloroethene	25	85.7	9.42	57.4-114
Tetrahydrofuran (THF)	*	*	*	
Toluene	25	88.0	6.66	68.0-108
1,1,1-Trichloroethane	25	91.8	7.66	68.8-115
1,1,2-Trichloroethane	*	*	*	
Trichloroethene	25	85.3	7.45	63.0-108
Trichlorofluoromethane	*	*	*	
Vinyl chloride	20	95.3	7.17	73.8-117
Xylenes	23	88.4	9.37	60.3-116

* Insufficient data for statistics.

(1) These Q.C. limits are also used for low soils analysis.

TABLE 4

Analyses of Seven Control Waters Spiked at 5x LOD

	Reportable Detection Limit (ug/L)	Average Recovery (%)	Standard Deviation	Calculated Method Detection Limit (ug/L)
Acetone	20.0	77.0	10.7	33.6
Benzene	1.0	95.6	5.04	0.79
Bromodichloromethane	1.0	99.7	5.72	0.90
Bromoform	2.0	93.6	7.48	2.35
Bromomethane	2.0	95.1	4.39	1.38
Carbon tetrachloride	1.0	98.1	2.95	0.46
Chlorobenzene	1.0	87.9	6.45	1.01
Chloroethane	1.0	92.9	5.33	0.84
2-Chloroethylvinyl ether	20.0	105	7.81	24.5
Chloroform	1.0	110	5.60	0.88
Chloromethane	1.0	90.0	6.56	1.03
Dibromochloromethane	1.0	99.7	8.31	1.30
1,2-Dichlorobenzene	5.0	85.1	5.54	4.35
1,3-Dichlorobenzene	5.0	82.9	8.95	7.03
1,4-Dichlorobenzene	5.0	55.0	6.84	5.37
1,1-Dichloroethane	1.0	106	7.20	1.13
1,2-Dichloroethane	1.0	114	19.5	3.06
1,1-Dichloroethene	1.0	99.6	6.02	0.94
1,2-Dichloroethene	1.0	104	8.14	1.28
1,2-Dichloropropane	1.0	103	7.00	1.10
Ethyl benzene	1.0	85.0	10.1	1.59
Methylene chloride	1.0	112	12.8	2.00
Tetrachloroethene	1.0	89.1	8.23	1.29
Tetrahydrofuran (THF)*	20.0	79.0	16.1	25.3
Toluene	1.0	92.4	7.01	1.01
1,1,1-Trichloroethane	1.0	102	3.57	0.56
Trichloroethene	1.0	96.0	4.41	0.69
Vinyl chloride	1.0	87.1	7.26	1.14
Xylenes	1.0	83.6	8.86	1.39

* THF spiked at 2.5 times the LOD.

TABLE 5

SURROGATE RECOVERY CONTROL LIMITS

Matrix Type	α,α,α Trifluorotoluene Number of Analyses	Average % Recovery	Relative Standard Deviation	Acceptable Recoveries
Water	34	87.9%	13.7%	50.0-120%
Low Soils	19	80.2%	11.0%	47.2-113%
Medium/High Soils	33	91.4%	16.1%	43.1-140%

Matrix Type	2-Bromo-1-chloropropane Number of Analyses	Average % Recovery	Relative Standard Deviation	Acceptable Recoveries
Water	32	80.0%	6.91%	60.0-120%
Low Soils	19	82.8%	12.6%	45.0-121%
Medium/High Soils	32	74.0%	11.1%	40.7-107%

TABLE 6

DETECTION LIMITS AND RESULTS FOR SPIKES
OF FIELD SOIL SAMPLES (Medium/High)

Compound	Number of Analysis	Detection Limit ug/kg	Average % Recovery	% Relative Standard Deviation	2 Standard Deviations Acceptable Recoveries
Benzene	7	50	87.6	11.8	64.0-111
Bromodichloromethane	8	50	89.1	19.8	49.5-129
Bromoform	7	100	79.6	14.7	50.2-109
Carbontetrachloride	7	50	96.9	12.7	71.5-122
Chlorobenzene	8	50	83.2	13.9	55.4-111
Chloroform	8	50	91.2	18.6	54.0-128
Dibromochloromethane	8	50	87.8	12.5	62.8-113
1,2-Dichlorobenzene	8	250	81.7	13.0	55.7-108
1,3-Dichlorobenzene	8	250	83.4	16.3	50.8-116
1,4-Dichlorobenzene	7	250	90.9	21.4	48.1-134
1,1-Dichloromethane	8	50	88.8	13.2	62.4-115
1,2-Dichloroethane	8	50	86.8	15.1	56.6-117
1,1-Dichloroethene	7	50	86.2	15.2	55.8-117
1,2-Dichloroethene	8	50	92.6	17.7	57.2-128
1,2-Dichloropropane	8	50	91.2	16.1	59.0-123
Ethyl benzene	8	50	89.7	11.7	66.3-113
Methylene Chloride	6	50	75.4	14.0	47.4-103
Tetrachloroethene	8	50	91.5	13.4	64.7-118
Toluene	6	50	83.8	12.8	58.2-109
1,1,1-Trichloroethane	7	50	94.7	12.6	69.5-120
Trichloroethene	8	50	91.8	13.0	65.8-118
Xylenes	7	50	87.7	14.4	48.9-116

Revision Date

6-17-87

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[ALM-13-2]

APPENDIX H

FIELD MEASUREMENT OF HYDRAULIC CONDUCTIVITY

WARZYN

FIELD MEASUREMENT OF PERMEABILITY
BAIL-DOWN TEST

INTRODUCTION

The objective is to determine hydraulic parameters (transmissivity, storativity, hydraulic conductivity) of the water-bearing strata. Single well aquifer tests are used because they may be conducted using a minimum of equipment, personnel, and time. They may also be done at many points within an aquifer and may be used to better plan a full-scale pumping test.

PROCEDURES

Each bail-down test is conducted by measuring the static water level with an electric water level indicator or cloth tape with attached sounding device, placing a pressure transducer (connected to a Hermit data logger[1]), below the water level, and removing one bailer volume from the well. The recovery of the water level back to the static level is recorded over the necessary period of time, using a logarithmic sampling mode on the data logger.

The electric water level tape and transducer are wiped, first with methanol and then with deionized water as they are placed in the wells, to prevent cross-contamination between wells. The bailer is lowered with an attached 1/8-inch stainless steel cable. The bailer and cable are decontaminated between uses by washing and rinsing with Liquinox soap and water, rinsing three times with methanol, and rinsing three times with deionized water. The bailer and cable are then allowed to air-dry on steel supports and are covered with new sheet plastic.

1 Hermit Environmental Data Logger, Model SE1000B, In-Situ, Inc., Laramie, Wyoming 82070.

DATA REDUCTION

The data accumulated during the bail-down tests are used to calculate hydraulic parameters using several published methods. Hydraulic conductivity is calculated for shallow, unconfined wells using the Bouwer and Rice method (1976). The NAVFAC method (1971) is used for comparison. Transmissivities and storativities for the deeper, confined wells are determined using the curve matching method described by Cooper, et al. (1967). For comparison, hydraulic conductivities are also calculated using the Hvorsley method (1951). All methods assume an infinite, homogenous, isotropic aquifer and an instantaneous change in head in the well.

The method described by Bouwer and Rice (1976) is based upon modifications to the Thiem equation, with the use of an analog model. A straight line is drawn through a semi-log plot of relative head versus time, and the hydraulic conductivity is calculated using the slope of that line and the geometry of the well and aquifer. The formulation assumes that draw-down of the water table around the well is negligible, that flow in the capillary fringe may be ignored, and that well losses are negligible. It is applicable to completely or partially penetrating wells in unconfined aquifers, but may be used for confined aquifers that receive water from the upper confining layer.

In the NAVFAC method (1971), a straight line is also drawn through a semi-log plot of recovery data for unconfined aquifers. The method is based on the Hvorsley method. It assumes that the well is cased below the water table, and the ratio of the screen length to the well radius (L/R) is greater than eight.

The Cooper, et al. (1967) formulation calculates the transmissivity of an aquifer by matching a plot of relative head (linear scale) versus time (logarithmic scale) to one of a set of type curves. The method assumes that the change in head after a known volume of water is injected or removed is instantaneous and that the (non-flowing) well is screened over the entire thickness of an artesian aquifer. It is directly applicable to fully penetrating screened wells in confined aquifers, but may be used to determine the transmissivity of the portion of an aquifer over which a partially penetrating well is screened, assuming no vertical flow occurs.

[1] Hermit Environmental Data Logger, Model SE1000B, In-Situ, Inc., Laramie, Wyoming 82070.

The Hvorsley method (1951) is based on a solution of the LaPlace equation and does not account for aquifer storage. A straight line is drawn through a semi-log plot of relative head versus time. The time that would be required for complete equalization of head difference if the original rate of inflow were maintained (defined as the basic time lag, T_o, and equal to the time when H-h/H-Ho = 0.37) is used to calculate the hydraulic conductivity. The value of T_o is measured graphically, and the ratio of the piezometer length to radius is assumed to be greater than eight (L/R>8).

[jap-600-71E]

1 Hermit Environmental Data Logger, Model SE1000B, In-Situ, Inc., Laramie, Wyoming 82070.