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WARZYN

PROJECT
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PLANNING DOCUMENTS
VOLUME III OF III
QUALITY ASSURANCE PROJECT PLAN
FINAL

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

BELOIT CORPORATION
ROCKTON FACILITY
ROCKTON, ILLINOIS

JUNE 1992

COPY

PREPARED FOR:
BELOIT CORPORATION
ROCKTON, ILLINOIS

...

PREPARED BY:
WARZYN INC.
MADISON, WISCONSIN

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MADISON, WISCONSIN

Kevin J. Domack
Project Manager

James E. Moser
RI Task Manager

LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Administrative Order on Consent
ARAR	Applicable or Relevant and Appropriate Requirement
ASTM	American Standards for Testing Materials
CDO	Central District Office
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quantitation Limits
CRL	Central Regional Laboratory
DL	Detection Limit
DQO	Data Quality Objective
ESI	Expanded Site Inspection
FIT	Field Investigation Team
FSP	Field Sampling Plan
GC	Gas Chromatograph
HRS	Hazard Ranking System
HSP	Site-Specific Health and Safety Plan
ICP	Inductively Coupled Plasma Spectrometer
IDL	Instrument Detection Limit
IEPA	Illinois Environmental Protection Agency
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NPL	National Priorities List
PA	Preliminary Assessment
PRP	Potentially Responsible Party
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	Quality Control
R&D Center	Research & Development Center
%R	Percent Recovery
RI/FS	Remedial Investigation/Feasibility Study
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SER	Site Evaluation Report
Site	Beloit Corporation, Rockton Facility
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TDS	Total Dissolved Solids
TCL	Target Compound List

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TIC	Tentatively Identified Compound
TOC	Total Organic Carbon
U.S. EPA	United States Environmental Protection Agency, Region V
VOA	Volatile Organic Analysis
Warzyn	Warzyn Inc.

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INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by U.S. EPA participate in a centrally managed quality assurance (QA) program.

Any party generating data under this program has the responsibility to implement minimum procedures to assure that precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written QA Project Plan (QAPP) covering each project it is to perform.

This 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) for the Beloit Corporation Rockton Facility Site. This QAPP also describes the specific protocols which will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory and field analysis.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPP is prepared by Warzyn Inc. (Warzyn) in accordance with the U.S. EPA QAPP guidance documents:

- U.S. EPA, December 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.
- U.S. EPA, May 1991, Region V Model Superfund Quality Assurance Project Plan.
- U.S. EPA, January 1989, Region V Content Requirements for Quality Assurance Project Plan.

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PROJECT DESCRIPTION

1.1 SITE HISTORY AND BACKGROUND INFORMATION

The Beloit Corporation Rockton Facility (site) is located in Rockton Township, in north-central Illinois. The site lies in a mixed industrial and residential area approximately 3/4 mile north of the City of Rockton. The site occupies part of the northern half of Section 13 and the southeast quadrant of Section 12, T46N, R1E, Winnebago County, Illinois.

The RI/FS boundary, as identified by the Illinois Environmental Protection Agency (IEPA), includes Beloit Corporation property, the neighboring Blackhawk Acres Subdivision and United Recovery Property. The site is bounded on the north by Prairie Hill Road, on the west by the Rock River, on the south by a line projected along a Beloit Corporation access road from Blackhawk Boulevard to the Rock River, and on the east by Blackhawk Boulevard.

The Beloit Corporation, a subsidiary of Harnischfeger Industries, is a manufacturer of machines that produce layered paper products from paper pulp. Site features located on the Beloit Corporation property include the plant, a research and development center (R&D Center), wastewater treatment facility, a foundry sand disposal area, fibrous sludge spreading area, gravel pit and storage areas. Additional buildings located on the site include United Recovery, Safe-T-Way, and homes in the Blackhawk Acres Subdivision.

1.2 PAST DATA COLLECTION ACTIVITY/CURRENT STATUS

The site was officially entered on the National Priorities List (NPL) on August 30, 1990, with a hazard Ranking Score of 52.08.

The site has been subject of a number of investigations since 1980. A discussion of the investigations is provided in the Site Evaluation Report (SER) (Warzyn, 1991).

1.3 PROJECT OBJECTIVES AND SCOPE

The purpose of the RI is to gather sufficient information to quantify risk to public health and environment (Baseline Risk Assessment) and to develop and evaluate viable remedial alternatives (Feasibility Study) at the site. The objectives of the RI are to determine the nature and extent of contamination at the site in order to

support the activities of the FS. The objective of the RI/FS is to develop and evaluate appropriate remedial action alternatives based on the RI/FS data.

The primary remedial objectives of the data collection are as follows:

- Collect sufficient data on all contaminated media to support the baseline risk assessment and feasibility study,
- Further define the nature and extent of groundwater contamination, as well as identify potential source(s) of the contamination,
- Acquire data necessary to determine potential contaminant migration pathways and transport rates,
- Further define the nature and extent of on site soil contamination, and
- Determine background groundwater and soil quality.

All tasks, subtasks and activities are directed toward the accomplishment of these primary objectives. Refer to the Work Plan for a detailed description of the RI tasks, subtasks and activities.

1.4 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale for sample locations is described in detail in Section 3 of the FSP (refer to Appendix A).

1.5 PARAMETERS TO BE TESTED AND FREQUENCY

Sample matrices, analytical parameters, and frequencies of sample collection can be found in Table 1-1. A summary of sample volume, bottle, preservative, and packaging requirements is provided in Table 1-2.

1.6 INTENDED DATA USAGE AND DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during the RI/FS activities, and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). For the site, data generated under DQO Level 1 are for health and safety monitoring. Health and Safety Monitoring activities are described in the Site-Specific Health and Safety Plan (HSP) and are not covered in this QAPP.

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile laboratory generated data depending on the level of quality control exercised. For the site, data generated under DQO Level 2 are: the determination of pH, conductivity, temperature, and water level measurements in groundwater. In addition, soil gas and groundwater VOC data generated by field GC are considered DQO Level 2.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround for screening, but without full quality control documentation, or confirmed identification and quantification data, with built-in QA/QC, including calibration runs, surrogate standards, etc., and external QA/QC such as duplicate samples, field blanks, etc.). For the site, data generated under DQO Level 3 are the physical characterization of soils for grain size distribution, and the indicator parameters (alkalinity, chloride, sulfate, total dissolved solids (TDS), nitrate and nitrite nitrogen, ammonia nitrogen, and total phenolics) for groundwater samples.

- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and Potentially Responsible Party (PRP) determination. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with U.S. EPA recognized protocol. For the site, data generated under DQO Level 4 are the Target Compound List (TCL) organic and Target Analyte List (TAL) inorganic parameters for groundwater and soil matrices.

- Non-Standard (DQO Level 5): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require

method development or adaptation. The level of quality control is usually similar to DQO Level 4 data. These analyses require full CLP analytical and data validation procedures in accordance with U.S. EPA recognized protocol. DQO Level 5 data is not anticipated for the site.

A summary of data generating activities, intended data uses and associated DQOs for the site are presented in Table 1-3.

1.7 PROJECT SCHEDULE

A schedule of RI/FS activities for the site is presented in the Work Plan.

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PROJECT ORGANIZATION AND RESPONSIBILITY

At the direction of the IEPA and the Beloit Corporation, Warzyn will be conducting all phases of the RI/FS. Warzyn will perform the field investigation, prepare the RI report, and perform the subsequent FS. Project management will also be provided by Warzyn, under the direction of the Beloit Corporation. The various quality assurance and management responsibilities of key project personnel are defined below. Refer to Figure 2-1 for the project organizational chart.

2.1 OVERALL RESPONSIBILITY

Illinois EPA Remedial Project Manager
The IEPA Remedial Project Manager (RPM) is Mr. Paul Takacs. The RPM has the overall responsibility for all phases of the RI/FS.

Beloit Corporation Site Project Manager
The Beloit Corporation Site Project Manager is Mr. Mike Radcliffe. The Site Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Site Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. He will report directly to the IEPA RPM and will provide the major point of contact and control for matters concerning the project.

Warzyn Project Manager
The Warzyn Project Manager is Mr. Kevin Domack. The Warzyn Project Manager has the overall responsibility for ensuring that the project meets IEPA objectives and Warzyn's quality standards. In addition, he is responsible for technical quality control and project oversight, and will provide the Site Project Manager with access to corporate management.

The Beloit Corporation Site Project Manager and Warzyn Project Manager will:

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- Define project objectives and develop a detailed work plan schedule;

- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient field leaders and support staff concerning the project's special considerations;
- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to assess its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all external reports (deliverables) before their submission to the IEPA;
- Ultimately be responsible for the preparation and quality of interim and final reports; and
- Represent the project team at meetings and public hearings.

Warzyn Remedial Investigation Leader

The Warzyn RI Leader is Mr. Thomas Karwoski. The RI Leader is a support to the Warzyn Project Manager. He is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The RI Leader is a highly experienced environmental professional and will report directly to the Warzyn Project Manager. Specific responsibilities include:

- Provision of day-to-day coordination with the Project Managers on technical issues in specific areas of expertise;
- Development and implementation of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;

- Coordination and management of field activities including sampling, drilling, and field staff;
- Implementation of QC for technical data provided by the field staff including field measurement data;
- Adherence to work schedules provided by the Project Managers;
- Review and approval of text and graphics required for field team efforts;
- Coordination and oversight of technical efforts of subcontractors assisting the field team;
- Identification of problems at the field team level, discussion of resolutions with the Project Managers, and provision of communication between team and upper management; and
- Participation in the preparation of the final RI report.

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

Warzyn Quality Assurance Officer
The Warzyn Quality Assurance Officer (QAO) is Mr. John Dadisman. The QAO will remain independent of direct job involvement and day-to-day operations, and has direct access to corporate executive staff as necessary to resolve any QA dispute. He is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, Warzyn's policies, and U.S. EPA requirements. Specific functions and duties include:

- Provide QA audit on various phases of the field operations;
- Review and approval of QA plans and procedures; and
- Provide QA technical assistance to project staff;

IEPA Quality Assurance Officer
The IEPA QAO has the responsibility to review and approve the QAPP. The IEPA QAO is Mr. Jim Shaw.

2.2 SPECIALIZED RESPONSIBILITIES

2.2.1 Monitoring and Sampling Operations and QC

- Principal Engineering Firm - Warzyn, Madison, Wisconsin
- Drilling - To be determined through bidding process.
- Sampling, Monitoring and Survey - Warzyn, Madison, Wisconsin
- On Site Day-to-day Field Activities - RI Leader, Warzyn, Madison, Wisconsin
- Quality Control - QAO, Warzyn, Madison, Wisconsin

2.2.2 Laboratory Key Personnel

Laboratory Project Manager - The Warzyn Laboratory Project Manager is Gordon Gerry. The CompuChem Laboratory Project Manager is Angela Haas. The Laboratory Project Manager is responsible for ensuring all resources of the laboratory are available on an as-required basis and providing an overview of the final analytical reports.

Laboratory Operations Manager - The Warzyn Laboratory Operations Manager is Gordon Gerry. The CompuChem Laboratory Operations Manager is Richard Bloom. The Laboratory Operations Manager coordinate laboratory analyses, and oversee sample analysis, data review, and report preparation. The Laboratory Operations Manager approves the final report prior to submission.

Laboratory Quality Assurance Officer - The Warzyn Laboratory Quality Assurance Officer (QAO) is Rick Mealy. The CompuChem Laboratory QAO is Robert Whitehead. The laboratory QAO is responsible for overview of all QA/QC, and as such conducts detailed data reviews. In addition, all corrective actions are approved by the QAO.

Laboratory Sample Custodians - The Laboratory Sample Custodian will:

- receive and inspect the incoming sample containers
- record the condition of the incoming sample containers
- sign appropriate documents
- verify correct chain-of custody
- notify appropriate laboratory of sample receipt and inspection

- assign unique laboratory identification number to each sample and enter into the sample receiving log
- initiate transfer of the samples to the appropriate lab sections
- control and monitor access to sample and extract storage, and maintain in-house custody

2.2.3 Laboratory Analysis

- Analysis of groundwater and soil samples as for TCL organics specified in Table 1-1:

  CompuChem Laboratory
  3308 Chapelhill/Nelson Highway
  Research Triangle Park, North Carolina, 27709

- Analysis of groundwater and soil samples for TAL inorganics as specified in Table 1-1:

  Warzyn Inc.
  One Science Court
  University Research Park
  Madison, Wisconsin, 53711

- Analysis of samples for indicator parameters (alkalinity, chloride, sulfate, total dissolved solids (TDS), nitrate and nitrite, ammonia, total phenolics) as specified in Table 1-1:

  Warzyn Inc.
  One Science Court
  University Research Park
  Madison, Wisconsin, 53711

- Analysis of sediment/soil samples for physical characteristics (grain size analysis) as specified in Table 1-1:

  Warzyn Soils Laboratory.
  505 Science Drive
  Madison, WI 53711

2.2.4 Laboratory Data and QC

Laboratory Data

- Analytical protocol specified - Warzyn, Madison, Wisconsin
- Review of analytical protocol - CompuChem, Warzyn technical staff
- Review of analytical procedures - IEPA Quality Assurance Section (QAS)
- Internal QA/QC - Laboratory QAO, CompuChem, Warzyn
- Final data review and validation - Chemist, Warzyn, Madison, Wisconsin
- Review of tentatively identified compounds and assessment of need for confirmation - Chemist, Warzyn, Madison, Wisconsin

2.2.5 Performance and Systems Audits

Field Operations

- Internal Audits - QAO, Warzyn, Madison, Wisconsin
- External Audits - IEPA

Analytical Laboratories

- Internal Audits - Laboratory QAO, CompuChem, Warzyn
- External Audits - IEPA

Final Evidence File

- Final Evidence File Audits - QAO, Warzyn, Madison, Wisconsin

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QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 LEVEL OF QUALITY CONTROL EFFORT

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

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

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOAs or extractable organics. However, aqueous MS/MSD

samples must be collected at triple the volume for VOAs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil). The number of field duplicate and field blank samples to be collected for this site are listed in Table 1-1. Sampling procedures are specified in the FSP (refer to Appendix A).

Soil and groundwater samples will be sent to CompuChem and Warzyn for analysis. Parameter lists and required quantitation levels are summarized in Tables 3-1, 3-2, and 3-3.

The level of laboratory QC effort for testing of TAL inorganics and TCL organics is specified in the current CLP Statements of Work (SOWs): ILM01.0 for inorganics, OLM02.0 for organics. The levels of laboratory QC effort for indicator parameters are specified in Table 3-6.

The level of field QC effort for pH, specific conductance and temperature is specified in the method SOPs, and summarized in Table 3-6. The level of QC effort for the field GC volatiles analysis is specified in the SOP presented in Appendix C3, and summarized in Table 3-6.

3.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSES

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

TCL Organics/TAL Inorganics
Laboratory analysis of TCL organics and TAL inorganics will follow the current CLP SOWs (OLM02.0 for organics, ILM01.0 for inorganics). The accuracy and precision, and sensitivity requirements are summarized within the CLP SOWs.

Indicators
SOPs for the indicator parameters (alkalinity, chloride, sulfate, TDS, nitrate and nitrite nitrogen, ammonia nitrogen and total phenolics) are provided in Appendix B. The accuracy and precision requirements of these analyses are summarized in Table 3-6. The sensitivity requirements of these analyses are summarized in Table 3-3.

Field Measurements
SOPs for the field equipment to measure pH, conductivity, and temperature are provided in Appendix C. The accuracy and precision requirements of these

analyses are summarized in Table 3-6. The sensitivity requirements of these analyses are summarized in Table 3-3.

The SOP for the Field GC VOC Screening procedure is provided in Appendix C. The accuracy and precision requirements are summarized in Table 3-6, and sensitivity requirements in Table 3-4.

Physical Characteristics
Determination of grain size distribution will follow the method referenced in Table 3-5. The SOP for the grain size distribution is provided in Appendix B. The accuracy and precision requirements and sensitivity requirements for grain size distribution analysis are summarized in Tables 3-6 and 3-3, respectively.

3.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the participating laboratories will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested using the SOWs and SOPs referenced in Section 3.2. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness (\%)} = A/B \times 100$$

where, A = number of valid data
B = number of samples collected for each parameter analyzed

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past site activities, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale of the sampling network is discussed in detail in the FSP. Representativeness will be satisfied by insuring that the FSP is followed, proper sampling technique are used, proper analytical procedure are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicate samples.

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

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SAMPLING PROCEDURES

Sampling procedures are described in the FSP which is contained in Appendix A of this document.

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SAMPLE CUSTODY AND DOCUMENTATION

It is U.S. EPA policy to follow the U.S. EPA sample custody, or chain-of-custody protocols described in "NEIC Policies and Procedures", EPA-330/9-78-DDI-R, Revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

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

- are in your possession;
- are in your view, after being in your possession;
- are in your possession and you place them in a secured location; or
- are in a designated secure area.

5.1 FIELD-SPECIFIC CUSTODY PROCEDURES

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact. The protocol for sample designations are included in the FSP, Appendix A, of this QAPP.

5.1.1 Initiation of Chain-of-Custody Field Procedures

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

All bottles will be tagged with sample numbers and locations.

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

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that

persons going to the site could re-construct a particular situation without reliance on memory.

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

The title page of each logbook will contain the following:

- person to whom the logbook is assigned,
- logbook number,
- project name,
- project start date, and
- project end date.

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

Measurements made and samples collected will be recorded. All entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The number of the photographs taken of the station, if any, will also be noted. The equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the FSP, Appendix A, of this QAPP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. A sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

5.1.3 Transfer of Custody and Shipment Procedures

Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When

transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of the samples from the sampler to another person to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

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

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

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

If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody forms as long as the custody forms are sealed inside the sample cooler, and the custody seals remain intact. The person shipping the samples should note the carrier name and airbill number on the chain-of-custody record.

5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

Laboratory custody procedures for the sample receiving and log-in; sample storage; tracking during sample preparation and analysis; and storage of data are described in the individual laboratory custody SOPs provided in Appendix D.

5.3 FINAL EVIDENCE FILE CUSTODY PROCEDURE

The final evidence file for the Beloit Corporation RI/FS will be located at and maintained by Warzyn. The content of the evidence file will include all relevant

records, reports, correspondence, logs, field logbooks, laboratory sample preparation and analysis logbooks/documentation, analytical data packages, pictures, subcontractor reports, chain-of-custody records/forms, data review reports, etc.. The evidence file will be under custody of the file custodian in a locked, secured area.

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6

CALIBRATION PROCEDURES AND FREQUENCY

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

6.1 FIELD INSTRUMENTS/EQUIPMENT

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

Equipment used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to determine that all prior equipment problems are not overlooked, and all necessary repairs to equipment have been carried out.

Calibration procedures and frequencies for field instruments are governed by the specific field SOPs provided in Appendix C. Field measurements include pH, specific conductivity, and temperature.

The field GC VOC screening instrument calibration requirements are similar to those for laboratory instruments, and are provided in Appendix C.

6.2 LABORATORY INSTRUMENTS

Procedures for the calibration of laboratory instruments must be established and maintained so that equipment is functioning properly and that data collected are accurate and reliable. Requirements include step-by-step calibration procedures, frequency of re-calibration, equipment maintenance logs, instrument accuracy criteria, corrective action procedures and equipment limitations (e.g., working ranges), and are described, in detail, in the SOPs provided in Appendix B, and the CLP SOWs for organics and inorganics.

7

ANALYTICAL PROCEDURES

Groundwater and soil samples collected during field sampling activities for the Beloit Corporation RI/FS will be analyzed by CompuChem and Warzyn. Refer to Table 1-1 for matrices, number of samples, parameters, and the laboratory performing the analysis.

7.1 FIELD SCREENING ANALYTICAL PROTOCOLS

The procedures for the field measurement of pH, temperature, and specific conductivity are described in the SOPs provided in Appendix C.

The procedure for the field GC VOC Screening analysis is described in the SOP provided in Appendix C.

7.2 LABORATORY ANALYSES

Samples (groundwater and soil samples) for CLP TCL organics will be analyzed by CompuChem according to the analytical procedures set forth in the current CLP SOW OLM02.0.

Samples (groundwater and soil samples) for CLP TAL inorganics will be analyzed by Warzyn according to the analytical procedures set forth in the current CLP SOW ILM01.0.

Groundwater samples for indicator parameters analyzed by Warzyn will follow the SOPs in Appendix B.

Analysis of soils for physical characteristics will be performed by the Warzyn Soils Laboratory using the methods provided in Appendix B.

Refer to Table 7-1 for a summary of analytical methods for all analyses.

8

INTERNAL QUALITY CONTROL CHECK

8.1 FIELD SAMPLE COLLECTION

The assessment of field sampling precision and accuracy will be made through the collection of field duplicates and field blanks in accordance with the procedures described in the FSP (Appendix A of this QAPP). Refer to Table 1-1 for a summary of sample numbers and required field QC samples.

8.2 FIELD MEASUREMENTS

QC procedures for field measurements of pH, specific conductivity, and temperature are limited to checking the reproducibility of the measurements by obtaining multiple readings on a single sample or standard, and by calibrating the instruments. Refer to Table 3-6 for a summary of QC requirements.

QC procedures for field GC VOC screening are similar to those used for laboratory analyses. Internal quality control checks are integrated into the analytical methods. The overall objectives of the internal quality control checks are to verify the established precision, accuracy and integrity of the methodology and to support the technical validity of the data. Internal quality control checks for field GC VOC analysis will include daily method blanks, field duplicates, and continuing calibration standards. Refer to Table 3-6 for a summary of QC requirements.

8.3 LABORATORY ANALYSIS

The laboratories used for the analysis of samples for the Beloit Corporation RI/FS (CompuChem and Warzyn Laboratories) have written QA/QC programs which provide rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory QAOs, which are independent of the operating departments. Laboratory procedures used are documented in writing and are provided or referenced in this QAPP. Internal quality control checks are an integral part of the analytical methods, and are discussed in detail within the analytical procedures. The overall objectives of the internal quality control checks are to verify the established precision, accuracy and integrity of the methodology and to support the technical validity of the data. Where appropriate,

internal quality control checks for other than those following the CLP SOWs will include method blanks, preparation/reagent blanks, calibration check samples, laboratory duplicates, matrix spikes and continuing calibration standards.

The required quality control frequency and performance criteria for TCL organics and TAL inorganics are summarized in the current CLP SOWs OLM02.0 and ILM01.0, respectively.

The required quality control frequency and performance criteria for the indicator parameters, and grain size distribution are summarized in Table 3-6.

The performing laboratories will document, in each data package provided, that both initial and ongoing instrument and analytical QC functions have been met. Samples analyzed in non-conformance with the QC criteria set forth, will be reanalyzed by the laboratory. It is expected that sufficient sample volume will be collected for reanalysis.

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9

DATA REDUCTION, VALIDATION, AND REPORTING

9.1 FIELD MEASUREMENTS AND SAMPLE COLLECTION

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. Data will be reviewed to ensure procedures were followed and QC requirements were met, however, no formal data validation effort will be performed. If the data are to be used in the project reports, they will be reduced onto data summary tables.

9.2 LABORATORY SERVICES

9.2.1 Data Reduction

Each laboratory is responsible for identification, quantification, data reporting, and data deliverables for the analyses performed. Data reduction of TCL organic will follow the requirements set forth in the CLP SOWs (OLM02.0). Data reduction of TAL inorganic and indicators data will follow the requirements set forth in the CLP SOW ILM01.0. Deliverables will include raw data, summaries of calibration standards, duplicates, spikes, blanks, and performance evaluation samples.

9.2.2 Data Validation

Organic data generated under a DQO Level of 4 will be validated by Warzyn using National Functional Guidelines for Organic Data Review, Revised June, 1991, or most current guidelines available for each fraction (volatiles, semivolatiles, and pesticides/PCBs).

Inorganic data generated under a DQO Level of 4 will be validated by Warzyn using Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, July 1988, or most current guidelines.

Indicator data generated under a DQO Level 3 will be validated by Warzyn using the Data Validation SOP provided in Appendix E.

Grain size distribution data (DQO Level 3) will be reviewed to ensure procedures were followed and QC requirements were met, however, no formal data validation effort will be performed.

Refer to Table 1-3 for a summary of data generating activities, intended data uses, and associated DQOs for the site.

9.2.3 Data Reporting

Analytical data generated for the site will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data qualifiers provided by the performing laboratory in accordance with the CLP SOWs, as well as qualifiers added by the data reviewer in accordance with the data validation procedures noted in Section 9.2.2 of this QAPP.

The laboratory-provided qualifiers will include such items as:

- non-detects,
- concentration below required detection limit,
- estimated concentration due to poor QC data, and
- concentration of chemical also found in the laboratory blank.

The data review qualifiers will indicate whether the data are:

- usable as a quantitative concentration,
- usable with caution as an estimated concentration, or
- unusable due to out-of-control QC results.

A summary of the validated data will be incorporated into the RI report.

10

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to determine that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPP. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

10.1 FIELD AUDITS

10.1.1 Internal Audits

Internal audits of field activities (sampling and measurements) will be conducted by the Warzyn QAO and/or RI Leader. The purpose of the field audit will be to evaluate and document adherence to procedures described in the QAPP and FSP. The audit will include review of field activities, sample documentation, chain-of-custody forms, field logbooks, and sampling and decontamination activities. Follow-up audits will be conducted to correct deficiencies, and to document that QA procedures are maintained throughout the investigation.

10.1.2 External Audits

External audits will be the responsibility of the IEPA.

10.2 LABORATORY AUDITS

10.2.1 Internal Audits

The purpose of the internal laboratory audit is to evaluate and document adherence to analytical procedures described in this QAPP. Internal audits of the participating laboratories are the responsibility of the individual laboratory QAO. System audits will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedure, sample preparation and analysis, instrument operating records, etc., and will be performed annually. Performance audits consisting of blind QC samples prepared and submitted to the laboratory for analysis, will be performed quarterly. Results of these blind QC samples will be reviewed by the laboratory QAO.

10.2.2 External Audits

The Illinois EPA DL/QAS may audit performing laboratories and provide recommendations for approval of the laboratory for the requested analyses to the IEPA RPM. The audit may consist of a review of analytical and chain-of-custody procedures, evaluation of performance samples, and may also include an on-site audit of each participating laboratory.

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11

PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENTS/EQUIPMENT

Field equipment used for this project include: thermometers, pH meter, conductivity meter, gas-tech meter, electronic water level indicator, and sampling and filtering equipment. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Field instruments will be checked and calibrated before they are transported to the field. These instruments will be checked and calibrated daily before use. Calibration checks will be performed as noted in Table 3-6 and will be recorded in the field logbook.

Critical spare parts such as electrodes, batteries, and pH probes will be kept on-site to minimize instrument down time. Backup instruments and equipment will be available to avoid delays in the field schedule.

Preventative maintenance of the gas chromatograph (GC) for screening of VOCs in the field will be as directed with manufacturer's specifications, instrument operating procedures, and the analytical method. Maintenance is carried out on a regular, scheduled basis, and is documented in the instrument service logbook. Emergency repair or scheduled manufacturer's maintenance is provided by an on-site technician or maintenance contract with the factory representative.

Routine preventative maintenance schedules are summarized in Table 11-1.

11.2 LABORATORY INSTRUMENTS

Preventative maintenance procedures for laboratory instrumentation and equipment for TCL organics are referenced in the current CLP SOW OLM02.0. Preventative maintenance procedures for laboratory instrumentation and equipment for TAL inorganics are referenced in the current CLP SOW ILM01.0.

Preventative maintenance of laboratory instruments associated with the indicator parameters will be as directed with manufacturer's specifications, instrument operating procedures, and analytical methods. Maintenance is carried out on a regular, scheduled basis, and is documented in the instrument service logbook for

each instrument. Emergency repair or scheduled manufacturer's maintenance is provided by an on-site technician or maintenance contract with the factory representatives.

Routine preventative maintenance schedules are summarized in Table 11-1.

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12

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

12.1 FIELD MEASUREMENTS

Field data will be assessed by the site QAO. The site QAO will review the field results for compliance with the established QC criteria that are specified in the QAPP and FSP. Accuracy of field measurements will be assessed using daily instrument calibration, calibration check, and blank data. Precision will be assessed on the basis of reproducibility by taking multiple readings of a single sample. Data completeness will be calculated as follows:

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

12.2 LABORATORY DATA

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis, and laboratory duplicate analyses for inorganic and indicator analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analysis results as follows:

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

where, S = first sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

Accuracy of laboratory results will be assessed based on the established QC criteria that are described in Section 3 of this QAPP, using the analytical results of

method blanks, matrix spike samples, field blanks, and bottle blanks. The percent recovery (%R) will be calculated as follows:

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

where, A = The analyte concentration determined experimentally from the spiked sample;
B = The background level determined by a separate analysis of the unspiked sample; and
C = The amount of the spike added.

12.2.3 Completeness

The data completeness of laboratory analysis results will be assessed for compliance with the amount of data required for decision making. Completeness is calculated by the following formula:

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

12.2.4 Sensitivity

The achievement of method detection limits depend on instrument sensitivity and matrix effects. It is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

13

CORRECTIVE ACTION

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

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Warzyn Project Manager, who in turn will notify the IEPA RPM. If the problem is analytical in nature, information on these problems will be promptly communicated to the Warzyn Project Manager, who in turn will notify the RPM or IEPA QAS. Implementation of corrective action will be confirmed in writing through the same channels.

Any noncompliance with the established quality control procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The IEPA RPM, or his designee will issue a nonconformance report for each nonconformance condition.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by stop-work order by the RPM.

13.1 SAMPLE COLLECTION/FIELD MEASUREMENT

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Warzyn RI Leader, or his designee. This manager, in consultation with the Warzyn Project Manager, will be responsible for assessing the suspected problems and making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the manager.

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

- evaluating all reported nonconformances;
- controlling additional work on nonconforming items;
- determining disposition or action to be taken;
- maintaining a log of nonconformances;
- reviewing nonconformance reports and corrective actions taken; and
- ensuring nonconformance reports are included in the final site documentation in the project files.

If appropriate, the Warzyn Project Manager will determine that, no additional work that is dependent on the nonconforming activity, is performed until the corrective actions are completed.

Corrective action for field measurements may include:

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

The Warzyn RI Leader, or his designee, is responsible for all site activities. In this role, the Warzyn RI Leader, at times is required to adjust the site programs to accommodate the site-specific needs. When it becomes necessary to modify a program, the Warzyn RI Leader notifies the Warzyn Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the Project Manager. The change in the program will be documented and signed by the appropriate personnel.

The Warzyn RI Leader for the site RI/FS is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties, including the IEPA RPM, the Warzyn Project Manager, and the Beloit Corporation Project Manager.

13.2 LABORATORY ANALYSIS

Corrective actions are required when an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis

and the event. Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA Department during internal or external audits , or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level, by the analyst who reviews the sample preparation procedure for possible errors and checks the instrument calibration, spike and calibration mixes, instrument sensitivity, etc.. If the problem persists, or cannot be identified, the matter is referred to the laboratory supervisor, manager, or QAO for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA Department.

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14

QUALITY ASSURANCE REPORTS TO MANAGEMENT

Reports will be submitted to the IEPA as described in the Work Plan. Reports will consist of monthly progress reports, technical memoranda, and the draft RI report.

Monthly progress reports submitted to the IEPA will include:

- a summary of the validated sampling and testing results,
- a description of activities completed during the past month, as well as actions which are scheduled for the next month,
- a summary of target and actual completion dates for each activity,
- changes in key personnel,
- problems encountered and how they were resolved, and
- anticipated problems and recommended solutions.

The results of specific RI activities such as the Risk Assessment, and Site Characterization will be submitted to the IEPA in the form of Draft Technical Memoranda. Technical Memoranda will include the following:

- Source Characterization Results,
- Contaminant and Migration Pathways Characterization Results, and
- Baseline Risk Assessment.

A draft RI Report summarizing the RI activities will be submitted to the IEPA. The report will characterize the site and summarize the data collected and conclusions. The RI report will not be considered final until site characterization activities are complete for the supporting remedial alternatives screening activities and a letter of approval is issued by the IEPA RPM.

TABLES

TABLE 1-1
Sample Type and Estimated Sample Numbers
Beloit Corporation RI/FS

Sample[1] Matrix	Lab[2]	No. of Samples	Field Duplicates	Field[3] Blanks	MS/MSD[4]	Total No. Samples	Test[5,6] Parameters	Field Parameters
SOURCE CHARACTERIZATION								
Soil Gas Survey	Field	100	10	1/day	-	110+	Field GC Volatiles	
Surface & Shallow Soils	CompuChem	36	4	-	2	40	TCL Organics	
	Warzyn II	36	4	-	2	40	TAL Inorganics	
Soil Borings	Warzyn I	20	-	-	-	20	Grain Size Analysis	
Groundwater Screen	Field	55	6	1/day	-	61+	Field GC Volatiles	
Groundwater (per round)	CompuChem	31	4	4	2	39	TCL Organics	pH, Conductivity Temperature
	Warzyn I	31	4	4	2	39	TAL Inorganics	
	Warzyn II	31	4	4	2	39	Indicators	

Notes:

1. Samples will be considered low concentration, and will be packaged and shipped accordingly.

2. CompuChem
   3308 Chapel Hill/Nelson Highway
   Research Triangle Park, NC 27709

   Warzyn Soils Laboratory (I)
   505 Science Drive
   University Research Park
   Madison, WI 53711

   Warzyn Analytical Laboratory (II)
   One Science Court
   University Research Park
   Madison, WI 53711

3. A trip blank for VOC analysis will be included with each cooler shipped for aqueous groundwater samples. Trip blanks are not included in the total number of samples.

4. EXTRA VOLUME REQUIREMENT: Extra volume is required for the MS/MSD quality control requirement (triple volume for VOCs, double volume for SVOCs and Pest/PCBs. Inorganics and general water quality indicator parameters require duplicate and spike analyses, however, do not require additional sample volume to meet the specified QC. MS/MSD samples are not included in the total number of samples.

5. Refer to Tables 3-1 and 3-2 for the TCL organic and TAL inorganic parameter lists and required detection limits.

6. Groundwater samples for metals analysis will be field filtered through a 0.45 micron filter prior to the addition of preservatives.

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TABLE 1-2
Sample Quantities, Containers, Preservatives and Packaging Requirements
Beloit Corporation Facility RI/FS

Analysis	Bottles and Jars	Preservation	Technical Holding Time(2)	Volume of Samples	Shipping	Normal Packaging (1)
Low Concentration (Organics)						
Groundwater Volatiles	Four 40-mL volatile organic analysis (VOA) vial.	1:1 HCL (2 drops/ vial), iced to 4°C.	14 days from sampling date	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite
Semi-volatiles	Two 1-Liter amber glass bottles	Iced to 4°C	Extract 7 days from sampling, analysis within 40 days after extraction	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
Pesticides/PCBs	Two 1-Liter amber glass bottles	Iced to 4°C	Extract 7 days from sampling, analysis within 40 days after extraction	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
Low Concentration (Inorganics)						
Groundwater Metals	One 1-liter high density polyethylene bottle	Field filter through 0.45 um filter. HNO_3 to pH<2. Iced to 4°C	180 days from sampling (26 days from sampling for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Cyanide	One 1-liter high density polyethylene bottle	Add NaOH to pH>12. Iced to 4°C	12 days from sampling	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Water Quality Indicator Parameters						
Groundwater Nitrate+Nitrite-N, Ammonia	One 1-liter high density polyethylene bottle	Field filter through 0.45 um filter. H_2SO_4 to pH<2. Iced to 4°C.	28 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Total-Phenolics	One 1-liter amber glass bottle	H_2SO_4 to pH<2. Iced to 4°C.	28 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite

TABLE 1-2
(Continued)

Analysis	Bottles and Jars	Preservation	Technical Holding Time(2)	Volume of Samples	Shipping	Normal Packaging (1)
Alkalinity, Chloride, and Sulfate	One 1-liter high density polyethylene bottle	Field filter through 0.45 um filter. Iced to 4°C	28 days (14 days alkalinity)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
TDS	One 500-mL polyethylene bottle	Field filter through 0.45 um filter. Iced to 4°C	7 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Low or Med Concentration (Organics)						
Soil and Sediment Samples Semi-volatiles	One 8-oz wide mouth glass jar	Iced to 4°C	Extract 7 days from sampling, analysis within 40 days after extraction	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite (Med in cans/ vermiculite)
Pesticides/PCBs	One 8-oz wide mouth glass jar	Iced to 4°C	Extract 7 days from sampling, analysis within 40 days after extraction	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite (Med in cans/ vermiculite)
Volatiles	Two 4-oz wide mouth glass jars	Iced to 4°C	14 days from sampling date	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite (Med in cans/ vermiculite)
Low or Med Concentration (Inorganics)						
Soil and Sediment Samples Metals and Cyanide	One 8-oz wide mouth glass jar	Iced to 4°C	180 days from sampling (28 days from sampling for mercury and 14 days from sampling for cyanide)	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite (Med in cans/ vermiculite)

TABLE 1-2
(Continued)

Analysis	Bottles and Jars	Preservation	Technical Holding Time(2)	Volume of Samples	Shipping	Normal Packaging (1)
Physical Analysis						
Soil Samples						
Grain size analysis	Two 8-oz wide mouth glass jars	NONE	Not established	Fill 3/4 full	Ship by carrier	Vermiculite

Notes:

1. The packing material should completely cushion the sample bottles - botttom, sides and top.
2. Technical hold times begin on the date sampled, and supersede contractual SOW hold times.

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TABLE 1-3

Summary of Data Generating Activities and Associated Quality Objectives
Beloit Corporation Facility RI/FS

Activity	Description	Intended Data Usages	Parameters	Data Quality Objective	Anticipated No. of Investigative Samples
SOURCE CHARACTERIZATION					
Soil Gas Survey	Collect soil gas from shallow soil probes and analyze for VOCs.	Identify potential source areas within the site boundaries.	Field GC Volatiles	Level II data	100
Surface Soils	Collect and analyze 12 surface samples including 1 background location for TCL and TAL parameters.	Characterize the nature of the surface soil contamination.	TCL Organics TAL Inorganics	Level IV Data Level IV Data	12 12
Shallow Soil Borings	Collect and analyze 2 samples each from 12 shallow soil borings including 1 background location for TCL and TAL parameters.	Characterize the nature of the subsurface soil contamination.	TCL Organics TAL Inorganics	Level IV Data Level IV Data	24 24
Deep Soil Borings	Drill 2 intermediate and 5 deep borings. Collect and analyze groundwater at 10 ft intervals for VOCs.	Soil borings will be used to characterize site geology, and screen groundwater for VOCs with changes in depth.	Field GC VOCs	Level II Data	57
MIGRATION PATHWAY ASSESSMENT					
Hydrogeologic Investigation	22 soil borings will be drilled, with soils to be collected and analyzed at selected depths. Install monitoring wells at 8 locations.	Describe geologic strata to determine groundwater flow direction and rate through soil boring logs, water level and hydraulic conductivity data, physical data, etc.	Grain Size Analysis	Level III	22
CONTAMINANT CHARACTERIZATION					
Groundwater Sampling	Sample and analyze 31 monitoring wells in 2 Rounds for TCL, TAL and indicator parameters.	Characterize and evaluate the contamination on site.	TCL Organics TAL Inorganics (1) Indicators (2)	Level IV Level IV Level III	31 31 31

NOTES:

1. Groundwater samples to be filtered for dissolved TAL Inorganic analysis and indicator parameters.

2. Indicator parameters consist of: chloride, sulfate, alkalinity, nitrate and nitrite-nitrogen, ammonia-nitrogen, total-phenolics, and TDS.

JAH/kml/JEM/JDD/BLH
[mad-403-008b]
1526800.137

TABLE 3-1

Target Compound List
and Contract Required Quantitation Limits - Organics

	Compound	Quantitation Limits* Water (ug/l)	Low Soil (ug/kg)	Medium Soil (ug/kg)
	Volatiles			
1.	Chloromethane	10	10	1200
2.	Bromomethane	10	10	1200
3.	Vinyl chloride	10	10	1200
4.	Chloroethane	10	10	1200
5.	Methylene chloride	10	10	1200
6.	Acetone	10	10	1200
7.	Carbon disulfide	10	10	1200
8.	1,1-Dichloroethene	10	10	1200
9.	1,1-Dichloroethane	10	10	1200
10.	1,2-Dichloroethene (total)	10	10	1200
11.	Chloroform	10	10	1200
12.	1,2-Dichloroethane	10	10	1200
13.	2-Butanone	10	10	1200
14.	1,1,1-Trichloroethane	10	10	1200
15.	Carbon tetrachloride	10	10	1200
16.	Bromodichloromethane	10	10	1200
17.	1,2-Dichloropropane	10	10	1200
18.	cis-1,3-Dichloropropene	10	10	1200
19.	Trichloroethene	10	10	1200
20.	Dibromochloromethane	10	10	1200
21.	1,1,2-Trichloroethane	10	10	1200
22.	Benzene	10	10	1200
23.	trans-1,3-Dichloropropene	10	10	1200
24.	Bromoform	10	10	1200
25.	4-Methyl-2-pentanone	10	10	1200

TABLE 3-1
(CONTINUED)

Compound	Quantitation Limits* Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
26. 2-Hexanone	10	10	1200
27. Tetrachloroethene	10	10	1200
28. Toluene	10	10	1200
29. 1,1,2,2-Tetrachloroethane	10	10	1200
30. Chlorobenzene	10	10	1200
31. Ethylbenzene	10	10	1200
32. Styrene	10	10	1200
33. Xylenes (total)	10	10	1200
Semi-volatiles			
34. Phenol	10	330	10000
35. bis(2-Chloroethyl) ether	10	330	10000
36. 2-Chlorophenol	10	330	10000
37. 1,3-Dichlorobenzene	10	330	10000
38. 1,4-Dichlorobenzene	10	330	10000
39. 1,2-Dichlorobeznene	10	330	10000
40. 2-Methylphenol	10	330	10000
41. 2,2'-oxybis-(1-Chloropropane)	10	330	10000
42. 4-Methylphenol	10	330	10000
43. n-Nitroso-di-n-dipropylamine	10	330	10000
44. Hexachloroethane	10	330	10000
45. Nitrobenzene	10	330	10000
46. Isophorone	10	330	10000
47. 2-Nitrophenol	10	330	10000
48. 2,4-Dimethylphenol	10	330	10000
49. bis(2-Chloroethoxy) methane	10	330	10000
50. 2,4-Dichlorophenol	10	330	10000
51. 1,2,4-Trichlorobenzene	10	330	10000
52. Naphthalene	10	330	10000
53. 4-Chloroaniline	10	330	10000
54. Hexachlorobutadiene	10	330	10000
55. 4-Chloro-3-methylphenol	10	330	10000
56. 2-Methylnaphthalene	10	330	10000

TABLE 3-1
(CONTINUED)

Compound	Quantitation Limits* Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
57. Hexachlorocyclopentadiene	10	330	10000
58. 2,4,6-Trichlorophenol	10	330	10000
59. 2,4,5-Trichlorophenol	25	800	25000
60. 2-Chloronaphthalene	10	330	10000
61. 2-Nitroaniline	25	800	25000
62. Dimethylphthalate	10	330	10000
63. Acenaphthylene	10	330	10000
64. 2,6-Dinitrotoluene	10	330	10000
65. 3-Nitroaniline	25	800	25000
66. Acenaphthene	10	330	10000
67. 2,4-Dinitrophenol	25	800	25000
68. 4-Nitrophenol	25	800	25000
69. Dibenzofuran	10	330	10000
70. 2,4-Dinitrotoluene	10	330	10000
71. Diethylphthalate	10	330	10000
72. 4-Chlorophenyl-phenyl ether	10	330	10000
73. Fluorene	10	330	10000
74. 4-Nitroaniline	25	800	25000
75. 4,6-Dinitro-2-methylphenol	25	800	25000
76. n-Nitrosodiphenylamine	10	330	10000
77. 4-Bromophenyl-phenyl ether	10	330	10000
78. Hexachlorobenzene	10	330	10000
79. Pentachlorophenol	25	800	25000
80. Phenanthrene	10	330	10000
81. Anthracene	10	330	10000
82. Carbazole	10	330	10000
83. Di-n-butylphthalate	10	330	10000
84. Fluoranthene	10	330	10000
85. Pyrene	10	330	10000
86. Butylbenzylphthalate	10	330	10000
87. 3,3'-Dichlorobenzidine	10	330	10000
88. Benzo(a)anthracene	10	330	10000

TABLE 3-1
(CONTINUED)

Compound	Quantitation Limits* Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
89. Chrysene	10	330	10000
90. bis(2-Ethylhexyl)phthalate	10	330	10000
91. Di-n-ocylphthalate	10	330	10000
92. Benzo(b)fluoranthene	10	330	10000
93. Benzo(k)fluoranthene	10	330	10000
94. Benzo(a)pyrene	10	330	10000
95. Indeno(1,2,3-cd)pyrene	10	330	10000
96. Dibenzo(a,h)anthracene	10	330	10000
97. Benzo(g,h,i)perylene	10	330	10000
Pesticides/PCBs			
98. alpha-BHC	0.05	1.7	1.7
99. beta-BHC	0.05	1.7	1.7
100. delta-BHC	0.05	1.7	1.7
101. gamma-BHC (Lindane)	0.05	1.7	1.7
102. Heptachlor	0.05	1.7	1.7
103. Aldrin	0.05	1.7	1.7
104. Heptachlor epoxide	0.05	1.7	1.7
105. Endosulfan I	0.05	1.7	1.7
106. Dieldrin	0.10	3.3	3.3
107. 4,4'-DDE	0.10	3.3	3.3
108. Endrin	0.10	3.3	3.3
109. Endosulfan II	0.10	3.3	3.3
110. 4,4'-DDD	0.10	3.3	3.3
111. Endosulfan sulfate	0.10	3.3	3.3
112. 4,4'-DDT	0.10	3.3	3.3
113. Methoxychlor	0.50	17	17
114. Endrin ketone	0.10	3.3	3.3
115. Endrin aldehyde	0.10	3.3	3.3
116. alpha-Chlordane	0.05	1.7	1.7
117. gamma-Chlordane	0.05	1.7	1.7
118. Toxaphene	5.0	170	170
119. Aroclor-1016	1.0	33	33
120. Aroclor-1221	2.0	67	67

TABLE 3-1
(CONTINUED)

Compound	Quantitation Limits* Water (ug/L)	Low Soil (ug/kg)	Medium Soil (ug/kg)
121. Aroclor-1232	1.0	33	33
122. Aroclor-1242	1.0	33	33
123. Aroclor-1248	1.0	33	33
124. Aroclor-1254	1.0	33	33
125. Aroclor-1260	1.0	33	33

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis as required by the method, will be higher.

JAH/kml/JDD/BLH
[mad-403-008c]
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TABLE 3-2

Target Analyte List and
Contract Required Detection Limits - Inorganics

	Analyte	Contract Required [1,2] Detection Limit (ug/L)
1.	Aluminum	200
2.	Antimony	60
3.	Arsenic	10
4.	Barium	200
5.	Beryllium	5
6.	Cadmium	5
7.	Calcium	5000
8.	Chromium	10
9.	Cobalt	50
10.	Copper	25
11.	Iron	100
12.	Lead	3
13.	Magnesium	5000
14.	Manganese	15
15.	Mercury	0.2
16.	Nickel	40
17.	Potassium	5000
18.	Selenium	5
19.	Silver	10
20.	Sodium	5000
21.	Thallium	10
22.	Vanadium	50
23.	Zinc	20
24.	Cyanide	10

Notes:

1. Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods - Catastrophic Failure) any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

   If the sample concentration exceeds five times the detection limit of the instrument or method in use, the values may be reported even though the instrument or method detection limit may no equal the CRDL. This is illustrated in the example below:

TABLE 3-2
(Continued)

For lead:
Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 200
CRDL = 3

The value of 200 may be reported even though the instrument detection limit is greater than the CRDL. The instrument or method detection limit must be documented as described in Exhibit E.

2. The CRDL are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix. Soil/sediment detection limits are approximately 200 times the CRDLs noted for water, and may vary as the soil/sediment results are reported on a dry weight basis. Soil/sediment results are reported in mg/kg.

JAH/kml/JDD/BLH
[mad-403-008d]
1526800.137

TABLE 3-3

Indicators, Field Measurements, and Physical Characteristics Parameter Lists and Required Detection Limits

Indicator Parameter	Required Detection Limit (mg/L)
Alkalinity	10
Chloride	2
Sulfate	10
Total Dissolved Solids	20
Nitrate+Nitrite Nitrogen	0.02
Ammonia Nitrogen	0.10
Total Phenolics	0.005

Field Measurement Parameter	Required Detection Limit
pH (s.u.)	sensitivity of 0.05
Conductivity @25 Deg. C (umhos/cm)	10
Temperature (Deg. C)	to nearest 0.5 degree

Physical Characteristic Parameter	Required Detection Limit
Grain size distribution	NA

NA = Not applicable to method.

JAH/kml/JDD/BLH
[mad-403-008e]
1526800.137

TABLE 3-4

Field GC VOC Target Analyte List Detection Limits

Compound	Detection Limit ng on column	Groundwater Reportable Detection Limit (ug/L)	Soil Gas Reportable Detection Limit (ug/L-Vol)
Benzene	10	2.0	5.0
1,1-Dichloroethane	10	2.0	5.0
1,2-Dichloroethane	10	5.0	5.0
1,1-Dichloroethene	5	1.0	5.0
cis-1,2-Dichloroethene	5	1.0	5.0
trans-1,2-Dichloroethene	5	1.0	5.0
Ethylbenzene	10	2.0	5.0
Tetrachloroethene	5	1.0	5.0
Toluene	5	1.0	5.0
1,1,1-Trichloroethane	5	1.0	5.0
Trichloroethene	5	1.0	5.0
Xylenes	15	3.0	5.0

JAH/kml/JDD/BLH
[mad-403-008f]
1526800.137

TABLE 3-5

Summary of Physical Characteristics Method References

Test	Method Reference
Grain Size Analysis	ASTM D422

JAH/kml/JDD/BLH
[mad-403-008g]
1526800.137

TABLE 3-6

Summary of Quality Control Requirements
Beloit Corporation Facility RI/FS

PARAMETER	AUDIT	FREQUENCY[1]	LIMITS[2]
TCL Organics	Requirements per OLM01.0 (or most current)		
TAL Inorganics	Requirements per ILM02.0 (or most current)		
Field GC VOCs			
	Method Blank	daily	< Detection Limit (DL)
	Check Standard	After every 10 samples and at the end of the run.	70 - 130 % Recovery
	Duplicate	1 per 10 samples	25% RPD (± 2xDL if sample concentration is <5 x DL)
Alkalinity, Chloride, Sulfate, Nitrate + Nitrite Nitrogen			
	Lab Blank	After calibration, every 10 samples, and at the end of the run.	< Detection Limit (DL)
	Check Standard	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
Total Dissolved Solids			
	Lab Blank	1 per set	< DL
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
Ammonia Nitrogen, Total Phenolics			
	Lab Blank	After calibration, every 10 samples, and at the end of the run.	< DL
	Check Standard	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	Preparation Blank	1 per set	< DL

TABLE 3-6
(continued)

PARAMETER	AUDIT	FREQUENCY[1]	LIMITS[2]
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	20% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
Grain Size Distribution	Lab Duplicate	1 per 10 samples	10% RPD or <2% by weight
pH (Field)	Check Standard	1 per 10 samples	± 0.05 pH unit of buffer selection
	Duplicate	1 per 10 samples	± 0.2 pH unit
Specific Conductance (Field)	Check Standard	1 per 10 samples	± 5% of standard
	Duplicate	1 per 10 samples	15% RPD (± 2xDL if sample concentration is <5 x DL)

1 Frequencies apply to each individual matrix.

2 Refer to Appendix B for required detection limits for each analyte.

JAH/kml/JDD/BLH
[mad-403-008h]
1526800.137

TABLE 7-1

Summary of Analytical Methods
Beloit Corporation Facility RI/FS

Parameter	SOP[1]	Method[2]
TCL Volatiles	SOW OLM02.0	CLP Protocol
TCL Semi-volatiles	SOW OLM02.0	CLP Protocol
TCL Pesticides/PCBs	SOW OLM02.0	CLP Protocol
TAL Inorganics	SOW ILM02.0	CLP Protocol
Alkalinity	LAAC/ALKAAC	EPA 310.2
Total dissolved solids	TDSC3	EPA 160.1
Chloride	CLAAC2	EPA 325.2
Sulfate	SO4AAC3	EPA 375.2
Ammonia nitrogen	NH3DTC	EPA 350.2
Nitrite + nitrate nitrogen	NO_2+NO_3 AAC2	EPA 353.2
Total Phenolics	PHENDISC2 PHENAAC1	EPA 420.1
Field Measurements		
Field GC VOCs	BC-FGC	SW-846 8010/8020
pH	pH1	EPA 150.1
Specific conductance	CONDYSIF	EPA 120.1

(1) SOP refers to either the EPA SOW, or the Warzyn laboratory document number.

(2) Method refers to the published analytical reference.

JAH/vlr/JDD/BLH [mad-403-008i]
1526800.137

TABLE 11-1

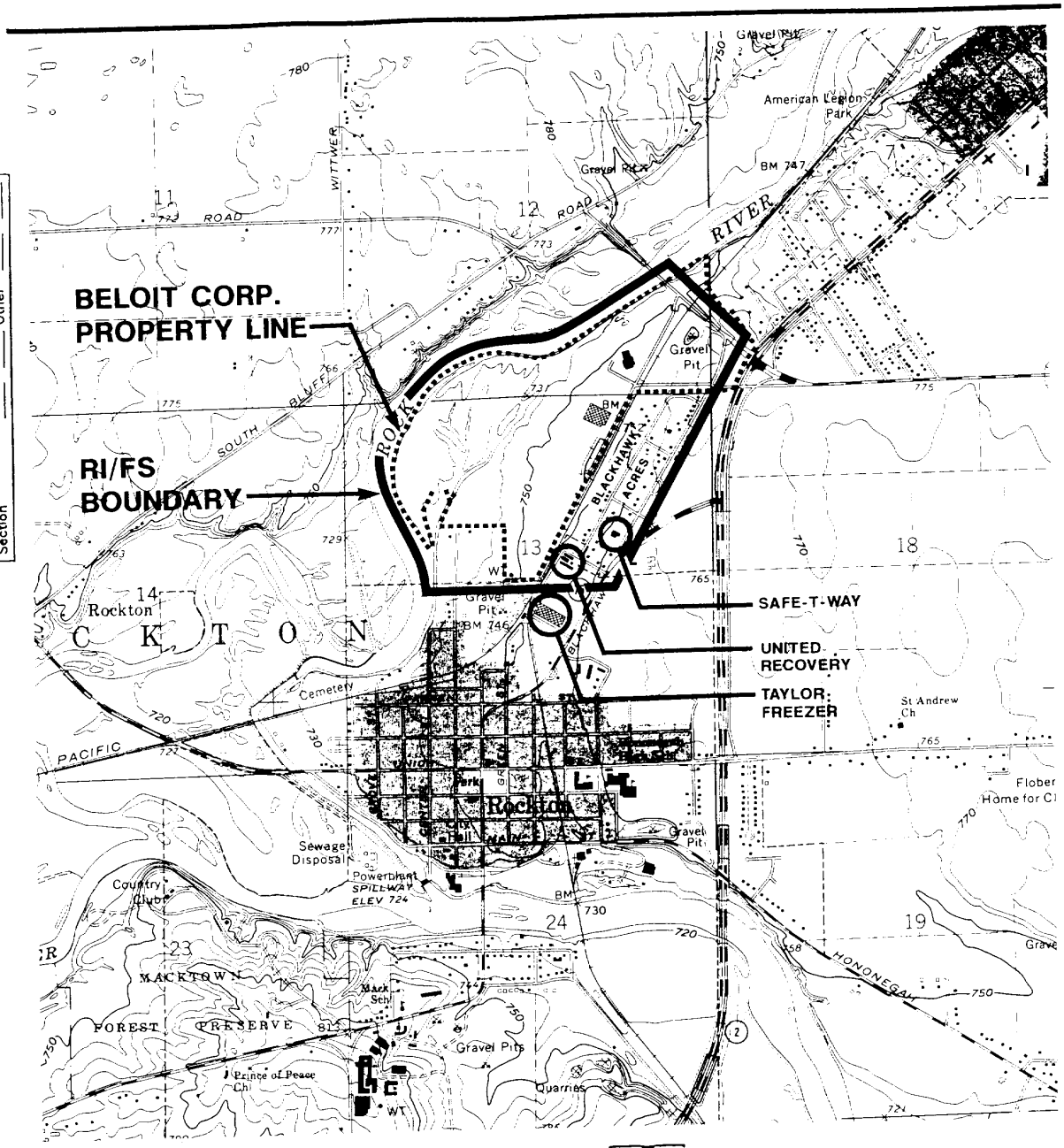
Routine Preventative Maintenance Procedures and Schedules
Beloit Corporation Facility RI/FS

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
CompuChem Instrumentation:		
Gas Chromatograph/Gas Spectrometry (GC/MS)-Volatiles	1. Change septa as needed. 2. Column maintenance as needed. 3. Clean injection port liner as needed. 4. Backflush purge and trap device as needed. 5. Change trap as needed.	1. Septa 2. GC columns 3. Syringes 4. Various electronic components 5. Traps
GC/MS - Semivolatiles	1. Change septa every 12 hours. 2. Clean or replace injection port liner every 12 hours. 3. Clean injection port every 12 hours. 4. Column maintenance very 12 hours.	1. Septa 2. Syringes 3. GC columns 4. Injection port liner 5. Various electronic components
GC - Pesticides/PCBs	1. Replace septa every 72 hours. 2. Replace first 2-3 in. of column packing every 72 hours. 3. Replace injector port liner every 72 hours. 4. Clean injector port with a series of four solvents every 72 hours.	1. Septa 2. Syringes 3. Column packing material 4. Injector port liner 5. Various electronic components
Warzyn Instrumentation:		
Graphite Furnace Atomic Absorption Spectrometer	1. Clean furnace chamber, contact rings, and furnace windows with alcohol daily. 2. Change contact rings as needed. 3. Ensure sufficient supply of D.I. H_2O in rinse bottle, daily. 4. Check gas pressure, daily.	1. Contact rings 2. Graphite tubes 3. Spare shroud
Flame Atomic Absorption Spectrometer	1. Clean burner head when necessary. 2. Check water trap to ensure it is full and clean prior to analysis. 3. Empty drain vessel when necessary. 4. Clean/inspect/optimize nebulizer monthly or as needed. 5. Clean & check o-rings monthly or as needed. 6. Rinse spray chamber with D.I. H_2O when run is completed.	1. Burner Head 2. Nebulizer tubing 3. O-rings 3. O-rings 4. Nebulizer
Inductively Coupled Plasma Spectrometer (ICP)	A. Daily 1. Check peristaltic pump rollers to ensure all are moving freely. 2. Change pump tubing. 3. Perform BEC check. 4. Rinse spray chamber with dilute HNO_3 followed by D.I. H_2O when finished with last run of day. 5. Check to ensure gas supply is adequate.	1. Torch 1. Torch 2. O-rings 3. Spray chamber 4. RF Coil 5. Nebulizer Tips 6. Pump tubing

TABLE 11-1
(continued)

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
	B. As Needed 1. Replace or clean nebulizer tips. 2. Disassemble torch, clean, and replace o-rings. 3. Perform wavelength calibration.	
Cold Vapor Atomic Absorption Spectrometer	1. Clean/inspect quartz cell daily. 2. Change pump tubing daily. 3. DI water rinse 10-15 minutes at end of run. 4. Check gas pressure daily. 5. Empty drain vessel daily.	1. Pump tubing 2. Quartz cell 3. Sampler probe
Lachat Autoanalyzer	1. Rinse manifold with degassed Milli-Q water before analysis. 2. Rinse manifold with recommended cleaning solution and degassed Milli-Q water after analysis. 3. Inspect pump tubings before analysis; replace if discolored or distorted. 4. Backup data files from computer onto a floppy disk every week. 5. Add water to heating block when necessary. 6. Clean work area daily.	1. Pump tubes 2. Quartz cell 3. Light bulbs
Spectrophotometer	1. Monitor and document absorbance for each analysis. 2. Rinse with DI water before and after analysis.	1. Light source bulbs
Ovens	1. Clean every month. 2. Monitor and document temperature daily.	1. Thermometer
pH Meter	1. Daily calibration. 2. Daily inspection of filling solution; add or replace when necessary.	1. Filling solution 2. pH buffers
Thermometers	1. Biannual calibration with an ASTM certified thermometer.	
Balances	1. Daily calibration with ASTM weights (S class). 2. Yearly calibration by an ASTM certified technician. 3. Daily cleaning.	
Warzyn Field Instrumentation:		
Gas Chromatograph -Field GC VOCs	1. Change septa as needed. 2. Column maintenance as needed. 3. Clean injection port liner as needed.	1. Septa 2. GC columns 3. Syringes
pH Meter	1. Daily calibration. 2. Replace electrodes as needed. 3. Spare electrodes	1. pH buffers 2. Batteries
Conductivity Meter	1. Daily calibration. 2. Check redline and replace batteries if does not calibrate.	1. Batteries

FIGURES



QUALITY CONTROL
Drawing Standards DLF
Lead Professional JSM
Section
INITIALS
DATE
KJA
4-5-90 Pm
4-5-90 Division
Other

NOTE

1. BASE MAP DEVELOPED FROM THE SOUTH BELOIT, ILLINOIS-WISCONSIN 7½ MINUTE U.S.G.S. TOPOGRAPHIC QUADRANGLE MAP, DATED 1971.

north

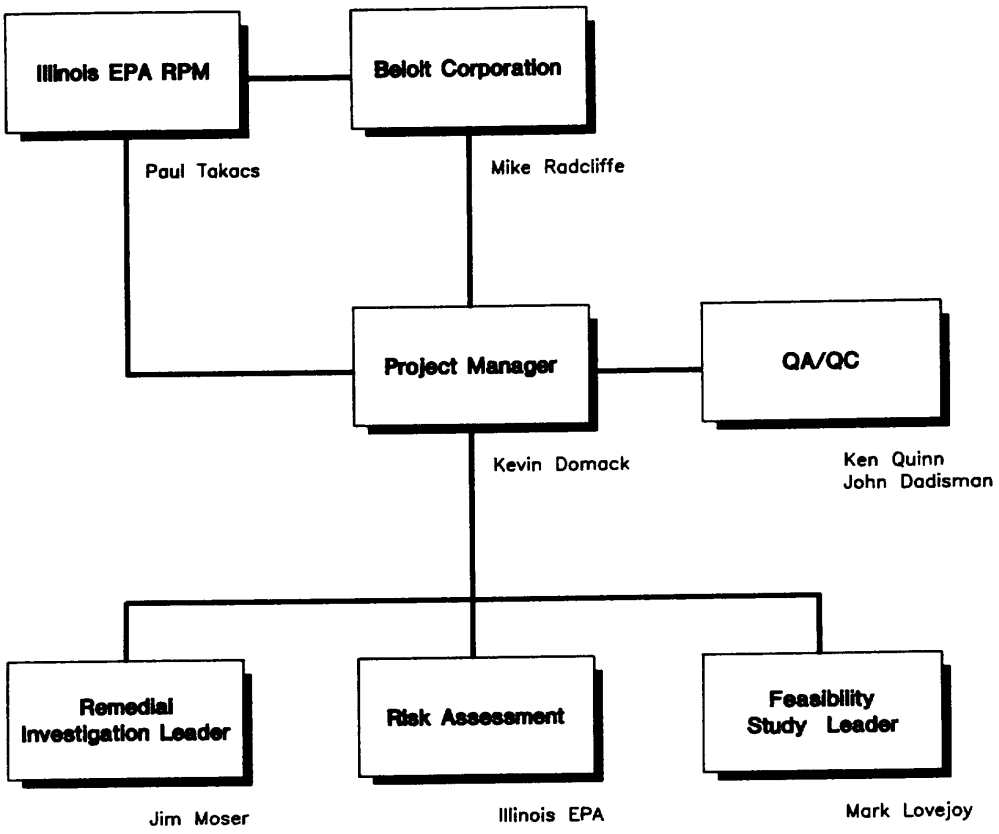
0 2000 4000

SCALE IN FEET

WARZYN	SITE LOCATION MAP SITE EVALUATION REPORT BELOIT CORPORATION ROCKTON, ILLINOIS	Drawn LCL Revisions	Checked TJK	App'd. LEA Date 10-4-91 15268 A1

WARZYN

Figure 2-1
Project Organization Chart
Remedial Investigation/Feasibility Study
Beloit Corporation, Rockton Facility



APPENDICES

Appendix A

Field Sampling Plan (FSP)

PROJECT
1526800

FIELD SAMPLING PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

BELOIT CORPORATION
ROCKTON FACILITY
ROCKTON, ILLINOIS

JUNE 1992

PREPARED FOR:
BELOIT CORPORATION
ROCKTON, ILLINOIS

...

PREPARED BY:
WARZYN INC.
MADISON, WISCONSIN

PROJECT
1526800

FIELD SAMPLING PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

BELOIT CORPORATION
ROCKTON FACILITY
ROCKTON, ILLINOIS

JUNE 1992

PREPARED FOR:
BELOIT CORPORATION
ROCKTON, ILLINOIS

...

PREPARED BY:
WARZYN INC.
MADISON, WISCONSIN

Kevin J. Domack
Project Manager

James E. Moser
RI Task Manager

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1

OBJECTIVES

The primary objective of the activities described in the Field Sampling Plan (FSP) is to obtain representative data for use during the Remedial Investigation/Feasibility Study (RI/FS) analysis. Sampling activities described in the FSP will be performed during the site investigation activities of the RI. Documentation procedures for sampling activities are described in the Data Management Plan (Work Plan, Appendix A).

The RI will focus on the definition of potential sources of soil or groundwater contamination, determine the nature and extent of contamination and collect data for the Baseline Risk Assessment and Feasibility Study.

The RI is designed to be implemented as an integrated investigation. Data collected during initial RI activities will be used to determine appropriate sampling activities and monitoring locations for subsequent activities of the RI. During initial RI activities, potential contaminant source areas will be identified and chemically characterized (Source Characterization), and potential contaminant migration pathways will be evaluated (Migration Pathway Assessment). Data generated from the Source Characterization and the Migration Pathway Assessment may be used in conjunction with data from the Site Evaluation Report (SER) to evaluate the extent and magnitude of contamination along the potential migration pathways. The magnitude and extent of contamination will be determined during Contaminant Characterization, by sampling media of concern.

Source Characterization data use objectives include:

- Identify the location and extent of potential contaminant source areas; and
- Characterize the chemical and physical aspects of potential contaminant source areas and contaminant source materials.

Migration Pathway Assessment data use objectives include:

- Identify the most likely pathways for contaminant migration;
- Characterize the hydrogeologic and hydrologic setting of the site; and

- Describe the characteristics of soils and sediments in the site area.

Contaminant Characterization data use objectives include:

- Describe the magnitude of groundwater, surface soil, and subsurface soil contamination; and

- Evaluate the extent of contaminant migration along potential migration pathways at the site.

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2

SCOPE OF SAMPLING ACTIVITIES

The FSP describes the procedures and practices for use in performing site investigation activities during the RI/FS. These procedures include a description of the sample designation system, personnel and their responsibilities, and the sampling methods to be used during RI activities. The scope of RI activities discussed in the FSP include:

- soil gas sampling;
- shallow soil borings and soil sampling;
- geophysical surveying (surface and downhole);
- site mapping;
- evaluation of existing wells;
- deep and intermediate groundwater quality borings and groundwater sampling;
- monitoring well installation/replacement and soil sampling;
- existing monitoring well and standpipe abandonment;
- hydraulic conductivity testing;
- water level measurements;
- surface water and sediment investigation;
- surface soil assessment;
- air assessment;
- ecological assessment; and
- groundwater sampling and chemical analysis.

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3

SAMPLING LOCATIONS AND NUMBERS OF SAMPLES

3.1 SOURCE CHARACTERIZATION

Source characterization will include soil gas sampling, shallow soil borings and soil sampling, and a electromagnetic (EM)/magnetometer surface geophysical survey.

3.1.1 Soil Gas Survey

A soil gas survey will be performed in known suspected contaminant source areas as well as areas where potential sources may exist. The objective of this survey is to locate potential sources of volatile organic compounds (VOCs). Previous investigations have indicated that several potential source areas may exist. However, other identified source areas may exist in the site vicinity. Drawing 15268-6 shows the proposed locations for the soil gas survey. Approximately 100 samples, ten field duplicate samples, and one field blank per day will be collected. Each sample will be analyzed by an in-field, portable gas chromatograph (GC) for target VOCs (Method EPA 8010/8020).

3.1.2 Shallow Soil Borings and Soil Sampling

Twelve shallow soil borings (SB8 through SB19) will be performed to identify the presence of VOCs in potential source areas. The borings will be drilled to a minimum depth of the water table (anticipated depth 25 to 35 ft). The proposed soil boring locations (refer to Drawing 15268-7) have been selected based on information gathered during the preparation of the SER. Modifications or additional soil boring locations may be established, pending the results of the soil gas and surface geophysical survey (see Section 5 of the Work Plan for coordination between Warzyn and IEPA). Soil samples will be collected at 2.0 ft intervals using a 3-in. split spoon sampler. The 3 in. sampler is primarily needed for aquiring enough sample volume to accommodate splitting samples with the IEPA. However, in some instances, a 2 in. split spoon sampler may be necessary for soil composition evaluation. Field decisions will be required to determine which sampler is most approprite for each boring. A soil boring log describing the materials encountered, visual evidence of contamination, and PID screening results will be recorded by the field geologist/engineer. Soil samples representative of each unique lithology will be retained in 8-oz soil sample jars.

A sufficient quantity of representative soils will be collected from each split spoon sample to fill, at a minimum , one 8-oz. wide mouth glass jar for potential semi-volatile analysis, one 8-oz wide mouth glass jar for potential pesticide/PCB analysis, one 8-oz wide mouth glass jar for potential metals and cyanide analysis, and two 4-oz. wide mouth glass jars for potential VOCs analysis, and to partially fill one 8-oz. wide mouth glass jar for field headspace analysis. Vertical split samples will be collected from the split spoon samples by cutting the soil sample in half (parallel with the long axis of the split spoon sampler) and collecting samples, top to bottom, from each half of the split spoon device. One half of the split spoon sample will be used to fill the 4-oz. sample jars immediately after opening the split spoon. The other half will be used to log the borehole and fill the four 8-oz. soil sample jars one of which is to be used for field headspace analysis. The field headspace method is described in Section 5.2.3 of the FSP.

Surface soil samples (6 in. depth) will be collected from each shallow boring location and analyzed, for a total of 12 samples. Additional surface soil samples will be collected from an off-site location for use as a background surface soil sample. Surface soils will be analyzed for U.S. EPA CLP Target Compound List (TCL) organics, and Target Analyte List (TAL) inorganics.

Twenty-four sets of subsurface soil samples will be selected for U.S. EPA CLP analysis based on the following criteria:

- Detectable PID (11.7 eV) or FID readings greater than background;
- Visual evidence of soil contamination; and
- Distinct soil stratigraphic changes (e.g., contact at aquifer/aquitard boundary).

Selection of subsurface soil samples for analysis will be determined in the field and based on in-field screening results. During sample collection, subsurface soils will be visually examined and screened for VOCs with a PID/FID. Subsurface soil samples with obvious visual contamination and/or detectable PID/FID field headspace readings will be selected for U.S. EPA CLP analysis. If no indications of VOC contamination are observed using in-field techniques, at least one set of subsurface soil samples will be collected from each boring for U.S. EPA CLP analysis using the following three criteria:

- At a distinct soil stratigraphic change;
- At a depth of 10 ft for borings in areas with low soil gas readings; or
- At a depth of 5 ft for borings in areas with high soil gas readings.

A minimum of 24 sets of subsurface soil samples (an average of two samples per soil boring) will be analyzed for U.S. EPA CLP parameters. At least one set of soil samples will be collected from each boring for analysis.

3.1.3 Electromagnetic/Magnetometer Survey

An electromagnetic ground conductivity meter and/or a proton precession Magnetometer will be used to perform a magnetic survey to locate potential buried metallic objects such as drums and underground storage tanks. The surveys will be performed on and around the 1314 Watts Avenue and Soterion properties in the Blackhawk Acres Subdivision (refer to Drawing 15268-6). Each instrument will be calibrated prior to each use as described in the users manual. The initial survey will collect readings on a profile at 10-ft centers in the areas of interest. A coarser grid (20 ft centers) may be employed in areas surrounding these properties. During the initial survey, anomalous areas will be located on a field map and marked with pin flags in the field. Subsequently, a second survey will be performed over the anomalous areas using a tighter grid (5-ft centers). Quality assurance will be effected by the duplication of randomly selected survey measurements to examine instrument response.

3.2 MIGRATION PATHWAY ASSESSMENT

3.2.1 Hydrogeologic Investigation

3.2.1.1 Site Mapping and Evaluation of Existing Monitoring Wells - A topographic map with a scale of 1 in. to 200 ft and a contour interval of 2 ft has been prepared for use in the RI/FS (Drawing 15268-7). This base map will be used to develop other drawings and maps showing RI data.

An inventory and evaluation of existing monitoring wells will be performed to assess their condition. This will include both IEPA and Beloit Corporation wells. The following will be evaluated:

- well location
- integrity of protective casing (to include cap, lock, and identification) and the surficial seal
- integrity of well casing and screen
- water level
- total well depth
- amount of sediment in well

Wells will be developed, if necessary, to provide representative water level measurements using procedures described in the next to last paragraph of subsection 5.4.3.2 of the FSP. Functional PVC and stainless steel wells will be included in the water level measurement task and functional stainless steel wells will be included in groundwater quality sampling activities. Wells which are determined to be non-functional will be properly abandoned according to IEPA monitoring well abandonment procedures (IEPA 1990) presented in paragraph two of Section 5.4.3.2 of the FSP.

Natural gamma ray logging will be conducted in existing functional wells for use in evaluating subsurface stratigraphic conditions. At locations of a well nest, only the deep well will be natural gamma ray logged.

3.2.1.2 Deep and Intermediate Groundwater Quality Borings and Groundwater Sampling - Five deep soil borings (DB1, DB2, DB3, DB4, and W22C) and two intermediate soil borings (W23B and IB1) will be performed within the RI/FS boundaries. The deep borings will be drilled to a minimum depth of 100 ft and the intermediate borings to a minimum depth of 70 ft at the proposed locations shown on Drawing 15268-7. Total actual depth is to be determined in the field based on stratigraphy and groundwater sample screening results using a field GC. Drilling and groundwater sampling will be discontinued at a boring when one of the following situations develops:

- When groundwater screening results indicate that VOCs are not present and the 100 ft (for deep borings) or 70 ft (for intermediate borings) minimum depth has been reached, sampled, and analyzed;

- When groundwater screening results indicate that VOCs are present and the minimum depth requirement of 100 ft (deep borings) or 70 ft (intermediate borings) has been achieved, and a minimum of two consecutive clean groundwater samples have been collected below the deepest VOC detection in that boring; or

- For intermediate borings at potential source areas, and deep borings where non-aqueous phase liquids have been identified, when a confining unit greater than or equal to 3 ft in thickness is encountered, irrespective of depth. In this situation, the last groundwater screening sample will be collected at the top of the confining unit.

None of these borings will be advanced into bedrock during the Phase 1 investigation.

Dual-tube reverse circulation drilling methods (Appendix A - A2), will be used to advance the deep groundwater quality borings. Plugged, screened hollow-stem augers will be used to advance the intermediate groundwater quality borings. During drilling, groundwater samples will be collected every 10 ft, beginning at the water table. Groundwater samples will be collected through the center of the drill string or casing with a bailer, or submersible pump. Groundwater samples collected from the seven borings will be analyzed for total VOCs with an in-field gas chromatograph (GC). The GC data from the groundwater samples will be used to obtain a vertical representation of the groundwater quality beneath the site.

Upon completion of the borehole, downhole geophysical logging will be performed to obtain a natural gamma ray log. The natural gamma ray log of the borehole will be used in conjunction with cuttings for subsurface interpretation of soil stratigraphy and assessment of potential subsurface preferred contaminant migration pathways.

Monitoring wells will be installed in those borings where the GC results indicate the presence of VOCs. Screen placement will be determined based on GC results, soil stratigraphy, and gamma ray response.

One deep geotechnical boring (GB1) will be advanced to approximately 100 ft using mud rotary drilling techniques. This boring will be used to ground truth the gamma ray logging tool and to provide a representative$_{on}$ stratigraphic sequence for the site. Methods are described in Section 5.4.3.

After completion of downhole logging of the borehole, any borehole not completed with a well, will be abandoned in accordance with IEPAs guidance document - Groundwater Monitoring Network for Non-Hazardous Solid Waste Disposal Facilities (1990). These abandonment procedures are detailed in Section 5.4.5.2 of the FSP.

3.2.1.3 Monitoring Well Installation/Replacement and Soil Sampling - Initially, twelve new wells will be installed (nine on Beloit Corporation property), including six water table wells and six piezometers. One well (W24) will be installed hydraulically upgradient of the site adjacent to IEPA monitoring well G107, to assess background groundwater quality. Existing monitoring wells W3, W5, W8, and W11 will be replaced. Additional monitoring wells may be installed during the Phase 2 investigation. The location, number and type of wells to be installed will be determined from information obtained during the initial investigation (Phase 1) activities.

Monitoring wells will be installed using hollow stem auger, air rotary, or rotary wash drilling methods. Split-spoon soil samples will be collected every 2.5 ft during drilling of the boreholes. Soil samples representative of local stratigraphy will be saved in glass sample jars. Twenty of the collected soil samples will be submitted for grain size analysis by ASTM D422. Grain size distribution results will be used to confirm and enhance the visual description of soils made by the site geologist during drilling.

In addition to obtaining a stratigraphic log from split-spoon sampling, each of the monitoring well boreholes will be natural gamma ray logged. The natural gamma ray logs of the boreholes will provide a continuous record of variations in the natural radioactivity of the borehole and an indication of soil stratigraphy. This information will be used during evaluation of site geology, by indicating variations in clay content of the unconsolidated deposits at the site.

New monitoring wells will be developed by alternatively surging and purging the wells for a minimum of 30 min. using a bailer. After the surge and purge cycles are completed, the well will be pumped until ten well volumes are removed or until the pH and specific conductance of water removed from the well has stabilized.

Drill cuttings, drilling fluids (mud and water) and extraneous soils derived from split spoon sampling will be evaluated in the open atmosphere during drilling with an 11.7 eV PID in the field. If PID readings are equal to or exceed 5 ppm then these cuttings, soils and fluids, will be placed in 55 gal drums, labeled and retained on-site on Beloit Corporation property. If PID readings are less than 5 ppm then these cuttings, mud and extraneous soils will be disposed of on-site on Beloit Corporation property. TCLP analysis will be performed on composites of the containerized cuttings, mud and soils (PID $\geq$ 5 ppm) in order to determine an appropriate disposal option. Disposal of water used as a drilling fluid is discussed below.

Development, drilling and purge waters will be separated based on the presence or absence of a non-aqueous phase liquid. Those development, drilling and purge waters containing a non-aqueous phase liquid will be considered a hazardous waste, drummed, stored separately and disposed of at an appropriate hazardous waste facility. Development, drilling and purge waters not containing a non-aqueous phase liquid will be disposed in the sanitary sewer along with decontamination water for treatment at the local POTW, assuming approval can be obtained from the POTW. If approval can not be obtained, these materials will also be drummed, stored in a secure area on Beloit Corporation property and disposed of at the time of the final remedy.

3.2.1.4 Existing Monitoring Well and Standpipe Abandonment - Four existing monitoring wells (W3, W5, W8, and W11) and seven standpipes (SP1, SP3, and SP8 through SP12) are to be abandoned during Phase 1 of the RI. They will be abandoned in accordance with IEPA's guidance document - Groundwater Monitoring Network for Non-Hazardous Solid Waste Disposal Facility (1990). These abandonment procedures are detailed in Section 5.4.3.2 of the FSP.

3.2.1.5 Hydraulic Conductivity Tests - Hydraulic conductivity tests will be performed in the new wells installed during the RI. Single well baildown test methods will be used and data analyzed using the Bouwer and Rice (1976) method. Additional baildown hydraulic conductivity testing may be performed in existing functional monitoring wells.

3.2.1.6 Water Level Measurements - A minimum of four rounds of water level measurements will be conducted during the RI. Water level measurements will be obtained with an electric water level indicator. One round of water levels will be measured following completion of the monitoring well installation. Water levels will be measured concurrent with groundwater sampling events. Bimonthly measurements will be conducted thereafter for a period of eight months, if necessary from the initial measurement event. The water level measuring device will be deconed between wells.

3.2.2 Surface Water and Sediment Investigation

Surface water drainage patterns will be identified and located based on field observations. These drainage patterns and runoff characteristics will be evaluated for the potential of erosional transport of contaminated surface soils off-site. It is anticipated that three staff gauges will be installed at the site (along the Rock River). Specific staff gauge locations will be determined based on field observations of optimum locations. Existing staff gauges at the site will be evaluated for integrity and monitoring use. Surface water level measurements will be collected concurrently with groundwater level measurements.

3.2.3 Surface Soil Assessment

The potential for exposure to contaminants via the surface soils pathway will be evaluated based on samples collected for chemical analysis from the 12 shallow borings (Drawing 15268-7).

3.2.4 Air Assessment

Wind patterns will be evaluated, based on available meteorologic information, to determine possible air pathway exposure scenarios. Air quality data collected from health and safety monitoring during site investigation activities will be used to assess the potential for release of contaminants into the air. Samples will not be collected for laboratory analysis.

3.2.5 Ecological Assessment

A preliminary ecological assessment of the site and adjacent areas will be performed. Ecological resources will be investigated by a site walkover by a field ecologist to note types and approximate sizes of plant communities of the site and adjacent undeveloped areas. Potential of the site and surrounding areas as habitat for vertebrate species will be assessed. Wetland areas will be classified according to the U.S. Fish and Wildlife Service (U.S. F&WS) system (Cowardin, et. al., 1979) and their sizes estimated by use of the Federal Manual for Identifying and Delineating Jurisdictional Wetlands (Federal Interagency Committee for Wetland Delineation, 1989).

Site observation will be supplemented by information available from site and federal agencies concerning the following ecological resources:

- Hydric soils (U.S. Soil Conservation Service)
- Rock River fishery (Illinois Department of Conservation)
- Threatened or endangered species (Illinois Department of Conservation and U.S. F&WS)
- Designated area wetlands (U.S. F&WS National Wetland Inventory Map)

A qualitative preliminary ecological assessment will be developed to evaluate potential contamination by site chemicals detected by RI sampling activities. Potential effects of these contaminants will be discussed for species likely to inhabit the site or areas affected by site contaminants. If a preliminary evaluation suggests a serious impact by the site on area natural resources, further investigation may be required (U.S. EPA Region V, Tasks 3-6, undated, unpublished memorandum).

3.3 CONTAMINANT CHARACTERIZATION

3.3.1 Groundwater Sampling

Groundwater samples will be collected from the following locations during the Round 1 sampling event:

- The 12 new groundwater monitoring wells installed during well installation activities of the RI (Refer to Drawing 15268-7);

- The four replacement wells W3, W5, W8, and W11;

- Existing monitoring wells W12 through W18 (7 wells), and G103S and D, G104, G107, G108S and D, G109, and G110 (8 wells); and

- Private wells as specified in the private well sampling document.

Groundwater sampling will be used to determine the chemical characteristics of groundwater contamination. Samples will be analyzed for the following chemical and physical parameters:

- U.S. EPA CLP TCL organics;
- U.S. EPA CLP TAL inorganics;
- Sulfate;
- Chloride;
- Alkalinity;
- Total phenolics;
- Nitrate-Nitrite nitrogen;
- Ammonia nitrogen;
- Total dissolved solids;
- Field pH; and
- Field specific conductance.

Inorganic analyses will be performed on filtered samples collected during Round 1, consistent with U.S. EPA Region V QA Directives. Round 2 samples will be analyzed for contaminants of concern based on the results of Round 1 sampling. Technical Memorandum No. 1 will describe analytical results and address data gaps requiring additional sampling and analysis to be performed during the Phase 2 investigation.

Appropriate sample containers, preservation, and packaging are described in Table 1-2 of the QAPP.

3.4 QUALITY ASSURANCE SAMPLING

Quality control samples will be collected during sampling activities associated with the chemical characterization of groundwater, surface soils, and subsurface soils. Quality control samples will consist of sample duplicates, field blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples, as described below.

3.4.1 Field Blanks (FB)

For soil samples, no field blanks will be collected due to the unavailability of suitable blank material. For water samples, one field blank will be prepared for each container type and size. Field blanks will be prepared according to the following schedule for each sampling activity:

- One field blank for every 10 or fewer samples of water collected; and

- For each sample period, a minimum of one blank for each group of parameters per sample matrix.

The field blank samples will be prepared using deionized water stored in polyethylene containers. For monitoring well samples, the water will be routed through the sampling device before transfer to the container. Soil gas field blanks will be prepared by collecting a sample of ambient air after decontamination of the sampling system.

3.4.2 Trip Blanks (TB)

A trip blank for volatile organic analysis (VOA) will be included in each sample shipment containing water matrix samples for VOA. The trip blank will consist of two 40-ml VOA vials filled with deionized water with a Milli-Q cleanup. It will be prepared in the office or laboratory, transported to the field and shipped with the other samples to the designated laboratory without being opened. It will be packaged using standard procedures as for other sample bottles.

3.4.3 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

For water samples, one sample per group of 20 or fewer samples collected for VOA and extractable organics analysis during each sampling activity will be selected for matrix spike/matrix spike duplicate (MS/MSD) analysis. For Base/Neutral Acids (BNAs) and Pesticides/PCBs, double the normal sample volume will be collected (i.e., four, 1-L bottles). For VOCs, triple the normal sample volume will be collected (i.e., six, 40 ml VOA vials).

3.4.4 Sample Duplicates

One duplicate sample will be collected for each increment of 10 or fewer samples collected for each matrix during each sampling period. A duplicate sample will consist of a sample obtained from the same sampling device as the original sample.

Due to the sample volume requirements and the soil volume obtained using split-spoon sampling methods, sample duplicates for subsurface soil samples may not be possible. Sample duplicates for subsurface soils will only be performed if the split-spoon sample volume is adequate during subsurface soil sampling procedures.

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4

SAMPLE DESIGNATION

A sample numbering system will be used to identify each investigative and quality control sample. Each sample identifier will include the project identifier code, sample type and location code, and a sampling event code. The sampler will maintain a log book containing the sample identification listings.

4.1 PROJECT IDENTIFIER CODE

A two-letter designation will be implemented to identify the sampling site. The project identifier will be "BC" to signify the Beloit Corporation Rockton Facility site investigation.

4.2 SAMPLE TYPE AND LOCATION CODE

Each sample collected will be identified by a two-letter code corresponding to the sample type. Sample type codes to be used for the various samples collected are:

- GD - groundwater sample from deep groundwater quality boring
- GI - groundwater sample from intermediate groundwater quality boring
- PW - groundwater from a private residential well or municipal well
- GW - groundwater sample from monitoring well
- SS - split spoon or subsurface soil sample
- SU - surface soil sample
- FB - field blank
- TB - trip blank

Other letter designators may be added for sample activities of later subtasks.

The location code will follow the sample type code. The location code consists of a two- to five-digit numeric or alpha-numeric code indicating the sample location. Field blanks and trip blanks will use a consecutive numbering system starting at 01, assigned in the field.

4.3 SAMPLING ROUND CODE/DUPLICATE CODE

A two-digit numerical code will be used to designate additional location information. For soil samples, the sampling code will represent the depth of the

sample in feet below the ground surface. Duplicate samples will be designated by the sampling code preceded by a 9.

Matrix spike and matrix spike duplicate samples are collected as additional sample volume at selected locations. Although identified as such on chain-of-custody records, specific sampling codes will not be provided for matrix spikes or matrix spike duplicates (MS/MSD) samples.

4.4 EXAMPLES OF SAMPLE NUMBERS

Examples of sample number codes are as follows:

- BC-SSMW03A-25 = Beloit Corporation, split-spoon soil sample from monitoring well MW03A at a depth of 25 ft.
- BC-GWMW03A-01 = Beloit Corporation, groundwater sample from monitoring well MW03A, first sampling round.
- BC-GWMW03A-91 = Beloit Corporation, duplicate groundwater sample from monitoring well MW03A, first sampling round.
- BC-SUSB10-00 = Beloit Corporation, surface soil sample from location of shallow soil boring number 10, sample collected at the ground surface.
- BC-GDDB01-30 = Beloit Corporation, groundwater sample collected while drilling deep boring DB1 at 30 ft from the ground surface.
- BC-GIIB01-50 = Beloit Corporation, groundwater sample collected while drilling intermediate boring IB1 at 50 ft from ground surface.

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5

GENERAL SAMPLING EQUIPMENT AND PROCEDURES

5.1 SOIL GAS SURVEY

5.1.1 Objective

The objective of the soil gas survey is to locate potential sources of VOCs at the site and identify potential near surface contaminant sources or soil gas associated with contamination at depth.

5.1.2 Personnel and Responsibilities

A team of two individuals will perform the soil gas survey. A technician will drive the probe into the ground and extract a sample into a glass vial. A site chemist will analyze the collected samples by operating the in-field gas chromatograph (GC).

5.1.3 Methods

Soil gas sample locations will be approximated in the field by using a compass to measure a bearing between soil gas points and measuring the distance between the reference points (trace). Permanent site features may be used as reference points. A detailed log will be maintained identifying reference points and associated bearing and trace. In addition, each soil gas location will be plotted on a field map as each location is identified in the field during sample acquisition activities. Additional soil gas samples may be collected at unspecified locations based on field observations and/or GC results.

Soil gas samples will be collected by:

- Driving a 1/2-in. or 5/8-in, which ever is appropriate, diameter solid rod to a depth of approximately 3 to 4 ft below ground surface to create a pilot hole.
- The rod is replaced with an AMS Retract-a-Tip gas vapor probe. This probe will be driven down the pilot hole using a slam bar.
- The screen casing is pulled back to expose the screen.

- The teflon vapor tube, which is exposed at the surface, is slipped over the sample tip of the Thermo Environmental (TE) 580B PID.

- The sample vessel (Tedlar bag) is connected to the exhaust (sample) port of the TE 580B PID with teflon tubing.

- Both ports on the Tedlar bag are opened.

- The pump (TE 580B PID) is switched on and allowed to draw in-situ soil gas through the system for a minimum of 2 minutes.

- After 2 minutes the sample port on the Tedlar bag will be closed and the bag allowed to fill.

- With the pump still running the input port of the Tedlar bag will be closed, then the pump will be switched off.

- The Tedlar bag will be disconnected from the pump, shielded from sunlight and placed on ice in a cooler.

- The pump (TE 580B PID) will be disconnected from the AMS soil gas probe teflon vapor tube.

- The screen casing will be closed (screen covered) and then the probe will be removed from the pilot hole.

- Prior to the next sampling the AMS gas vapor probe will be decontaminated as described below, then the probe, pump and teflon tubing will be connected and purged with atmospheric air by running the pump for a minimum of 1 minute. Tedlar bags that have been used may be re-used if:

  - G.C. results show no detectable VOCs, and
  - The Tedlar bag has been decontaminated with high purity helium gas.

  The external surface of the gas probe and the pilot rod will be decontaminated by wiping both with paper towels moistened with TSP or liquinox cleaning solution followed by paper towels moistened with rinse water.

- Move to next sample location.

The sample will be delivered to the in-field GC for analysis of VOCs. Results of the soil gas analyses will be plotted in the field on the aforementioned site map.

Used paper towels will be disposed in trash bags and placed with other investigative wastes. Field blanks will be collected at a frequency of one blank per ten samples, consistent with sampling of other media.

5.2 SHALLOW SOIL BORINGS AND SOIL SAMPLING

5.2.1 Objective

The objective of this activity is to chemically characterize site surface and subsurface soils, characterize potential source areas, and supplement information obtained during the soil gas survey.

5.2.2 Personnel and Responsibilities

A drill crew of two individuals will perform the shallow soil borings. A geologist will supervise their efforts, record soil boring logs, collect soil samples, and function as the Site Safety Officer.

5.2.3 Methods

Twelve shallow soil borings will be drilled to a minimum depth of 25 to 35 ft (water table) using 4 1/4-in. I.D. or 6 1/4-in. I.D. hollow stem augers. Soil samples will be collected at 2-ft intervals with a 3-in. split spoon sampler, if possible. If the 3-in. split spoon sampler will not retain the sample, a 2-in. split spoon sampler will be employed. The samples will then be visually classified in the field by a geologist, placed into appropriate sampling jars as specified in Section 3.1.2 of this Field Sampling Plan, and screened using the field headspace method. A log of the soil boring, identifying materials encountered, will be maintained by the site geologist/engineer. Soil samples will be visually classified in accordance with the Unified Soil Classification System (USCS).

Twelve surface soil samples will be collected, one at each boring location. A background surface soil sample will be collected at the location of the background monitoring wells. Surface and selected subsurface soils collected during shallow soil borings will be analyzed for U.S. EPA CLP TCL and TAL parameters.

As indicated in Section 3.1.2, subsurface soil samples will be selected for analysis based on elevated PID/FID readings, visual observations of contamination, and changes in soil stratigraphy. Selection of subsurface soil samples for analysis will be determined in the field and based on field observations at the time of sampling.

Subsurface soil samples will be evaluated for VOCs using the field headspace method. An 8 oz. glass sample jar is half filled with representative soil collected

from a 3-in. split spoon sample. A piece of aluminum foil, sufficient in size to cover the opening and threads of the sample jar, is then placed over the jar opening. The teflon lid of the jar is then secured snuggly over the aluminum foil. The contents of the jar are to be heated for 30 minutes. After 30 minutes the lid is removed (keep the aluminum foil cover in place) and an 11.7 eV PID (Appendix A - A4) or FID (Appendix A - A6) probe is forced through the aluminum foil seal to collect a headspace sample from the jar. A decision to analyze samples from each location will be made after all samples at that location have been collected, screened, and visually described.

A minimum of 24 sets of subsurface soil samples with at least one collected from each boring will be selected for U.S. EPA CLP analysis. Information regarding QA/QC sample numbers and frequency are contained in Table 1-1 of this QAPP. Appropriate sample containers, preservation and packaging information are described in Table 1-2 of this QAPP and Section 3.1.2 of the FSP.

A temporary decontamination facility will be constructed on-site on Beloit Corporation property. Decontamination will include steam cleaning or high pressure hot water washing the drilling equipment and tools between boreholes, and detergent washing and water rinsing the split spoon samplers after each collected sample. Decontamination procedures are detailed in the Health and Safety Plan (Volume II of the Planning Documents).

Shallow soil borings will be abandoned in accordance with IEPAs Monitor Well Plugging and Abandonment Guidance Document (IEPA 1990) (Section 5.4.3.2 of the FSP). The disposition of excess borehole cuttings generated during drilling is discussed in Section 3.2.1.3 of the FSP.

5.3 ELECTROMAGNETIC(EM)/MAGNETOMETER SURVEY

5.3.1 Objective

The objective of the EM/magnetometer survey is to determine whether buried metal containers or underground metal tanks exist in the area of the 1314 Watts Avenue and the Soterion property in the Blackhawk Acres Subdivision.

5.3.2 Personnel and Responsibilities

A team of two individuals, including a geologist/geophysicist and an assistant will be responsible for performing these surveys. The geologist/geophysicist will operate the instrument and the assistant will help lay out grid lines, flag locations and record activities.

5.3.3 Methods

An electromagnetic conductivity meter (see Appendix A - A1) and/or a proton precession magnetometer (see Appendix A - A3) will be used to perform these surveys. The team will set up the geophysical survey grid with 10 ft spacings across each area to be surveyed. The geologist/geophysicist will traverse the area while operating the EM or magnetometer. Areas of anomalous readings will be plotted in the field with pin flags and on a site map. A base location, away from the potentially contaminated area, will be set up as a calibration verification point for the geophysical instrument. The instrument will be checked at this location before, after, and during the investigation. Instrument readings and drift at the base location will be recorded.

Intrusive activities will not be performed during these surface geophysical surveys, therefore decontamination and waste disposal procedures are not anticipated.

5.4 HYDROGEOLOGIC INVESTIGATION

5.4.1 Objective

The objective of the hydrogeologic investigation is to obtain information for characterization of the hydrogeologic setting, determine groundwater flow direction, assess the potential for preferential groundwater flow, and identify the physical characteristics of the geologic media at the site.

5.4.2 Personnel and Responsibilities

Deep and intermediate groundwater quality borings, monitoring well borings, and a geotechnical boring will be performed and installed by a drill crew of two individuals. Drilling operations will be supervised by a geologist/engineer, who will collect groundwater and soil samples, obtain natural gamma logs, and function as the Site Safety Officer. Water levels will be measured by a technician. Hydraulic conductivity tests will be performed by a geologist or field technician.

5.4.3 Methods

5.4.3.1 Deep Borings and Groundwater Sampling - Five deep (DB1-DB4 and W22C) and two intermediate (IB1 and W23B) groundwater quality borings, and one deep geotechnical boring (GB1) will be performed at locations inside the RI/FS site boundaries. The primary objective of the deep and intermediate borings is to assess groundwater quality with depth in the aquifer. The groundwater analytical data will be used exclusively as screening data to identify target VOCs in groundwater at different zones in the aquifer. The primary objective of the geotechnical boring is to provide a borehole for ground-truthing the natural gamma ray logger and for obtaining characteristic soil descriptions for the site. The proposed locations for the borings are shown on Drawing 15268-7.

Dual-tube reverse circulation drilling methods (Appendix A - A2) and plugged, screened hollow stem auger drilling methods will be used to advance the deep and intermediate boreholes, respectively. Representative groundwater samples will be collected during drilling with a stainless steel bailer or submersible pump with inflatable packer at selected zones in the aquifer. Groundwater samples will be collected every 10 ft beginning at the water table. If groundwater does not recover, or is not expected to recover within 30 minutes due to the presence of a fine grained, low permeability zone, no sample will be collected from that zone.

Groundwater samples will be collected through the center of the drill string or casing. A bailer, B-K pump, or submersible pump with an inflatable packer will be used to purge three times the standing water volume in the drill rod or auger before a sample is collected. If a packer is used, then three times the volume of the packed-off zone will be purged. Groundwater samples collected during each boring will be screened for VOCs with an in-field GC using the methods specified in the QAPP. The deep borings will be drilled to a minimum depth of 100 ft and the intermediate borings to a minimum depth of 70 ft at the proposed locations shown on Drawing 15268-7. Total actual depth is to be determined in the field based on stratigraphy and groundwater sample screening results. Drilling and groundwater sampling will be discontinued at a boring when one of the following situations develops:

- When groundwater screening results indicate that VOCs are not present and the 100 ft (for deep borings) or 70 ft (for intermediate borings) minimum depth has been reached, sampled, and analyzed;

- When groundwater screening results indicate that VOCs are present and the minimum depth requirement of 100 ft (deep borings) or 70 ft (intermediate borings) has been achieved, and a minimum of two consecutive clean groundwater samples have been collected below the deepest VOC detection in that boring; or

- For intermediate borings at potential source areas, and deep borings where non-aqueous phase has been identified, when a confining unit greater than or equal to 3 ft in thickness is encountered, irrespective of depth. In this situation, the last groundwater screening sample will be collected at the top of the confining layer.

GC results of groundwater samples will be used to assess the vertical extent of VOC contamination at each boring location, and will be used to determine installation depths for monitoring wells that may be installed during this pphase of work and placement of monitoring wells that may be installed during a Phase 2 investigation.

Due to the drilling methods (dual tube and plugged hollow stem auger), split-spoon samples will not be obtained during drilling. A boring log based on discharged drill cuttings will be kept by the geologist/engineer supervising drilling activities. To supplement subsurface information, a natural gamma ray log will be obtained through the drill casing upon completion of the borehole. A Mount Sopris Model 1000-C borehole logger (Appendix A - A5) and natural gamma ray logging tool will be used to obtain the downhole geophysical log.

Geotechnical boring GB1 will be advanced to a minimum depth of 100 ft using a pure bentonite mud and rotary wash drilling methods (minimum borehole size of 4-in.). Standard penetration test (ASTM D1586) soil samples will be collected every 5 ft from 2 ft to 30 ft, every 2 ft from 30 ft to 80 ft, and every 5 ft from 80 ft to 100 ft. Representative soils will be placed in 8 oz sample jars and retained. A detailed soil borehole log will be maintained by the field geologist/engineer supervising drilling activities.

When the mud rotary drilling technique has reached a depth of 100 ft, in geotechnical boring GB1, and is able to maintain an open hole to this depth, the drill string can be removed and the borehole logged with the natural gamma ray logging device. The natural gamma ray detecting probe is lowered to the bottom of the borehole and is then advanced to the surface at a logging rate of slightly less than 1 ft/sec. This gamma ray trace will be the gamma ray tool ground-truth log for the site.

Deep and intermediate groundwater quality boreholes, not instrumented with a monitoring well, and the geotechnical boring will be abandoned in accordance with IEPAs Monitor Well Plugging and Abandonment Guidance document (IEPA, 1990). Drill cuttings and fluid will be screened with a PID during drilling operations. The disposition of excess borehole cuttings and drilling fluids generated during drilling is discussed in Section 3.2.1.3 of the FSP.

To minimize potential inadvertent contamination by drilling equipment and/or inter-borehole contamination, steam cleaning will be used between boring locations (except between borings at a well nest). The drill rig and drilling tools will be steamed cleaned before mobilization onto the site, and will be steam cleaned between each boring (except between borings at a well nest). The disposition of decon water is discussed in Section 6 of the FSP.

5.4.3.2 Monitoring Well Installation/Abandonment - Twelve groundwater monitoring wells (eight new and four replacements) are proposed including nine wells at four locations on Beloit Corporation property, one well at one location upgradient of the site, and two wells at one location downgradient of the site.

Four replacement wells are proposed for existing monitoring wells W3,W5, W8, and W11. Proposed locations are shown on Drawing 15268-7.

Existing non-functional wells and standpipes will be abandoned in accordance with IEPA monitoring well abandonment procedures (IEPA, 1990). Surface casing will be removed a minimum of 12 inches below ground surface. A neat cement slurry (5 gal water per 94 lb bag cement) will be mixed and slowly pumped into the well casing through a tremie pipe. The tremie pipe will be placed at the bottom of the well and then slowly withdrawn as the cement slurry is pumped into the well casing. Pumping will continue until the cement slurry flows out the top of the well casing. The 12 inch deep hole will then be filled with natural soils.

Water table well borings (anticipated depth: 25 to 35 ft) will be drilled using 4 1/4-in. or 6 1/4-in. I.D. hollow stem augers. If sand blow-in through the augers occurs during drilling, rotary wash methods using clear water will be used to remove material from the interior of the augers. Split spoon soil samples will be collected at 2.5 ft-intervals to the bottom of the boring (only the deep boring at a well nest will be split spoon sampled) where feasible. If sand blow-in is severe, no sampling will be conducted and natural gamma ray logging will be substituted.

Intermediate depth (anticipated depth: 60 to 70 ft) piezometer borings will be drilled using 4 1/4-in. I.D. or 6 1/4-in. I.D. hollow stem augers or rotary wash drilling methods. Split spoon soil sampling intervals (2.5 ft) will be similar to water table well borings.

Based on past subsurface drilling at the site, augering methods may not be possible below those depths where extensive sand blow-in occurs. Due to limitations in augering methods, rotary drilling methods using casing will be used when appropriate. Alternative methods include use of dual-tube reverse air rotary or clear water wash rotary. In the event clear water does not remove cuttings or the borehole is unstable, a pure bentonite mud wash may be necessary to complete the boreholes. During drilling operations, attempts will be made to minimize the amount and use of bentonite drilling mud. Bentonite with chemical additives will not be used at the site.

Water used during drilling activities will be obtained from the City of Rockton municipal water supply. As a quality assurance procedure, a sample of the water used for drilling and other activities at the site will be collected and analyzed for VOCs (CLP SOW OLMO2.0) during initial drilling activities.

A log of the monitoring well boring will be recorded by the site geologist. Soil samples will be visually classified in accordance with the Unified Soil

Classification System (USCS). Twenty (20) soil samples will be analyzed by a soils laboratory for grain size. Samples will be selected following completion of the drilling program to obtain a representative selection of the various soil types encountered during the investigation. The purpose of these analyses is to confirm field identification of soils and to assist in assessing subsurface soils as a contaminant migration pathway. Soil samples will be field screened in the open atmosphere with a PID to assess potential hazards to the field personnel. The disposition of excess soils is discussed in Section 3.2.1.3$_2$ of the FSP.

Final soil boring logs will be prepared based on field observations, natural gamma ray log response, and soil testing and laboratory sample classification. Soil samples will be retained for Beloit Corporation until termination of the RI/FS. Procedures for sample handling are discussed in the QAPP.

Monitoring wells will be constructed as shown in Drawings 15268-A7 and 15268-A8 in the Work Plan. Water table wells will be constructed with 10-ft long, No. 10 slot (0.010-in.) continuous wrap 2-in. I.D. stainless steel screens placed so the screen is likely to intercept the water table. Piezometers will be constructed with 5-ft long No. 10 slot (0.010-in.) continuous wrap 2-in. I.D. stainless steel screens and a 10 ft section of stainless steel riser. The riser above the 10 ft stainless steel riser section will consist of 2-in. I.D. schedule 40 PVC pipe for wells less than 80 ft deep and schedule 80 PVC pipe for wells greater than 80 ft deep.

The annular space between the well and the edge of the borehole will be backfilled with clean silica sand to approximately 2 ft above the top of the well screen. A minimum 3-ft hydrated bentonite pellet seal will be placed directly above the sand pack. At water table wells, the annulus will be backfilled with granular bentonite. At piezometers, a fine silica sand or bentonite pellets will be placed above the sand filter pack and bentonite slurry placed above the fine silica sand or bentonite pellets, using a tremie pipe. A granular bentonite surface seal will be installed at each well. A locking steel protective casing will be installed at the surface of each well.

The following partial abandonment procedures are to be followed when installing monitoring wells in water quality borings.

If the backfill interval is greater than 50 ft then the water quality borehole is to be abandoned following IEPA (1990) monitoring well abandonment guidelines (see paragraph 2, Section 5.4.3.2). A monitoring well will then be installed in a new borehole that will be drilled approximately 5 ft from the abandoned water quality borehole.

If the backfill interval is less than 50 ft then the following guidelines will be used to install monitoring wells in water quality borings:

- A tremie pipe will be used to install approximately 3/8-in. diameter bentonite pellets or chips (interlayered with a clean silica sand in permeable zones) in water quality borings that are less than 250 ft deep and with a standing water depth of less than 150 ft.

- 6.25-in. I.D. hollow-stem augers will be used to advance intermediate water quality boreholes that may require partial abandonment.

Bentonite material must be installed at the base (minimum 2 ft thick) and top (minimum 2 ft thick) of the backfill interval and must also extend through impermeable zones. In the permeable zones a minimum of 2 ft of bentonite material will be installed for every 4 ft of silica sand. If the backfill interval is 10 ft or less then bentonite will be the only backfill material utilized.

New monitoring wells will be developed by alternatively surging and purging the wells for a minimum of 30 min. using a bailer. After the surge and purge cycles are completed, the well will be pumped until 10 well volumes are removed or until the pH and specific conductance of water from the well has stabilized. The disposition of purge water is discussed in Section 3.2.1.3 of the FSP.

To minimize potential inadvertent contamination by drilling and sampling equipment, and/or inter-borehole contamination, steam cleaning will be used between borings (except between borings at a well nest). The drill rig and drilling tools will be steamed cleaned before mobilization onto the site, and will be steam cleaned between each boring. The split spoon sampler will be cleaned in a liquinox or non-phosphate detergent solution between monitoring well boreholes except at well nests. The disposition of decon water is discussed in detail in Section 6 of the FSP. PVC and stainless steel well pipe and stainless steel screen will be steam cleaned or washed with a high pressure hot water washer prior to installation.

5.4.3.3 In Situ Hydraulic Conductivity Tests - Hydraulic conductivity will be measured by baildown testing in water table wells and/or by air pressure methods in deeper piezometers. The Bouwer and Rice Method (1976) will be used to analyze the baildown test data. The methods to be used are as follows:

- Measure water level with a tape and sounding device or electronic water level indicator.

- 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 pressure 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 until water level reading is stable.

- After a stabilized water level reading is obtained from the pressure transducer, the well (piezometers only) is pressurized with sufficient air pressure to displace several ft of water (0.4 PSI/ft of water).

- Air pressure is maintained until the water level reading from the transducer is constant (for piezometers only).

- The air pressure is then instantaneously released while running the pressure transducer recorder in the log sampling mode (for piezometers only).

- At water table wells a single bailer of groundwater is removed to reduce the water level, while recording the pressure transducer readings with the data logger.

- The test results may be immediately printed out to obtain a hard copy.

- Data are transferred from the data logger to a computer for analysis.

5.4.3.4 Water Level Measurements - Water levels in existing and new monitoring wells will be measured. An electric water level indicator will be lowered into the monitoring wells until the water level is reached. Depths will be recorded to the nearest 0.01 ft.

5.5 SURFACE WATER AND SEDIMENT INVESTIGATION

5.5.1 Objective

The objective of this activity is to determine surface water drainage patterns at and around the site. The staff gauge measurements will be used in the general assessment of site water levels and the affects of surface water/groundwater flow interactions.

5.5.2 Personnel and Responsibilities

A survey crew or field technician will install three staff gauges in the Rock River. A technician will measure water levels. A scientist or engineer will identify and map drainage patterns.

5.5.3 Methods

Staff gauges will be metal rods with a scale in hundreds of feet. The gauge will be installed at least one foot into the river sediments, or until stable, so the scale extends at least one foot above and below the water line. The reference line on each gauge will be surveyed for location and elevation. Surface water level measurements will be obtained at the staff gauge locations concurrently with groundwater level measurements. Measurements will be made by reading the water level mark on the staff gauge and subtracting the measurement from the staff gauge reference elevation.

A scientist or engineer will visually locate, identify, and map drainage patterns at the site. The drainage patterns will be plotted on a drawing developed from the site base map. The data will be used to assess the potential for erosional transport of contaminants off-site.

5.6 SURFACE SOILS ASSESSMENT

5.6.1 Objective

The objective of this activity is to assess the potential for direct contact exposure to contaminated surface soils.

5.6.2 Personnel and Responsibilities

A geologist will collect surface soil samples at the twelve shallow boring locations and the background location. The background location is anticipated to coincide with the background monitoring well location. A scientist or engineer will identify the erosion potential of surface soils for assessing the surface soil contamination pathway.

5.6.3 Methods

An inspection of the site will be conducted. Surface soils will be observed and monitored with a PID during sampling activities. Based on this site inspection and analytical data collected during Source Characterization and Migration Pathway Assessment activities, the need for additional surface soil sampling will be determined.

5.7 AIR ASSESSMENT

5.7.1 Objective

The objective of this activity is to assess the potential for release of contaminants to air.

5.7.2 Personnel and Responsibilities

The geologist/engineer on each drilling rig will monitor air quality using a PID during soil borings and monitoring well installation.

5.7.3 Methods

Air quality will be monitored using a PID. Monitoring results above background will be recorded in each individuals field logbook. Monitoring during intrusive activities is assumed to approximate "worst case conditions", when potentially contaminated soil or waste is exposed to the atmosphere.

5.8 GROUNDWATER SAMPLING

5.8.1 Objectives

The objective of groundwater sampling is to determine the nature, magnitude and extent of groundwater contamination.

5.8.2 Personnel and Responsibilities

A crew of two technicians will collect samples.

5.8.3 Methods

Groundwater sampling will proceed from wells expected to have the lowest contaminant concentrations (based upon observations during drilling and existing groundwater quality data), to the wells suspected of having the highest contaminant concentrations. Each sampled well will be purged immediately prior to sampling using a submersible sampling pump at a high pumping rate. A packer will be used in all wells screened below the water table, and inflated within the stainless steel riser so as to seal off the PVC riser.

The volume of water removed from the well will be measured so that a minimum of three volumes are removed. Specific conductance, temperature and pH will also be monitored for stabilization during purging. After well purging has been completed, the pumping rate will be decreased to approximately 100 ml/min and the VOC sample collected directly from the pump discharge. Sample bottles, filtering, and preservation will be as specified in Table 1-2 of the QAPP.

The disposition of purged water is discussed in Section 3.**2.1.3** of the FSP.

The sampling equipment and water level measurement tape will be cleaned between wells with a Liquinox or another non-phosphate detergent solution and rinsed with deionized water. Decontamination of the pump and tubing will consist of pumping the decon solution into the wash tanks for several minutes. The pump will then be moved into the rinse tank and pumping will resume with rinse water.

Groundwater sample blanks will be collected by pumping deionized water through the sampling pump into the sample bottles. Matrix spike/matrix spike duplicate samples will be collected using the same sampling device. Duplicate samples for volatile organics analysis will be obtained by alternately filling VOC vials from the pump. The remainder of the sample will be split between the principal sample and the duplicate sample.

[mad-603-15g]

6

DECONTAMINATION PROCEDURES

Procedures to be followed to decontaminate equipment and personnel are described in the Site Health and Safety Plan. The procedures are summarized below.

Site Personnel Decontamination Procedure:

- Equipment drop;
- Wash outer booties and rinse;
- Dispose of outer latex booties;
- Wash boots in Liquinox bootwash;
- Clean outer gloves in Liquinox wash solution (discard if too soiled to clean thoroughly);
- Dispose of polycoated Tyvek suits;
- Dispose of surgical gloves;
- Wash hands in hand wash;
- Wash face and neck in face wash; and
- Clean and sanitize face mask.

Site personnel will perform the above mentioned decontamination procedure prior to leaving the site. Discarded clothing and other articles will be collected in double-lined, heavy duty garbage bags.

Equipment and vehicle decontamination procedure:

- Decontamination will be performed prior to site entry;
- Decontamination will be performed on site;
- Gross contamination will be removed with a brush and Liquinox solution; and
- Steam cleaning or hot water high pressure washing will follow.

The drilling equipment will be steam cleaned or high pressure hot water washed at the temporary decon facility and between boring locations prior to exiting the site. Another area, located away from the equipment decontamination area, will be designated as a site personnel decontamination area.

Decontamination will include steam cleaning or hot water high pressure washing the drill rig, drilling equipment, and tools between boreholes, and detergent washing and water rinsing the split spoon samplers after each collected sample. The drill rig and equipment may also require scrubbing of accessible parts with a detergent/water solution. Well materials will also be steam cleaned or hot water high pressure washed and wrapped in plastic until installed. The bailer cable, trowels, spatulas, stainless steel bucket and water level measurement tape will be cleaned with Liquinox or another non-phosphate detergent solution, and rinsed with deionized water.

All decontamination water will be captured by the decontamination pad, pumped into appropriate storage containers and then routed to the local POTW for treatment, assuming that approval can be obtained from the POTW. If approval can not be obtained, these materials will also be contained, stored in a secure area on Beloit Corporation property and disposed of at the time of the final remedy.

[mad-603-15h]

7

SAMPLE HANDLING AND ANALYSIS

7.1 PARAMETERS

Samples collected for chemical analysis will be analyzed by a laboratory approved by the U.S. EPA Region V Contract Program Management Section (CPMS). Chemical parameters for which groundwater will be analyzed are:

- U.S. EPA CLP TCL organics;
- U.S. EPA CLP TAL inorganics;
- Field pH;
- Field-specific conductance;
- Chloride;
- Sulfate;
- Alkalinity;
- Total phenolics;
- Nitrate-Nitrite nitrogen.
- Ammonia nitrogen; and
- Total dissolved solids.

Measurement of pH, specific conductance, and temperature will be performed in the field. The groundwater samples to be analyzed for U.S. EPA CLP TAL metals will be field filtered with 0.45 micron filters, as soon as possible after collection. Shallow soil boring samples (soil) will be analyzed for U.S. EPA TCL and TAL parameters. Select samples from monitoring well installation borings will be analyzed for grain size by a soils laboratory.

7.2 SAMPLE PRESERVATION

Samples will be collected and preserved in a manner appropriate for the analyses they receive (see Table 1-2 of the QAPP). The portion of groundwater samples requiring field filtering prior to analysis (see Table 1-1 of the QAPP) will be filtered using a pressure filtration device, through a 0.45 micron filter, as soon as possible after collection. Filtered portions of the samples will be preserved, as appropriate, immediately after filtration. Sample fractions will be preserved before shipment according to the procedures shown in Table 1-2 of the QAPP.

Preservatives added to the samples will be prepared using reagent grade chemicals. Table 1-2 of the QAPP should be consulted for details regarding sample packaging and shipping.

[mad-603-15i]

8

SAMPLE DOCUMENTATION

Field sampling activities will be documented using a bound waterproof notebook/logbook. Information recorded in the field logbook will include date of sampling, sampler, weather conditions, observations, and methods of preservation. Additional data pertaining to sampling may also be included in the logbook.

Samples will be collected under chain-of-custody procedures. Standard forms including sample labels, sample tags, chain-of-custody forms, and custody seals used for sample tracking will be maintained (see attachments). A brief description of sample documents follow:

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 all samples.

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 all samples.

C. Sample Tags

1. Each sample container must have a sample tag affixed to it.
2. Sample tag numbers are recorded on the Chain-of-Custody Forms.

3. Use for all samples.

D. Sample Identification Record Form will:

1. Provide means of recording crucial sample shipping and tracking information.

2. Contain information such as:

- Sample number
- Sample matrix
- Sample location code
- Sample round
- Chain-of-custody number
- Lab code
- Date sampled
- Date shipped
- Airbill number
- Sampling tag number

Paperwork accompanying the samples being shipped to the laboratory 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 with 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.

[mad-603-15j]

Tables

TABLE 1

SUMMARY OF DATA GENERATING ACTIVITIES
AND ASSOCIATED QUALITY OBJECTIVES
BELOIT CORPORATION ROCKTON FACILITY
ROCKTON, ILLINOIS

Activity	Use of Data	Data Quality Objective
TASK 1		
· Site Mapping	Prepare a site base map for use in developing other maps showing RI/FS data.	Accurately depict existing conditions, and prepare accurate RI data interpretation presentations.
· Evaluation of Existing Monitoring Wells	Assess the condition of existing wells at the site. Supplement the evaluation of geologic conditions at the site.	Identification of wells to be used during the RI.
TASK 2		
SOURCE CHARACTERIZATION		
· Electromagnetic/ Magnetometer Survey	Assess the presence and location of buried metal drums and/or underground storage tanks.	Obtain consistent relative differences among sampling points.
· Soil Gas Survey	Identify potential contaminant source areas. To optimize placement of groundwater quality and soil borings.	Identification of VOCs present in soil gas samples. Obtain relative differences among sampling points.
· Shallow Soil Borings and Soil Sampling (surface and subsurface soils)	Collect surface and subsurface soils and quantify contamination in potential source areas.	Identification of compounds present at source areas. Meet performance criteria for TCL parameters for organics and TAL parameters for inorganics as stated in the QAPP.

TABLE 1
(continued)

Activity	Use of Data	Data Quality Objective
MIGRATION PATHWAY ASSESSMENT		
Hydrogeologic Investigation		
· Deep and Intermediate Groundwater Quality Borings	Define hydrostratigraphy, subsurface geology and the potential for preferential groundwater flow pathways.	Screen the vertical extent of contaminant migration. Meet the performance criteria for methods described in the FSP.
· Monitoring Well Installation and Replacement	Characterize site geology and hydrogeology. Obtain groundwater samples for assessment of contaminant migration pathways.	Meet performance criteria for methods described in the FSP. Accurately and consistently describe the distribution of unconsolidated deposits.
· Hydraulic Conductivity Testing	Assess hydraulic conductivity rates at the site.	Obtain valid test results.
· Groundwater Level Measurements	Define site water table and groundwater flow directions. Evaluate vertical and horizontal hydraulic gradients; assist in evaluation of potential changes in contaminant migration.	Accurate water elevation measurements to the ±0.01 ft.
· Location and Elevation Survey	Locate soil borings, monitoring wells and other sampling locations.	Locate sampling points to an accuracy of ±1 ft horizontally; and monitoring wells to an accuracy of ±0.01 ft vertically.
· Surface Water and Sediment Assessment	Evaluate drainage patterns and runoff characteristics.	Inspection of the site by a qualified scientist.
· Air Assessment	Assess air migration pathway through health and safety monitoring during site activities.	Accurate collection of data with health and safety monitoring instruments.

TABLE 1
(continued)

Activity	Use of Data	Data Quality Objective
Contaminant Characterization		
· Groundwater Sampling	Provide preliminary assessment of contaminant source impact on groundwater quality, and to aid in assessment of groundwater migration pathways.	Meet performance criteria for TCL organics as stated in CLP SOP's. Meet performance criteria for inorganics and general water quality indicator parameters stated with method descriptions in the QAPP.

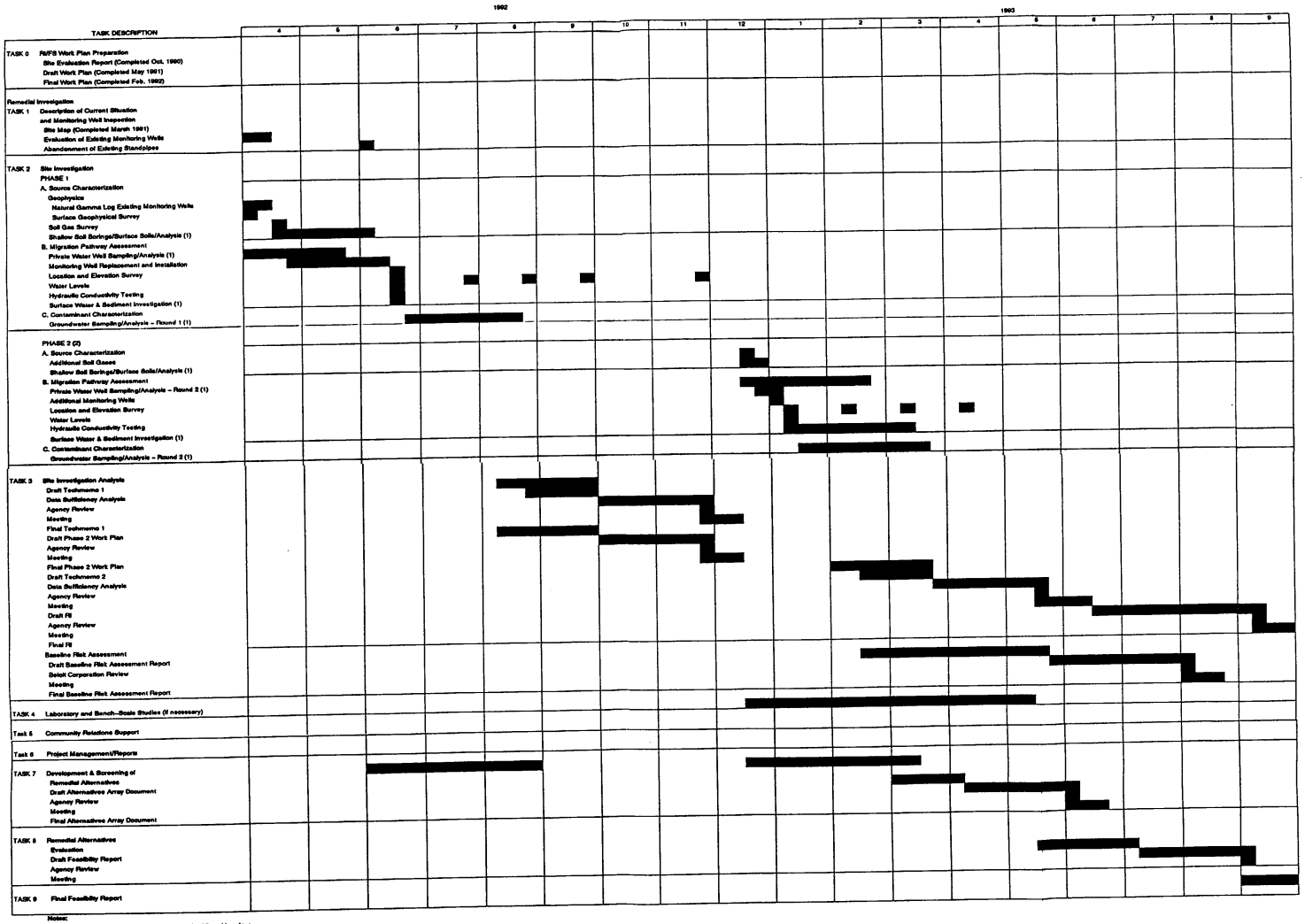
TJK/kml/JEM/KJD/KJQ
[mad-403-09]

Figure

FIGURE 1

PROPOSED PROJECT SCHEDULE
BELOIT CORPORATION RI/FS

YEARS/MONTHS



Notes:

(1) Analytical turnaround time is estimated to be 35 working days.

(2) Phase 2 activities are subject to change based on Phase 1 results, proposed activities and IEPA/U.S. EPA approval.
Phase 2 investigation activities may include none, some, or all of the listed activities.

Appendix A-A

Equipment

Appendix A-A1

Operator's Manual for the EM-31D Electromagnetic Concuctivity Meter

GEONICS LIMITED
1745 Meyerside Dr. Unit 8 Mississauga, Ontario Canada L5T 1C5

Tel. (416) 676-9580
Telex 06-968688
Cables: Geonics

OPERATING MANUAL

for

EM31-D

NON-CONTACTING

TERRAIN CONDUCTIVITY METER

Revised June 1984.

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1. INTRODUCTION

Measurement of ground resistivity is one of the oldest geophysical techniques. Table 1, taken directly from Heiland*, lists typical values of resistivity for a variety of geological materials (pages 4-7). The values given are in ohm-centimeters and must be divided by one hundred to give ohm-meters.

It will be observed that in most cases the actual resistivity itself is not diagnostic and a knowledge of the way in which the resistivity varies laterally and with depth is of great importance, since this permits us to "see" features as a result of their shape rather than their actual resistivity values. There is thus a requirement for instrumentation which permits the rapid and accurate measurement of terrain resistivity. Since the EM31 does not require electrical contact with the ground it fulfills this objective.

The basic principle of operation of the EM31 is simple. With reference to Fig. 1 a transmitter coil located at one end of the instrument induces circular eddy current loops in the earth. Under certain conditions fulfilled in the design of the EM31 the magnitude of any one of these current loops is directly proportional to the terrain conductivity in the vicinity of that loop. Each one of the current loops generates a magnetic field which is proportional to the value of the current flowing within that loop. A part of the magnetic field from each loop is intercepted by the receiver coil and results

* Heiland, C.A. Geophysical Exploration. Hafner Publishing Co., New York 1968

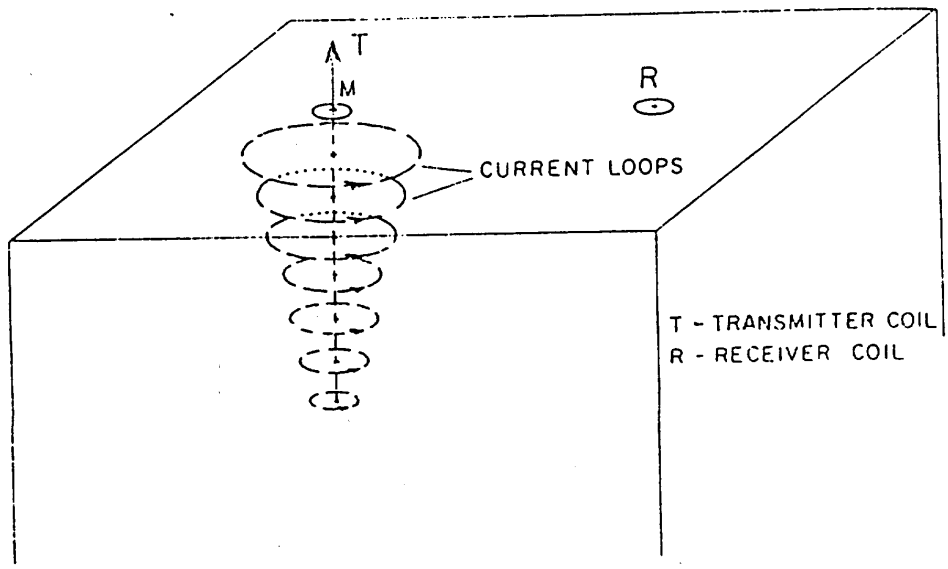


Fig. 1 INDUCED CURRENT FLOW IN GROUND

1. INTRODUCTION (cont'd)

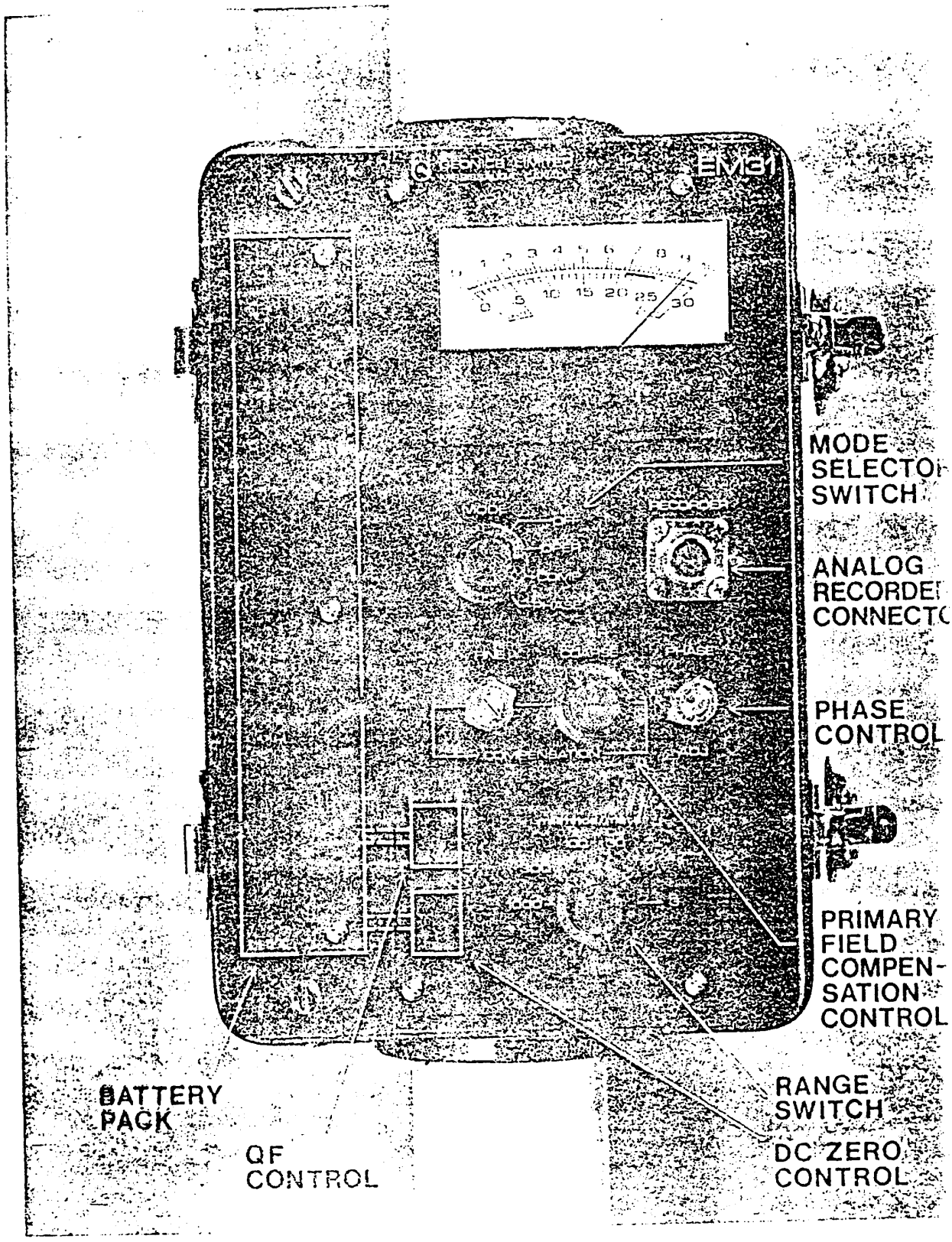
in an output voltage which is therefore also linearly related to the terrain conductivity.

This instrument is calibrated to read the correct conductivity when the earth is uniform. In the event that the earth is layered, with each layer of different conductivity, the instrument will read an intermediate value as discussed in more detail in Section 5.2.

The unit of conductivity used is the millimho per meter. To obtain resistivity in ohm-meters the instrument reading is divided into 1000 - i.e., a reading of four millimhos per meter divided into 1000 gives two hundred and fifty ohm-meters.

Theoretical calculations show, as will be quickly evident to the operator, that the reading obtained is essentially independent of the orientation of the instrument with respect to the earth. There is, however, a small dependence on the height above the ground; lifting the instrument from the surface of a uniform earth to the normal operating height of about one meter results in a reduction in the reading of 12%. The calibration has been adjusted at the factory so that the instrument reads correctly over a uniform half-space when worn as shown in the data sheet. If the earth is layered, raising the instrument from the surface of the earth to the normal operating position can result in a reading which stays constant or even increases slightly with height. In general, readings made with the instrument at hip height will be sufficiently accurate, but for maximum accuracy the instrument can be laid on the ground as discussed in Section 5.2.

There are two components of the induced magnetic field measured by the EM31. The first is the quadrature-phase component which gives the ground conductivity measurement as described. The second is the inphase component used primarily in the EM31 for calibration purposes. The inphase component however is significantly more sensitive to large metallic objects and hence very useful when looking for buried metal drums. Although this component has been available in the standard EM31 by operating in the compensation mode (Section 2.2), both components are now available in the EM31-D from an analog output connector when coupled to a dual channel recorder.


EM31
MODE SELECTOR SWITCH
ANALOG RECORDER CONNECTOR
PHASE CONTROL
PRIMARY FIELD COMPEN-SATION CONTROL
RANGE SWITCH
DC ZERO CONTROL
BATTERY PACK
QF CONTROL

EM31-D SPECIFICATIONS

Measured Quantities	(1)	Apparent conductivity of the ground in millimhos per meter
	(2)	Inphase component of the induced magnetic field
Primary Field Source		Self-contained dipole transmitter
Sensor		Self-contained dipole receiver
Intercoil Spacing		3.66 meters
Operating Frequency		9.8 kHz
Power Supply		8 disposable alkaline 'C' cells (approx. 20 hrs life continuous use)
Conductivity Ranges		3, 10, 30, 100, 300, 1000 mmhos/meter
Measurement Precision		± 2% of full scale
Measurement Accuracy		± 5% at 20 millimhos per meter
Noise Level		<0.1 millimhos per meter
Operator Controls		•Mode Switch •Conductivity Range Switch •Phasing Potentiometer •Coarse Inphase Compensation •Fine Inphase Compensation
Analog Output		Connector on EM31-D for dual channel analog recording of ground conductivity and inphase component
Dimensions		Boom : 4.0 meters extended 1.4 meters stored Console : 24 x 20 x 18 cm Shipping Crate: 155 x 42 x 28 cm
Weight		Instrument Weight: 9 kgm Shipping Weight: 23 kgm

2. OPERATING INSTRUCTIONS

The EM31 can be used both to measure the electrical conductivity of the ground and to detect buried metal objects. Section 2.1 describes the procedure for measuring ground conductivity and section 2.2 for buried metal detection.

2.1 Ground Conductivity Measurements

2.1.1 Initial Set-up Procedure

a) Using the identifying labels on the tubes select the transmitter coil tube, align it with respect to the main tube, insert it and fix it with the clamp.

b) Check the battery condition, plus and minus, by setting the Mode switch (MODE SELECTOR SWITCH) to the OPER position and the Range switch to the +B and -B positions respectively. If the needle reads inside the BATT mark on the meter, batteries are in good condition, otherwise replace the batteries with a fresh set of C size alkaline batteries.

c) Check the zero reading by setting the Mode switch to the OPER position and the Range switch to the least sensitive position 1,000 mmhos/meter (this minimizes any external noise interference while checking the zero position). If a zero adjustment is required adjust the DC ZERO CONTROL located under the front panel to obtain a zero reading. To do this the battery pack must be removed to gain access to the controls.

d) Align and connect the receiver coil tube to the main frame tube. The instrument is now ready to proceed with the functional checks.

2.1.2 Equipment Functional Checks

The Range switch should be set at 30 millimhos/meter position for all the following tests. (If the reading is off scale, i.e., greater than 30 millimhos/meter, see note.)

a) Set the Mode switch to the COMP position and adjust the meter reading to zero using the COARSE and FINE COMPENSATION controls.

b) To check the phasing of the instrument set the Mode switch to the PHASE position. Note the meter reading and rotate the COARSE control one step clockwise. If the meter reading remained the same, the phasing is already correct; return the COARSE control to its original position (one step counter clockwise) and no further adjustment is necessary.

If there was a difference in the meter readings taken before and after the COARSE control was rotated one step clockwise then a phase adjustment is required. With the COARSE control in its original position adjust the PHASE potentiometer about 1/4 turn clockwise and note the new meter reading. Rotate the COARSE control one step clockwise, take a reading, and return the COARSE control to its original position. If the difference in meter readings has decreased, repeat the procedure using a further clockwise adjustment, until rotating the COARSE control the one step clockwise produces no change in the meter reading.

If, on the other hand, the difference in meter readings was increased, the PHASE potentiometer should be rotated in a counter clockwise direction instead and the procedure described above repeated until there is no change in the meter readings. Always remember to set the COARSE control back to its original position. This can be confirmed by setting the Mode switch in the COMP position and checking that the meter reads zero. If it does not read zero, repeat steps (a) and (b).

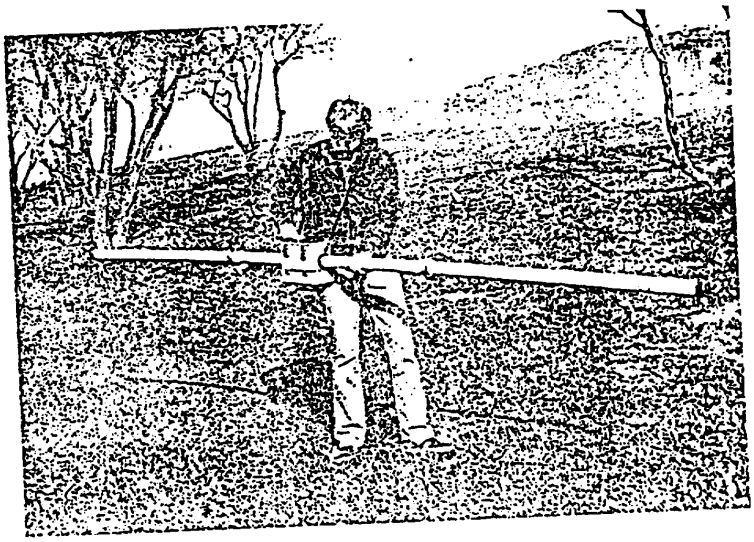
c) To check the sensitivity of the instrument, set the Mode switch to the COMP position and rotate the COARSE control clockwise one step. The meter should read between 75% and 85% (22 to 26 millimhos/meter) of full scale deflection (inside black mark). It is unlikely that the sensitivity of the instrument will vary, however it may be useful to record the actual meter reading for comparison at a later date.

Return the COARSE switch to its original position and the EM31 is now ready to make ground conductivity measurements.

NOTE: When conducting the functional tests over ground of higher conductivity than 30 millimhos/meter, the Range switch should be set at the appropriate level. At whatever level the Range switch is in, the reading taken in (c) should still be between 22 and 26 millimhos/meter.

2.1.3 Operating Procedure

a) Wearing the instrument with the shoulder strap adjusted so that the instrument rests comfortably on the hip as shown, switch the Mode switch to the OPER position and rotate the Range switch so that the meter reads in the upper two thirds of the scale. The full scale deflection is now indicated by the Range switch and the instrument is reading the terrain conductivity directly in millimhos per meter.



b) The EM31 can be operated continuously while moving from one measurement station to the next however, the instrument has a time constant of about one second for which the operator should adjust his walking speed to obtain greatest accuracy.

Alternatively, to extend battery life the instrument can be switched on at each measurement station when not using a recorder. The operator will notice that the type of integrator used results in a slight initial overshoot of the needle at turn on. This is normal, and that at least two seconds should be allowed after turn on before the measurement is taken.

2.2 Buried Metal Detection

2.2.1 Set-up and Operating Procedure

The inphase component of the magnetic field is significantly more sensitive to large metallic objects than the quadrature-phase component used for ground conductivity measurements.

a) The inphase component is readily measured with the EM31 by simply taking the reading with the Mode switch in the COMP position rather than in the OPER position.

It is recommended that the 30 mmho/m range be used since it usually gives adequate sensitivity, however more or less sensitive positions of the Range switch may also be used.

b) To carry out a survey measuring the inphase component set the Mode switch to the COMP position and adjust the COARSE and FINE compensation controls so that a meter deflection of about 20% of full scale deflection is obtained. (The procedure of adjusting to 20% of the full scale deflection rather than to zero is only a convenience to allow for negative readings on the meter and under certain conditions a possible change in the reference level. For example, a sudden jar to the instrument can result in a small positive or negative change in the inphase reference level).

The survey is then carried out exactly as if the conductivity were being measured.

c) This lack of a true zero reference should not cause any serious problems since when using the inphase component one is usually only looking for buried metallic objects.

These will be easily recognizable by relatively localized meter deflections occuring either singly or in a series of responses depending on the number of buried objects, their spacing and depth of burial.

Notes (1): The 20% offset should only be used when the inphase component only is being measured continuously. If both conductivity (quadrature phase) and inphase measurements are being made at each station the procedure described in 2.1.2 (a) should be used so that accurate ground conductivity measurements are obtained. If negative inphase measurements are obtained one should check the inphase reference level and/or the area for buried metallic objects or utilities.

(2) Similarly when the dual channel recorder is being used there should be no zero offset as described in 2.2.1 (b) and the meter reading set to zero using the COARSE and FINE controls as described in 2.1.2 (a).

A zero offset for negative inphase readings can however be allowed for on the recorder by setting the zero position of the pen at some arbitrary positive value when calibrating the recorder.

3. INSTRUMENT CALIBRATION

Prior to shipping, the instrument is calibrated in the factory to read properly. If necessary, calibration procedures are easily carried out as described below. IMPORTANT The most critical adjustment is the QF (quadrature fine) potentiometer which has been precisely adjusted at the factory.

Before any adjustments are made it is strongly recommended that the instrument first be set up at a fixed height over a known location and the ground conductivity carefully noted. If this adjustment is misaligned the instrument will have to be recalibrated over ground of known conductivity.

3.1 Null Calibration

The zero setting of the EM31 can be readily set by following the procedure described in section 2.1.1 (c).

3.2 Absolute Calibration

Absolute instrument calibration is easily achieved if an area of ground is available of known and constant conductivity down to the depth of penetration of the instrument. The procedure is simple; the instrument is located over the known area at the usual operating height (approximately 1m) and the QF compensation control is adjusted until the meter reads the correct terrain conductivity. If the ground conductivity is high the graph of Fig. 3 must be used to correctly set the instrument reading.

It is wise to maintain such an area as a calibration check area even if the variation of the conductivity with depth at that area is not accurately known. This is useful for cross checking with future measurements.

Note:

The QF and NULL controls are located under the front panel. Battery pack should be removed to have access.

4. SURVEY TECHNIQUE

Surveying with the EM31 is straightforward. As pointed out in a previous section, measurements may be made either continuously or on a station-by-station basis. In either case it is always recommended, as for any other geophysical survey, that survey lines and measurement stations be carefully laid out, and the survey performed in a systematic fashion with the resulting data accurately plotted for each measurement station. The most common survey error is to have the survey lines too short, in which case they do not extend sufficiently far off the supposedly anomalous region to permit the operator to establish the background values of terrain conductivity.

The EM31 yields good spatial resolution and measurement stations should be ten meters apart if the maximum resolution is to be employed. In many cases this spacing will be smaller than necessary, and twenty or perhaps forty meters will be adequate. The decision as to the correct spacing will be based on a knowledge of the lateral dimensions of the anticipated resistivity anomaly. To ensure the correct spacing it is useful, at the start of the survey, to continuously observe the conductivity values that are encountered as the operator moves down the survey line.

The resolution in conductivity of the EM31 is also high, with changes of 5% being quickly perceived. This instrument is capable of giving an extremely precise survey with information on small variations in the terrain conductivity.

4. SURVEY TECHNIQUE (cont'd)

It was seen in Section 1 that current flow within the earth consists of a series of concentric circles, assuming that the conductivity is laterally uniform. Therefore in the case of a uniform half-space, rotation of the instrument in a horizontal plane about the transmitter coil as a pivot will produce no change in the meter reading. Conversely, any change in the reading as this procedure is carried out is an indication of lateral inhomogeneities in conductivity. It is simpler and usually sufficiently accurate for the operator to rotate the instrument through 90° using himself as pivot at each measurement station. Thus if the lines are in a north-south direction the operator would normally walk along the line with the instrument pointing in a north-south direction; at each measurement station he can also take a reading with the instrument pointing east-west to check that this is essentially the same as the north-south reading. In the event that this reading is significantly different it may be worthwhile for the operator to then rotate the instrument to the points where the conductivity reading is both a maximum and a minimum, and to record both values. The average value can then be used for the data reduction.

The EM31 is somewhat sensitive to underground conductors such as large pipes, etc. These are usually easily recognized by the large meter fluctuations which occur within a short distance, as shown in Fig. 2. In an actual survey, since the negative-going peak is often off-scale, it is not possible to use it to locate the pipe; finding the point half way between the two positive-

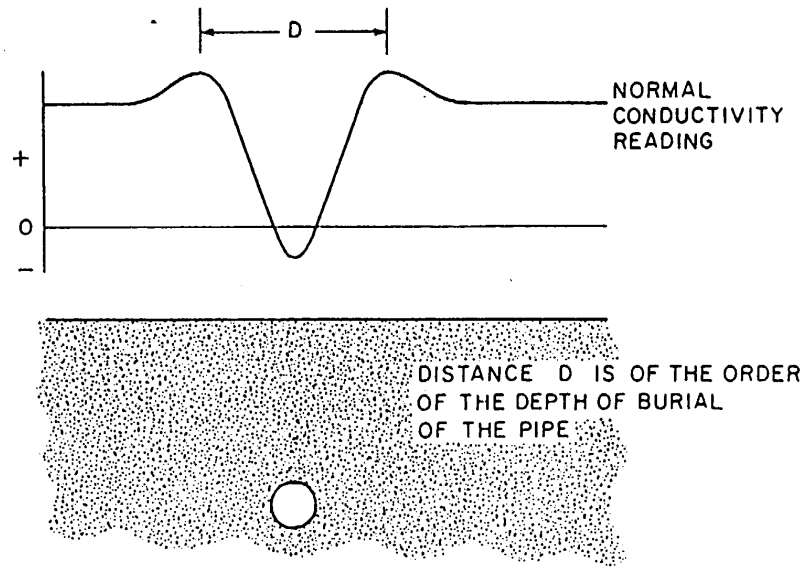


Fig. 2 TYPICAL RESPONSE OVER A PIPE

4. SURVEY TECHNIQUE (cont'd)

going peaks approximately performs the same task. It is then possible to accurately determine the location and strike direction (azimuth) of the conductor axis as follows: the approximate location is determined as above, and a traverse is then made over the conductor with the EM31 pointing in the approximate direction of the conductor axis. The meter reading will now be a positive maximum when the instrument is both directly over the conductor and pointing accurately along the conductor axis.

The instrument is relatively unaffected by fences, overhead power lines, and other nearby metallic objects. In order to determine whether the reading is influenced by such structures the operator should rotate the instrument to check for changes in reading, becoming suspicious if a maximum or minimum occurs when the instrument points either perpendicular or parallel to the structure. Before recording the measurement the operator should move away from the structure until no evidence of lateral inhomogeneity is seen when the instrument is rotated.

It should be remembered that the EM31 is an electromagnetic tool and care should be taken near obvious conductors until the operator has satisfied himself as to their possible affect. In every case this is determined by rotating the instrument and determining whether there is a maximum and minimum which appears to be realted to the structure. If a structure is giving such an effect it is not advisable to take the average value of the two readings as an indicator of the terrain conductivity.

4. SURVEY TECHNIQUE (cont'd)

In general the conductivity readings obtained with the EM31 will vary smoothly from one region to another. In some cases however, as for example where a well defined vertical contact separates a poor conductor from a very good conductor, edge effects may be seen in which the readings vary rapidly with position and are no longer a good indicator of the terrain conductivity. Edge effects may also occur where a very good conductor (a few ohm-meters or less) has dimensions of the order of the inter-coil spacing, and again the indicated readings may not accurately reflect the true terrain conductivity. In any circumstance where the apparent conductivity varies significantly in a distance which is short compared with the intercoil spacing the possible presence of edge ef-fects or local subsurface conductors must be considered.

Finally, particularly during mid-summer afternoons, electrical static (electromagnetic radiation from local or distant thunderstorms) may cause the meter readings to become noisy. This is usually evidenced by sudden flicks of the meter needle, however in very severe cases the meter may simply wander about an average reading. Should this occur it is recommended that measurements cease until the "spherics" are over, usually later in the afternoon. Similarly, noisy readings may also be noted when making measurements near large power lines.

5. DATA INTERPRETATION

5.1 Uniform Halfspace

The EM31 has been designed to operate over a range of resistivities from a few ohm-meters to thousands of ohm-meters. For smaller resistivities the instrumental response departs from linearity as shown in Fig. 3, where it will be seen that up to one hundred millimhos per meter the indicated conductivity (σ_a) closely approximates the true conductivity. A departure from linearity is evident for true conductivity greater than one hundred millimhos per meter and beyond one thousand millimhos per meter the instrument response decreases with increasing conductivity. As stated in the introduction, it is generally more informative to observe the spatial variations of terrain conductivity rather than the absolute value of conductivity itself. Fig. 3 shows that as long as the terrain conductivity does not fall below five hundred millimhos per meter (2 ohm-meters) the instrument will be adequately sensitive to small changes in conductivity although it does not read the actual value of conductivity accurately. If necessary, Fig. 3 can be used to correct values of indicated conductivity to actual conductivity.

It should be noted that the graph refers to the worst case viz that of the uniform halfspace. If only a portion of the subsurface ground beneath the instrument is of high conductivity as in the case of horizontal layering, the influence of the high conductivity layers will be proportionately reduced and the indicated conductivity will accurately read the "apparent conductivity" as defined in the following section.

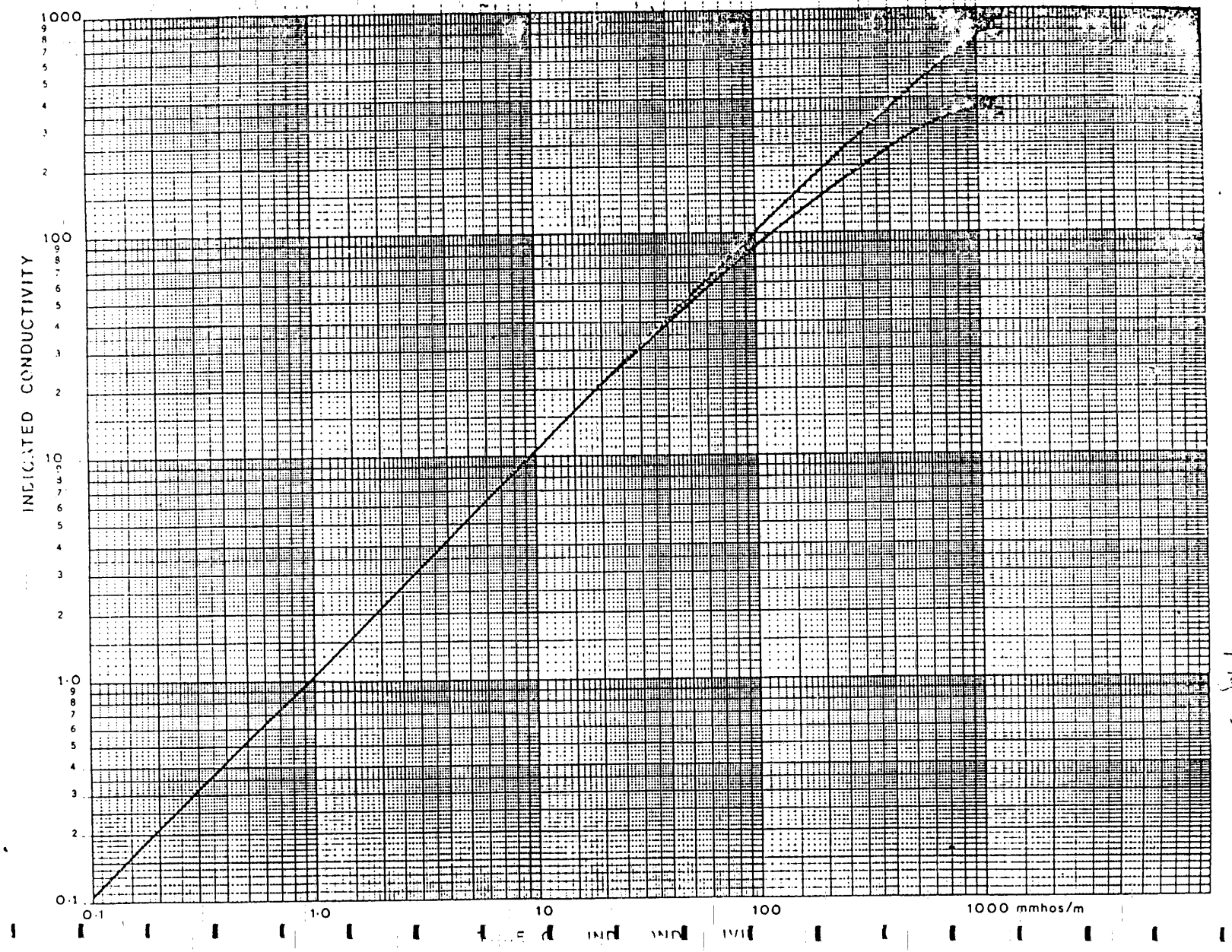

INDICATED CONDUCTIVITY
1000
100
10
1·0
0·1
0·1
1·0
10
100
1000 mmhos/m

Fig 2

5.2 Multi-Layered Earth

A geophysical model that is of particular importance is the horizontally layered earth, and the EM31 allows a very simple interpretive technique for this model. In order to utilize the model the terrain layering must be well defined and constant over a lateral distance of at least five meters in any direction from the instrument. This condition is often satisfied and this fact is responsible for the usefulness of the technique described herein.

Fig. 4 is a plot of "R(Z)", a function which describes the cumulative relative contribution of all of the material below a depth Z to the instrument reading. Thus if we multiply this function by one hundred (so as to yield percent) all of the ground below a depth of two meters yields 59% of the response, the ground below three meters yields 47%, the ground below six meters yields 29%, etc., assuming that the conductivity itself does not vary with depth.

The algebraic expression given on the figure illustrates the technique which is used to calculate the "apparent conductivity" that will be measured by the instrument for any number of layers, for any values of conductivity and for any thicknesses. Consider for example Fig. 5, which illustrates in cross section a buried river valley cut into shale and subsequently infilled with glacial till. We wish to calculate the instrument response as such a structure is traversed. This is a two layer problem and the expression for the apparent conductivity reduces to:

$$\frac{\sigma_a}{\sigma_1} = 1.000 - R(Z_1) + k_2 R(Z_1) \qquad (1)$$

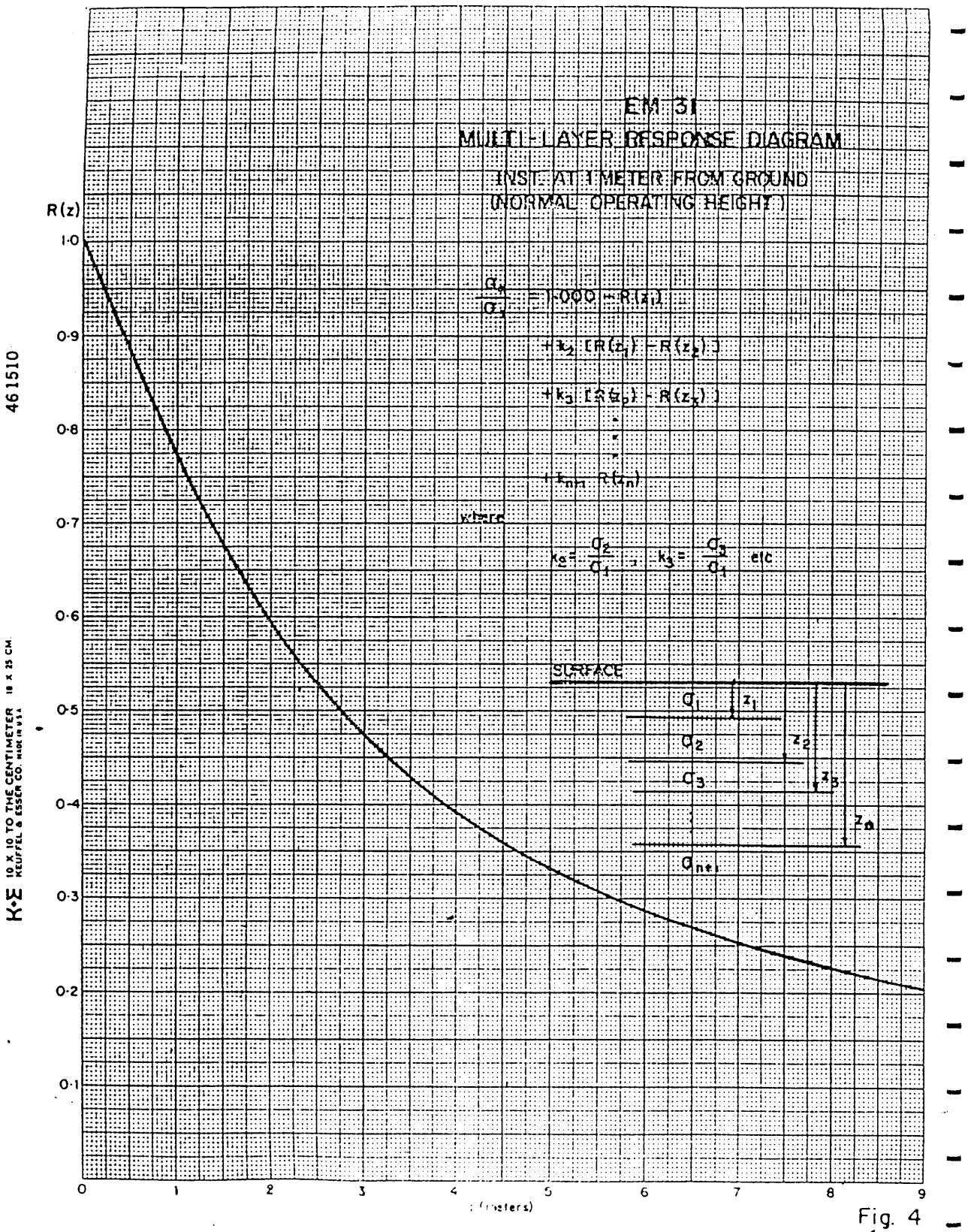


Fig. 4

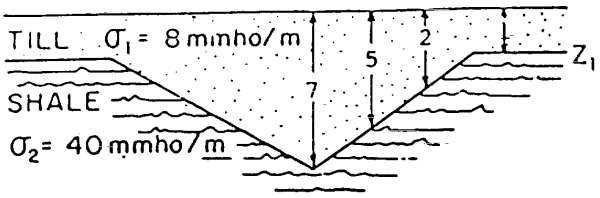
46 1510
K+E 10 X 10 TO THE CENTIMETER 18 X 25 CM.
KEUFFEL & ESSER CO. MADE IN U.S.A.

5.2 Multi-Layered Earth (cont'd)

The table accompanying the figure shows the calculations which yield the value of apparent conductivity at various thicknesses of the upper layer. Thus as we traverse such a structure we would find that the apparent conductivity fell from 32.6 millimhos per meter at a large distance from the buried river valley to 16.0 millimhos per meter at the center of the valley.

Fig. 6 illustrates a second situation where now the objective is to locate bedrock highs within muskeg. This is again a two layer case and the table illustrates the calculations to determine the apparent conductivity. For this configuration, i.e. a conductor on top of an insulator and k_2 a small quantity, the measured apparent conductivity becomes relatively independent of the actual value of the conductivity of the lower layer. For $k_2 << 1$, the apparent conductivity simply becomes a function of the thickness of the upper conductive layer.

Finally a third example is given in Fig. 7. In this case we wish to traverse a thickening gravel deposit; the maximum thickness is such that the instrument still responds to material below the deposit. This is an example of the situation of an insulator sandwiched between two conductors and is inevitably the most difficult geometry for electromagnetic systems to detect, as evidenced by the tabulated values of apparent conductivity. Even at a thickness of five meters (station D) there is still significant response from the till beneath the deposit and this response tends to keep the apparent conductivities high.

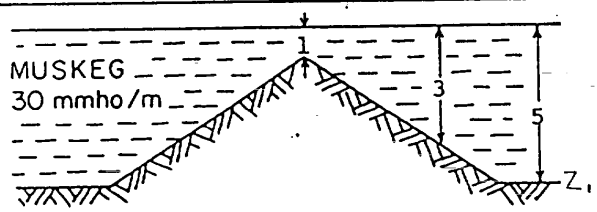


$$\frac{\sigma_a}{\sigma_1} = 1{\cdot}000 - R(Z_1) + k_2 R(Z_1) \qquad (1)$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{40}{8} = 5$$

z_1 = 1m, σ_a = 8(1.00 - 0.77 + (5 x 0.77)) = 32.6 mmho/m
z_1 = 2m, σ_a = 8(1.00 - 0.59 + (5 x 0.59)) = 26.9 "
z_1 = 5m, σ_a = 8(1.00 - 0.33 + (5 x 0.33)) = 18.6 "
z_1 = 7m, σ_a = 8(1.00 - 0.25 + (5 x 0.25)) = 16.0 "

Fig. 5 CROSS SECTION BURIED RIVER VALLEY

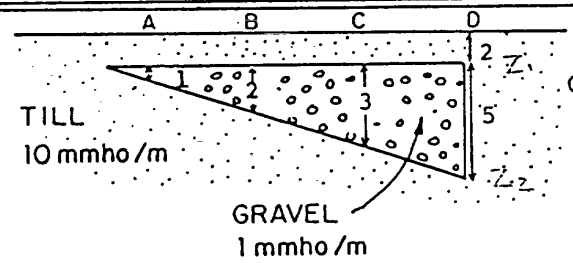


$$\frac{\sigma_a}{\sigma_1} = 1{\cdot}000 - R(Z_1) + k_2 R(Z_1)$$

$$k_2 = \frac{0{\cdot}1}{30} = 0{\cdot}0033$$

z_1 = 5m, σ_a = 30(1.00 - 0.33) = 20.1 mmho/m
z_1 = 3m, σ_a = 30(1.00 - 0.47) = 15.9 "
z_1 = 1m, σ_a = 30(1.00 - 0.77) = 6.9 "

Fig. 6 CROSS SECTION BEDROCK HIGH



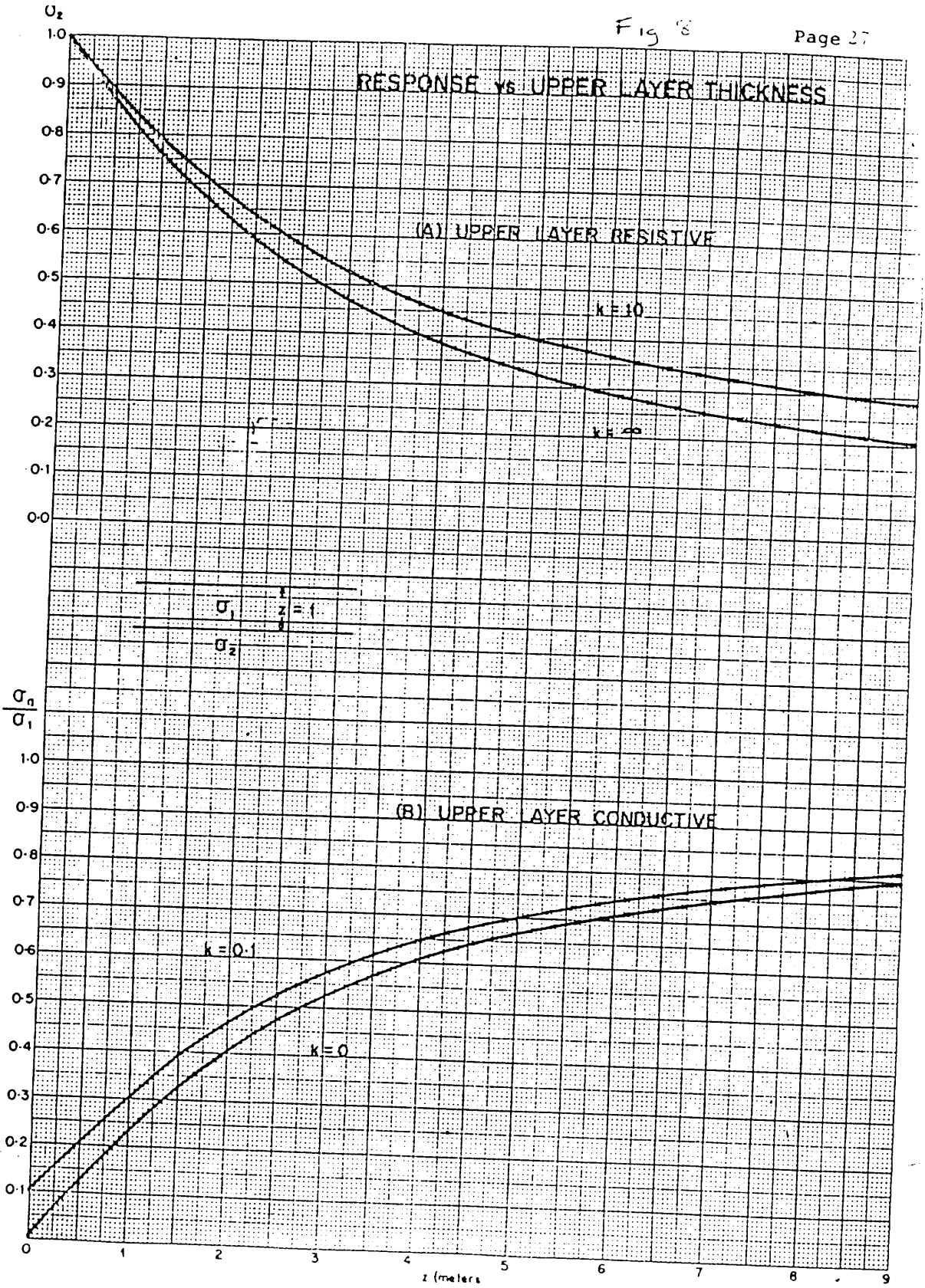
$$\sigma_a = \sigma_1 [1{\cdot}000 - R(Z_1) + k_2 R(Z_1) - k_2 R(Z_2) + k_3 R(Z_2)]$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{1}{10} = 0{\cdot}10$$

$$k_3 = \frac{\sigma_3}{\sigma_1} = 1{\cdot}00$$

(A) σ_a=10 (1.00-0.59+(0.10x0.59)-(0.10x0.47)+(1.00x0.47)) = 8.9 mmho/m
(B) σ_a=10 (1.00-0.59+(0.10x0.59)-(0.10x0.39)+(1.00x0.39)) = 8.2 "
(C) σ_a=10 (1.00-0.59+(0.10x0.59)-(0.10x0.33)+(1.00x0.33)) = 7.7 "
(D) σ_a=10 (1.00-0.59+(0.10x0.59)-(0.10x0.25)+(1.00x0.25)) = 6.9 "

Fig. 7 CROSS SECTION GRAVEL DEPOSIT



K+E 10 X 10 TO THE CENTIMETER 18 X 25 CM
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5.2 Multi-Layered Earth (cont'd)

The three examples show how to calculate the response of the EM31 over a variety of geological environments. Since the instrument operates at a fixed intercoil spacing it is not possible to completely "sound" or evaluate differing conductivity layers with depth. Section 5.3 will show how to further resolve the two-layer case, however it is always useful to be able to calculate the apparent conductivity which would arise from a given multi-layer model to see whether that model fits the measured data.

Fig. 8 shows the apparent conductivity over the two limiting cases of a two layer geometry where the conductivity contrast is very large. In Fig. 8A the upper layer is assumed to be very resistive and the figure shows the apparent conductivity with respect to the lower layer conductivity (assumed known) as a function of the depth below the surface of the lower layer, for values of k of ten and infinity. Thus for large k the figure permits the operator to quickly convert the measured values of apparent conductivity to depth, and to estimate the error if the conductivity contrast is not infinite.

Fig. 8B performs the same task for small values of conductivity contrast. It should be noted that Fig. 8B plots the apparent conductivity with respect to the upper layer conductivity.

It is stated in the data sheet that the effective depth of penetration of the EM31 is approximately six meters. Justification for this claim is shown in Fig. 8 where it is seen that for either a resistive ($k = \infty$) or conductive ($k = 0$) upper layer the apparent conductivity is still

5.2 Multi-Layered Earth (cont'd)

satisfactorily sensitive to changes in the upper layer thickness at six meters. The limitation in resolving further changes in thickness is imposed by probable variations in the conductivity of either layer.

5.3 Geometrical Sounding of Two Layered Earth

It is possible with the EM31 to determine whether the conductivity increases or decreases with depth by laying the instrument on the ground, noting the reading, and then placing the instrument on its side so that the meter face is vertical and again noting the instrument reading. The reason for this is that when the instrument is on its side the new transmitter and receiver coil orientation with respect to the earth causes the effective depth of penetration of the instrument to be essentially halved. It should be noted that in this configuration the current flow does not exhibit the circular symmetry shown in Fig. 1. Thus let σ_a be the apparent conductivity indicated by the instrument on the ground in its normal operating position and let σ_a' be the apparent conductivity indicated when the instrument is lying on its side. If σ_a is greater than σ_a' we know that the conductivity is increasing with depth and vice versa. The ability of the EM31 to indicate whether the conductivity is increasing or decreasing with depth is often of considerable diagnostic value.

It will be observed from the above that it is now possible to obtain two measured quantities at each measurement station. In the event that the earth consists only of two layers, there are three unknown quantities i.e.

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

the conductivity of the upper and lower layer and the thickness of the upper layer. If one of these is known it is possible, using the technique described above, to determine the other two. More importantly, when there is a large conductivity contrast so that the conductivity of either the upper or lower layer can be ignored with respect to the other layer then it is possible to completely resolve the two layered earth. An example of the importance of this can be seen in Fig. 8 which gives the instrumental response as a function of upper layer thickness for either a resistive or conductive upper layer. In order to use these graphs it is necessary to know the conductivity of the lower or upper layer respectively, since both graphs are normalized with respect to these quantities. An inaccurate knowledge of σ_2 in the case of a resistive upper layer or σ_1 in the case of a conductive upper layer can result in a substantial error in the calculated thickness of the upper layer. Any technique which allows us to determine the conductivity of the more conductive layer is of considerable importance since by definition the EM31 is most useful when one layer presents a significant conductivity contrast with respect to the other.

Fig. 9 shows a multi-layer response diagram R(Z), completely analogous to Fig. 4 but with the instrument now laying on the ground. Fig. 10, which presents the function R'(Z) as a function of depth is the same function as Fig. 9 but now with the instrument rotated on its side so that the meter face is vertical. It will be noted from Fig. 9 that with the instrument on the ground in its normal operating configuration the total contribution from

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

all ground in excess of a depth of five meters is 34% whereas Fig. 10 shows that with the instrument lying on its side the ground in excess of five meters causes a total contribution of only 17.5% of the total response. This is the justification for stating earlier that the instrument has effectively one half the depth penetration when lying on its side. Fig. 9 also contains a plot of the ratio R'/R and Fig. 10 a plot of the ratio (1-R')/(1-R), both as a function of depth.

Case 1. σ_1 and σ_2 comparable and σ_1 known. It is shown in Appendix 1 that for this case

$$\frac{R'(Z)}{R(Z)} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1}, \qquad \sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R'} = \frac{\sigma_a' - \sigma_1 + R'\sigma_1}{R'}$$

thus given σ_a', σ_a, and σ_1, R'/R is calculated; Fig. 9 then gives the thickness of the upper layer as a function of R'/R; with this thickness Fig. 9 or 10 is used to determine R or R' and the second equation given above is used to determine σ_2 thus fully resolving the two layer case.

Case 2. σ_1 and σ_2 comparable and σ_2 known. In this case Appendix 1 shows that

$$\frac{1 - R'(Z)}{1 - R(Z)} = \frac{\frac{\sigma_a'}{\sigma_2} - 1}{\frac{\sigma_a}{\sigma_2} - 1}, \qquad \sigma_1 = \frac{\sigma_a - \sigma_2 R}{1 - R} = \frac{\sigma_a' - \sigma_2 R'}{1 - R'}.$$

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

Knowing σ_a', σ_a, and σ_2, (1-R')/(1-R) is calculated, Fig. 10 gives the thickness and given the thickness Fig. 9 or 10 gives R or R' so that the second equation given above is used to calculate σ_1.

Case 3. Upper layer resistive. σ_1 much less than σ_2. In this case Appendix 1 shows that

$$\frac{R'(Z)}{R(Z)} = \frac{\sigma_a'}{\sigma_a}, \qquad \sigma_2 = \frac{\sigma_a}{R} = \frac{\sigma_a'}{R'}$$

and the same proceedure as for Case 1 above is used to determine the thickness of the upper layer and the conductivity of the lower layer.

Case 4. Upper layer conductive. σ_1 much greater than σ_2. Again Appendix 1 shows that

$$\frac{1 - R'(Z)}{1 - R(Z)} = \frac{\sigma_a'}{\sigma_a}, \qquad \sigma_1 = \frac{\sigma_a}{1 - R} = \frac{\sigma_a'}{1-R'}$$

and the calculations for Case 2 yield the thickness and the conductivity of the upper layer.

To illustrate the above let it be assumed that the upper layer is known to be 10 millimhos per meter and the EM31 indicates 3.8 millimhos per meter when laying on the ground in its normal configuration and 6.5 millimhos per meter when lying on its side. Fig. 3, which also gives the correction factor to use when the instrument is lying on its side, shows that no correction is necessary.

5.3 Geometrical Sounding of Two-Layered Earth (cont'd)

Since the instrument has been calibrated to be operated at one meter above the ground both of these numbers must be multiplied by .88 since the instrument is being used on the ground. This results in $\sigma_a = 3.3$ millimhos per meter and $\sigma_a' = 5.7$ millimhos per meter. Then

$$\frac{R'(Z)}{R(Z)} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1} = \frac{\frac{5.7}{10} - 1}{\frac{3.3}{10} - 1} = 0.64$$

whereupon Fig. 9 shows that the thickness of the upper layer is 1.25 meters and R(Z) = 0.825 whereupon

$$\sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R} = \frac{3.3 - 10 + 0.825 \times 10}{0.825} = 1.9$$

The technique for the other cases described above is exactly the same.

The calculations described above assume that the earth is known to be two-layered. It is possible to determine whether this is true by measuring the apparent conductivity with the EM31 elevated to various heights above the ground and comparing the results with a family of curves given in Appendix II. If the measured variation of apparent conductivity with height matches one of the curves, one can immediately calculate the conductivity of both layers and the thickness of the upper layer, as described therein.

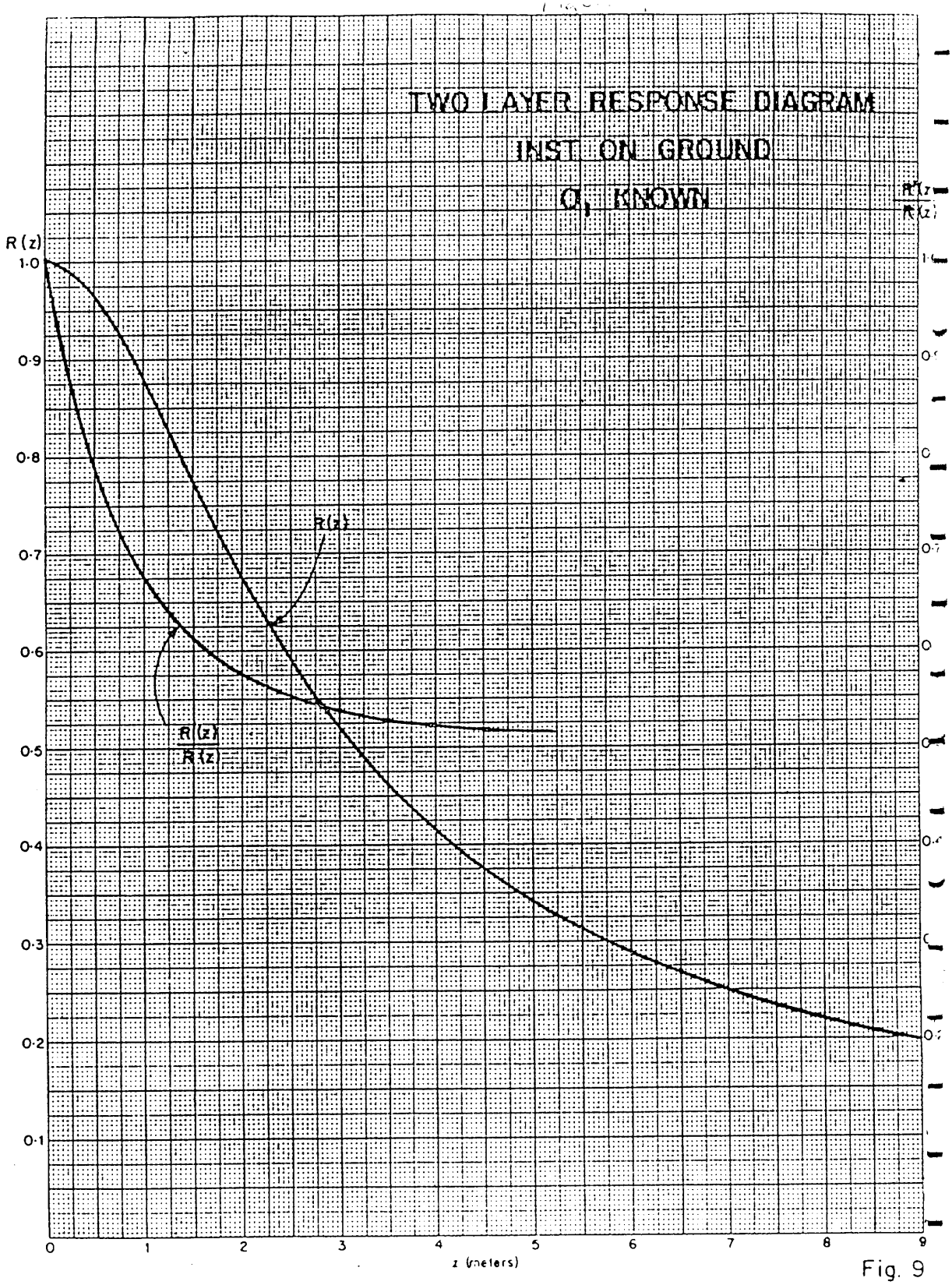


Fig. 9

461510
K+E 10 X 10 TO THE CENTIMETER 18 X 25 CM.
KEUFFEL & ESSER CO. MADE IN U.S.A.

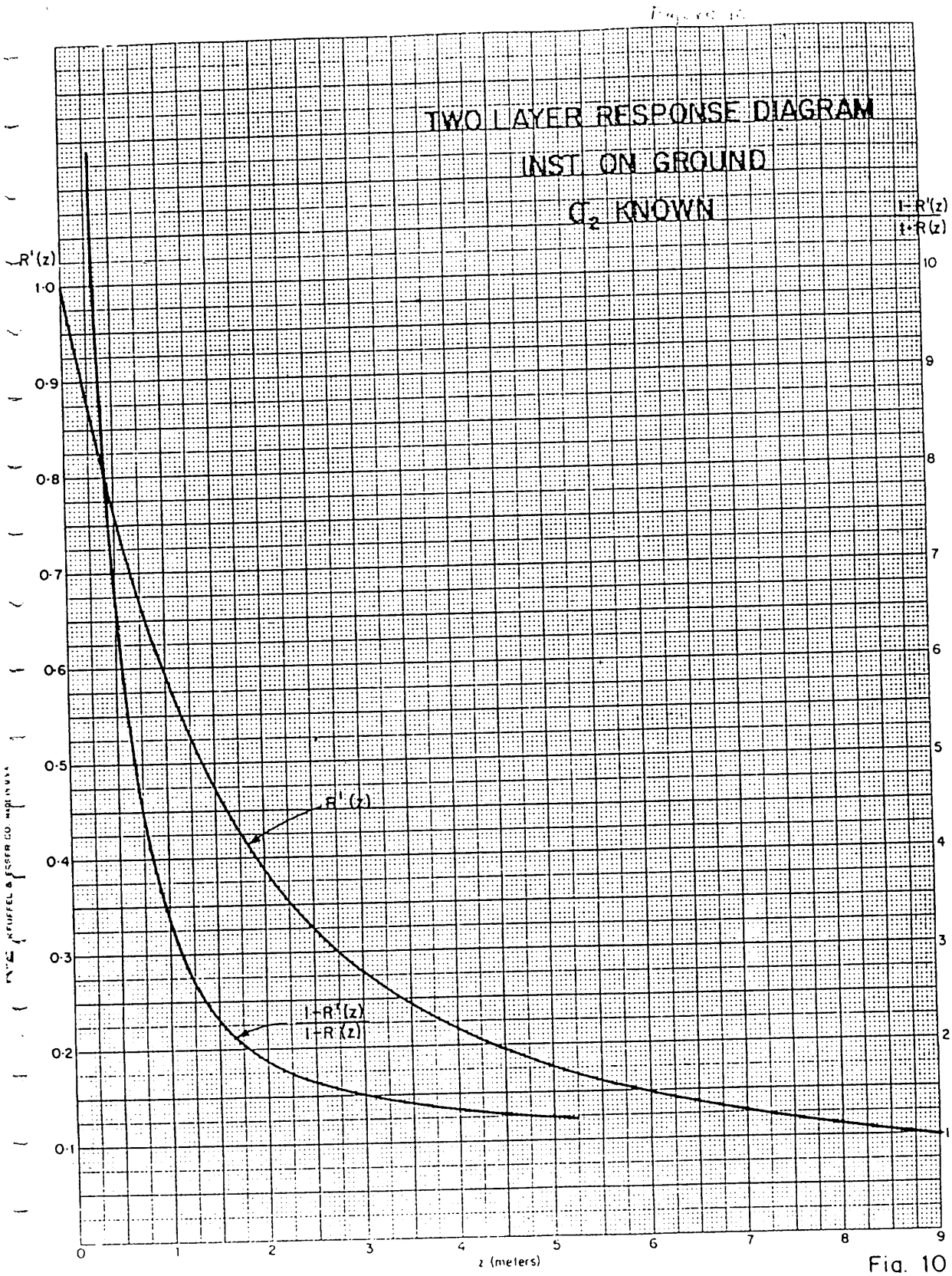

TWO LAYER RESPONSE DIAGRAM
INST. ON GROUND
C_2 KNOWN
R'(z)
1-R'(z)
1+R(z)
R'(z)
1-R'(z)
1-R(z)
z (meters)
0
1
2
3
4
5
6
7
8
9
0·1
0·2
0·3
0·4
0·5
0·6
0·7
0·8
0·9
1·0
10

Fig. 10

6. CASE HISTORIES: ELECTROMAGNETIC NON-CONTACTING GROUND RESISTIVITY MAPPING

6.1 Introduction

This short note gives some illustrative examples of surveys that have been carried out using electromagnetic techniques to measure terrain resistivity. The instruments employed were the Geonics Limited EM31 and a prototype version of the Geonics Limited EM34. Both instruments were calibrated to read terrain conductivity in millimhos per meter directly; however in some of the case histories illustrated in this note the measurements have been converted to resistivity in ohm-meters.

Two features which often limit the usefulness of conventional ground resistivity surveys are their high cost and in some regions (granular material, frozen ground) difficulties associated with generating sufficient current in the ground. The use of inductive electromagnetic techniques avoids both problems since ground probes are not required. This allows measurement over any type of terrain and greatly reduces the time to perform a survey.

Basically the technique consists of energizing a small coil at an audio frequency and measuring the resultant total magnetic field from this coil and the ground with another coil a fixed distance away. Theoretical studies show that, if the intercoil spacing is maintained at a small fraction of the electrical skin depth in the ground, all of the information about the

6.1 Introduction (cont'd)

ground conductivity is in the quadrature-phase response. Furthermore, the quadrature-phase response is essentially linearly related to ground conductivity, thus permitting an instrument design in which the output is calibrated to read conductivity (or resistivity) directly. Also under these conditions, the effective depth penetration of the system is determined by the intercoil spacing and is independent of skin depth and thus of ground conductivity. This feature greatly simplifies interpretation of survey results. In order to vary the depth to which the resistivity is sensed one simply varies the intercoil spacing in analogy with conventional resistivity surveys. Conversely each survey carried out at a fixed intercoil spacing is essentially analogous to a survey carried out with conventional resistivity equipment at a fixed interprobe spacing.

The Geonics EM31 is a one-man portable instrument designed for engineering geophysical applications down to depths of the order of six meters. The intercoil spacing is fixed at twelve feet (3.66 meters). The effective penetration referred to above is an average value; in those regions where a conductive medium is to be located beneath a resistive layer the penetration depth is substantially larger.

The prototype EM34 is operated at two intercoil spacings vis 50 and 100 feet, resulting in effective depth penetrations of the order of 75 and 150 feet (25 to 50 meters) depending on the intercoil spacing employed for the particular survey. Operation of the EM34 requires two men; however measurements are still taken virtually as fast as the team walks.

6.1 Introduction (cont'd)

Most of the case histories in the technical note have been taken with the EM31; however in some cases data from EM34 surveys are presented in order to further elucidate the features of inductive electromagnetic terrain resistivity mapping.

6.2 Example A: Heart Lake, Ontario (EM31)
Measurement Interval: 100 feet over till,
10 feet over sand/gravel

This survey line compares the results obtained with conventional resistivity equipment (Wenner array with "a" spacings of 1 foot and 20 feet) and the Geonics EM31. It is seen that over the till, where the resistivity is slowly varying, the agreement between the two techniques is excellent. In the region shown as "sand and interbedded gravel" there was a good deal of concretion which caused the resistivity to vary greatly over short distances and which accounts for the discrepancy between the two techniques. Over the till the EM31 was read continuously however the data was recorded, with one exception, at every 100 feet. The exception occurred at station 7+50 where it was noted that a local resistivity high occurred; this was of course not observed on the Wenner array since measurements were taken only at every 100 feet.

6.3 Example B: Sunnybrook Park (EM31, EM34)
Measurement Interval: 100 feet

This case history shows measurements made with both the EM31 and the EM34 and illustrates the effective depth penetration of the two systems. The second sheet shows

6.3 Example B: (cont'd)

the results of expanding Wenner spreads at station 4+00 and 12+00. At station 4+00 we would expect the EM31 to read approximately 40 ohm-meters and the EM34 50 to 60 ohm-meters, which is the case. At station 12+00 the resistivity increases with depth and thus the EM31 should read a relatively low value and the EM34 a higher value which increases with intercoil spacing. This is indeed the case.

6.4 Example C: Cavendish Test Site (EM31)
Measurement Interval: 50 feet

This survey, which was carried out over line C to establish the overburden resistivity shows that with the exception of the swamp area the overburden is extremely resistive. The value of resistivity obtained over the swamp is in good agreement with that from other measurements. The example also illustrates the performance of the instrument over zones A & B, both of which are small highly conductive mineralized zones and cause the instrument to read off scale.

6.5 Example D: Lake Scugog (EM31)
Measurement Interval: 50 feet

This survey illustrates the extremely high resolution achievable with the EM31 or the EM34 systems since neither technique requires electrical contact with the ground. Resolution in conductivity of the order of 3% or 4% is easily achieved and completely repeatable as long as the terrain remains unchanged. The example also illustrates the speed with which a survey can be carried out. In this particular case 1.9 line miles of survey

6.5 Example D: (cont'd)

was performed in seventy minutes with a station interval of 50 feet. Furthermore since the measurements were actually taken continuously any unusual activity in the resistivity between stations would have been recorded.

6.6 Example E: Cooksville/Mississauga, Ontario (EM31)
Measurement Interval: 25 feet

This example illustrates a survey carried out with the EM31 to search for a buried river channel. The channel, which is filled with glacial till, has been cut into the Dundas shale which, as seen from the example, has a resistivity of the order of 25 to 30 ohm-meters. The total time to plot out the profiles shown in the figure was approximately 2½ hours, with a measurement interval of 25 feet. A shortcoming of the technique is seen on line 5, where a region was encountered which was so conductive that it was not possible to take readings.

The second sheet illustrates the application of the two layer curves to interpret the survey results in terms of depth.

6.7 Example F: Discontinuous Permafrost (EM31)
Measurement Interval: Variable

These two examples were taken in Northern Canada and compare the results obtained with the EM31 with a medium frequency version of the Radiohm (Geonics EM16R) technique operating at 250 kHz. The data interval is fairly coarse, nevertheless there is good agreement between the two techniques, which is particularly interesting in view of the fact that the current distribution in the ground

6.7 Example F: (cont'd)

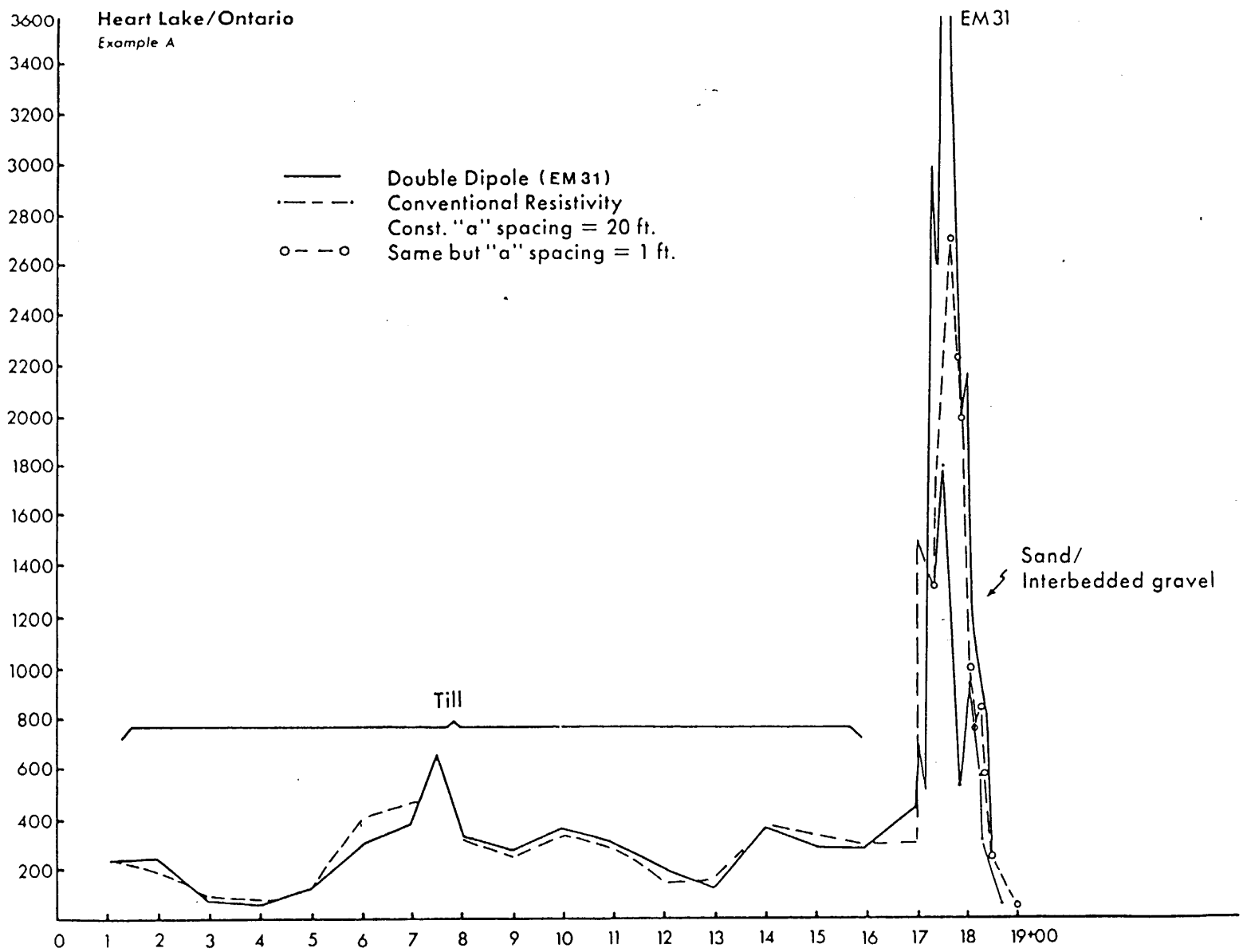
is totally different for the two systems. Furthermore the EM31 operates at a frequency of approximately 40 kHz whereas the MF Radiohm operates at 250 kHz.

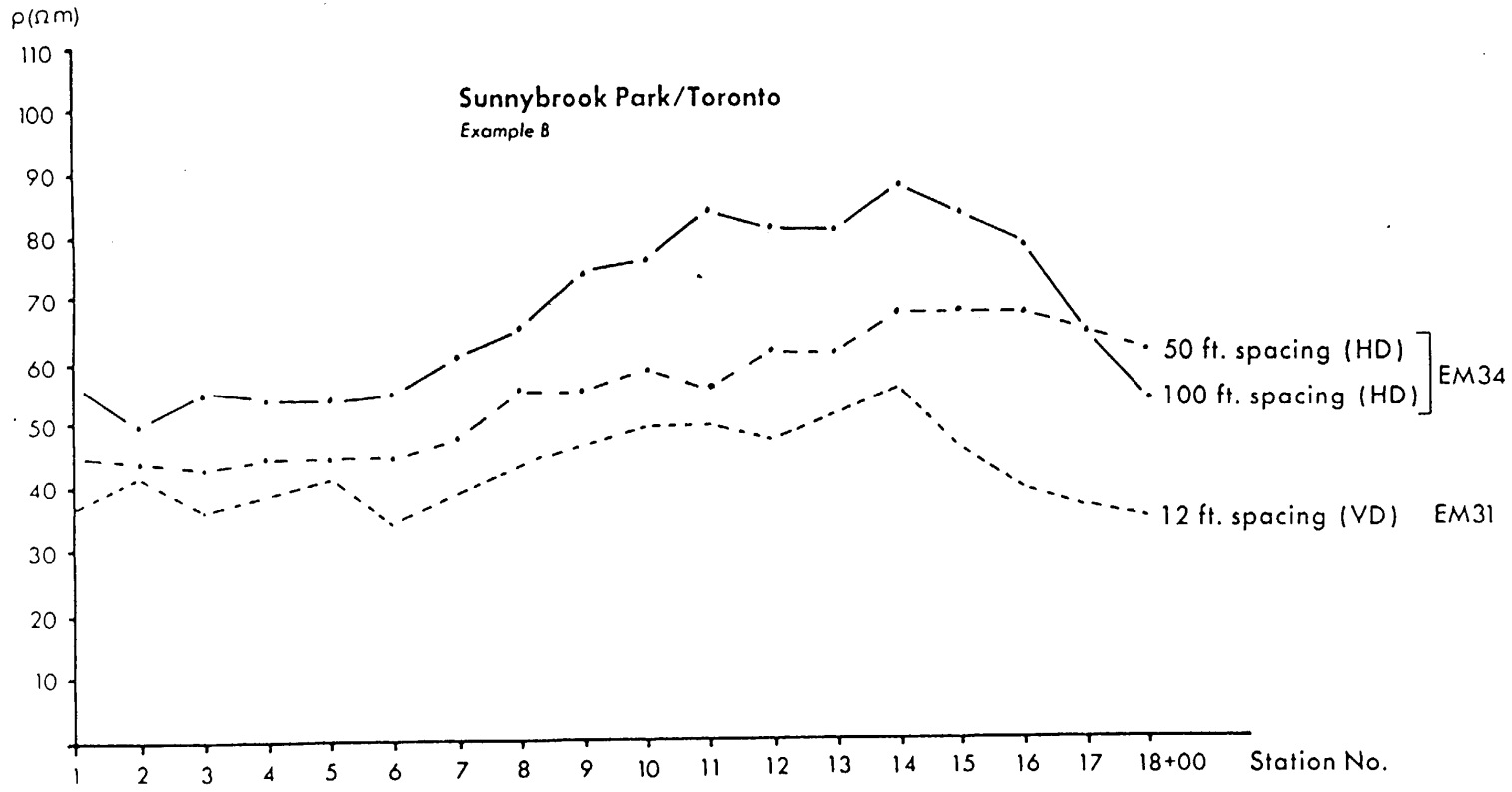
6.8 Example G: Pre-glacial River Valley (EM31, EM34)
Measurement Interval: 100 feet

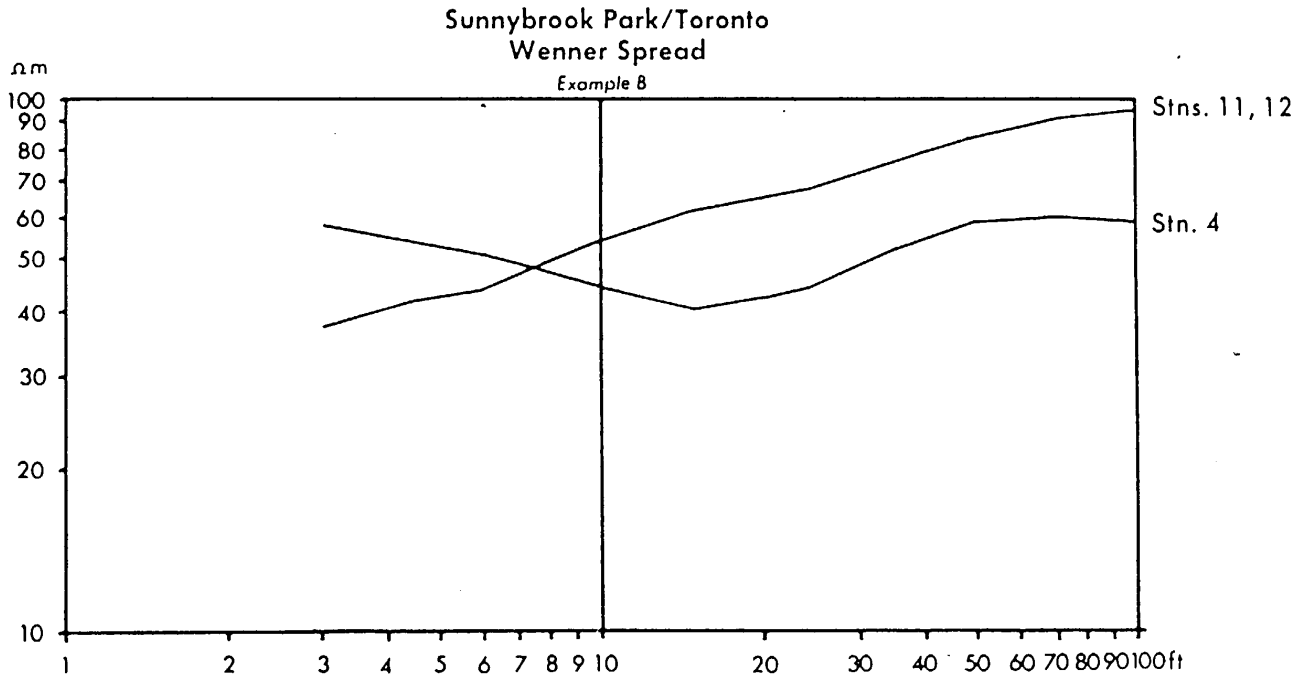
This survey was performed to outline a pre-glacial river valley whose existance had been suggested from water-well data. The survey was performed with the EM34 at a station interval of 100 feet and with intercoil spacing of both 50 and 100 feet. At either spacing the time required to complete the 8400 foot survey line was 1½ hours. The same line was subsequently surveyed with the EM31.

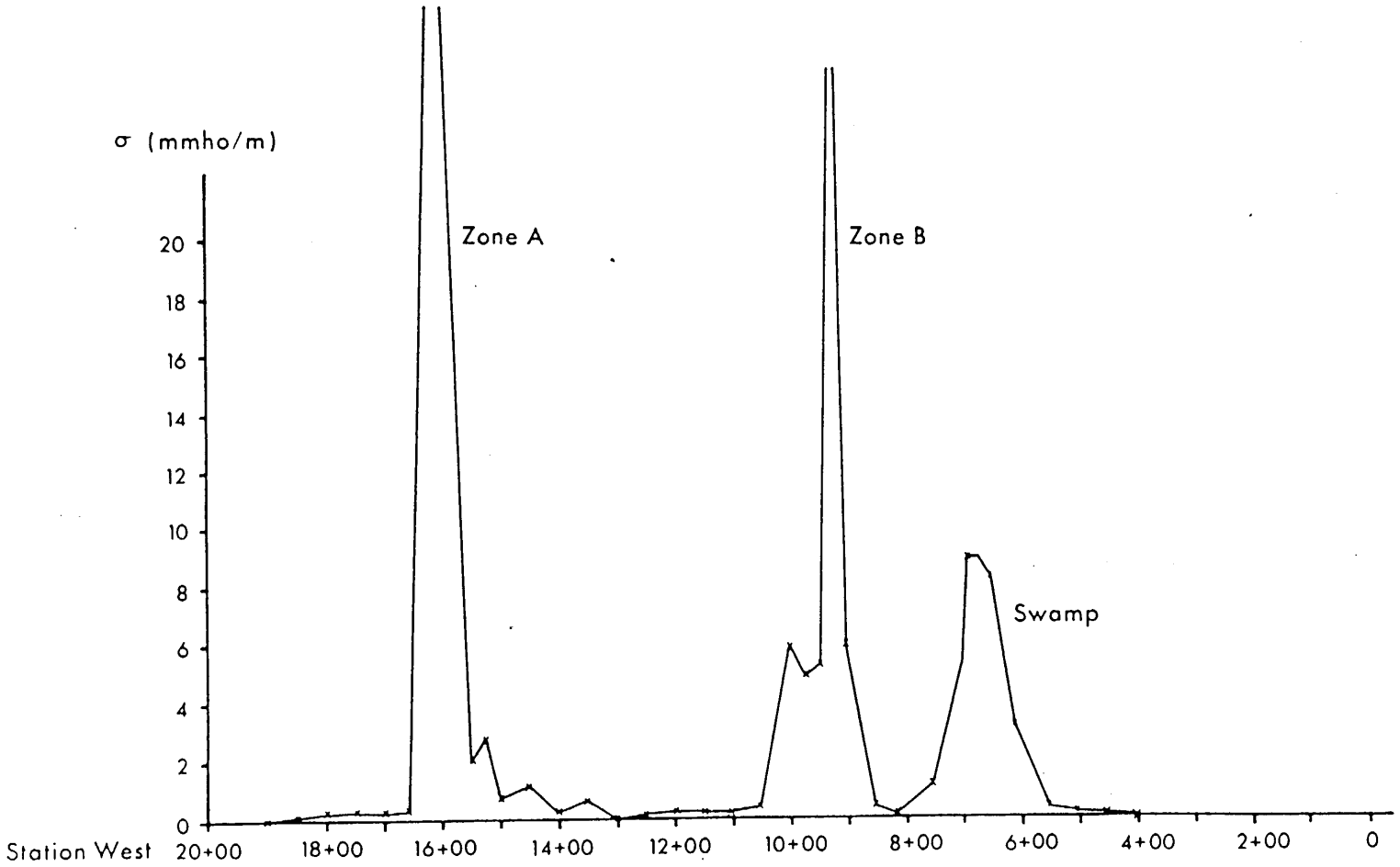
Typical bedrock conductivity in the area is approximately 32 millimhos per meter, whereas an average value for the conductivity of the infilling glacial till is of the order of 8 to 12 millimhos per meter. Thus the EM34 at either intercoil spacing yields approximately 30 to 34 millimhos per meter at the valley edges where the overburden is thin and 12 to 14 millimhos per meter at the valley center. The EM31 yields values of 14 to 18 millimhos per meter at the valley edges (slightly affected by the presence of bedrock) and approximately 10 millimhos per meter at the valley center. The interpreted depth of the valley, based on the model shown in the figure, is approximately 120 feet which is in reasonable agreement with the water-well data value of 150 feet, bearing in mind that the three sets of data show that a two layer model is an over simplification.

The conductivity high which occurs between station 32 and 38 results from a very large pile of waste furnace ash lying on the surface.


Heart Lake/Ontario
Example A
EM 31
Double Dipole (EM 31)
Conventional Resistivity
Const. "a" spacing = 20 ft.
Same but "a" spacing = 1 ft.
Till
Sand/
Interbedded gravel
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19+00


ρ(Ωm)
110
100
90
80
70
60
50
40
30
20
10
Sunnybrook Park/Toronto
Example B
50 ft. spacing (HD)
100 ft. spacing (HD)
EM34
12 ft. spacing (VD)
EM31
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18+00
Station No.


Sunnybrook Park/Toronto
Wenner Spread
Example 8
Ωm
100
90
80
70
60
50
40
30
20
10
1
2
3
4
5
6
7
8
9
10
20
30
40
50
60
70
80
90
100
ft
Stns. 11, 12
Stn. 4

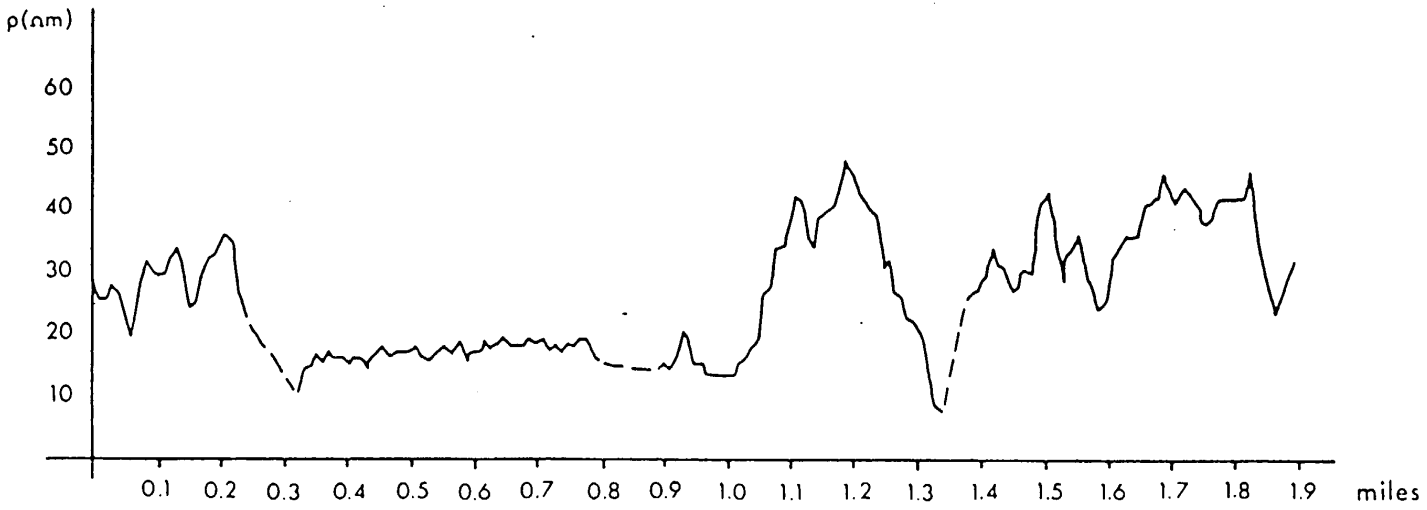


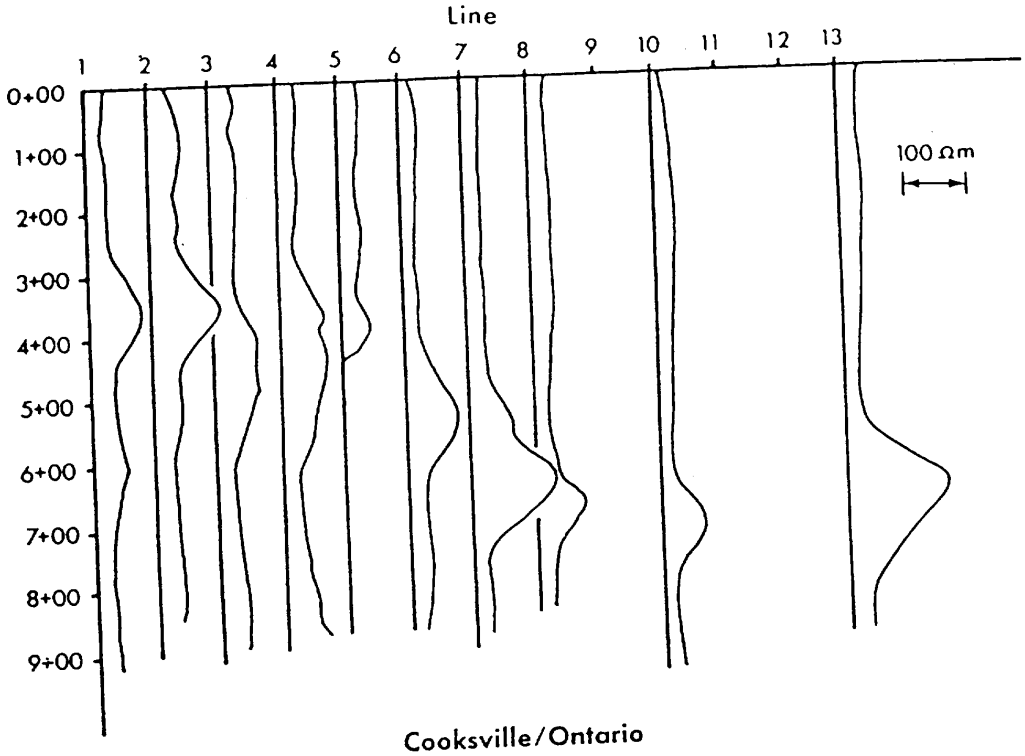
Cavendish Test Range — Line C

Example C

Lake Scugog/East Causeway *Example D*

Note (1) Station interval = 0.01 mile
(2) Survey duration =70 minutes
(3) Total no. of stations = 190
(4) 2.7 stations per minute

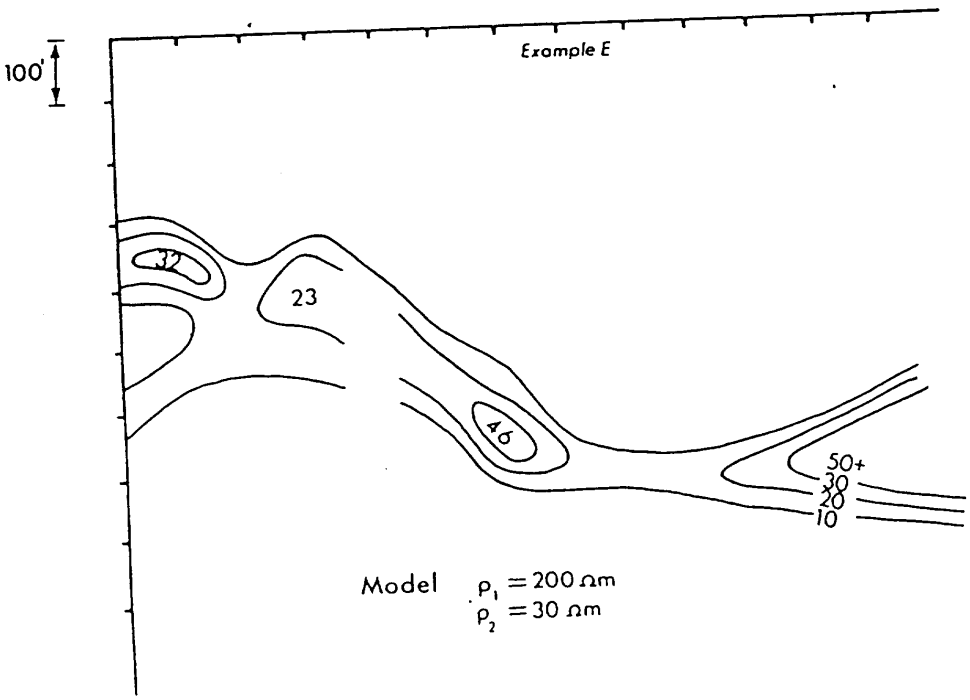




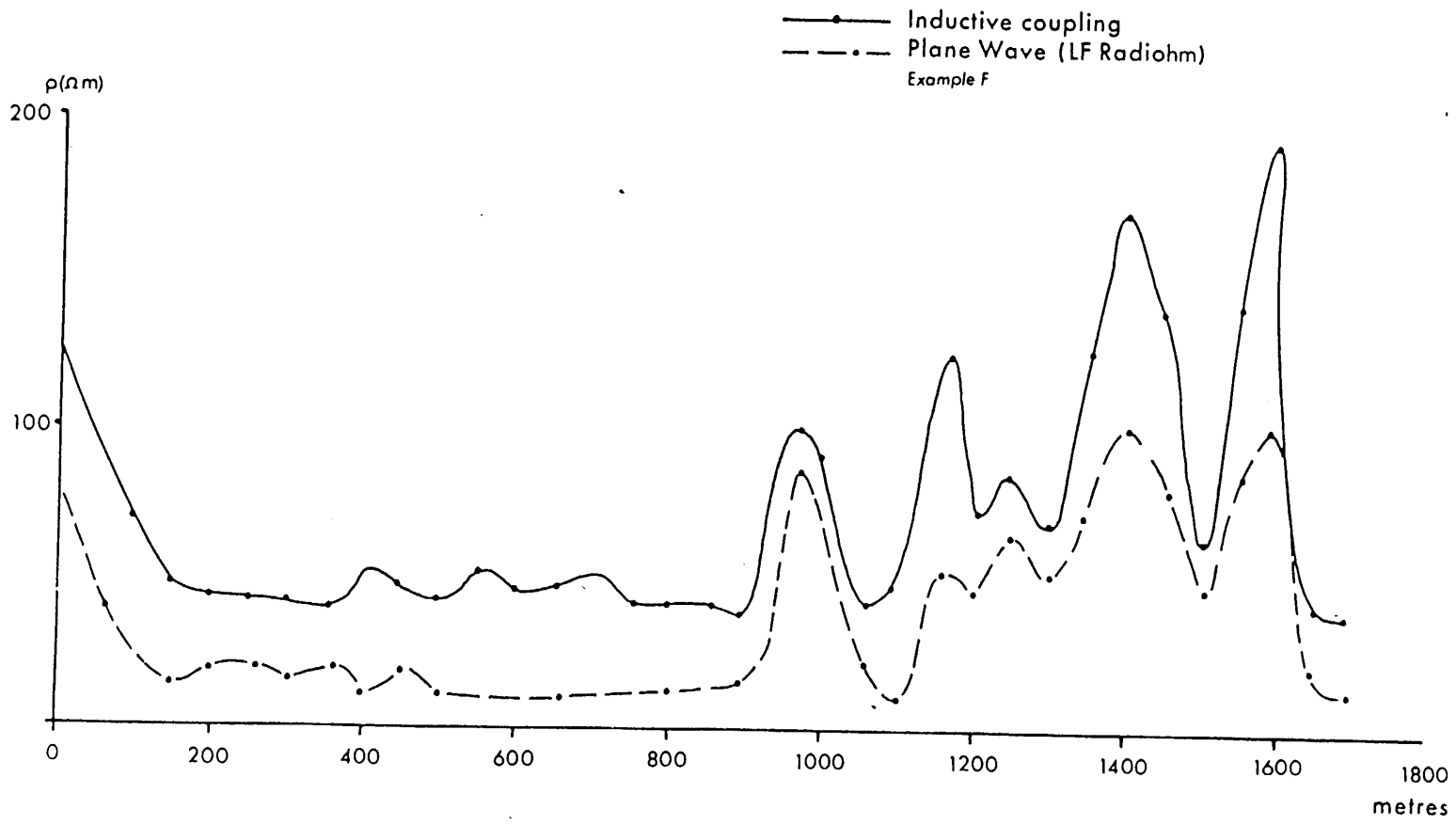
Cooksville/Ontario

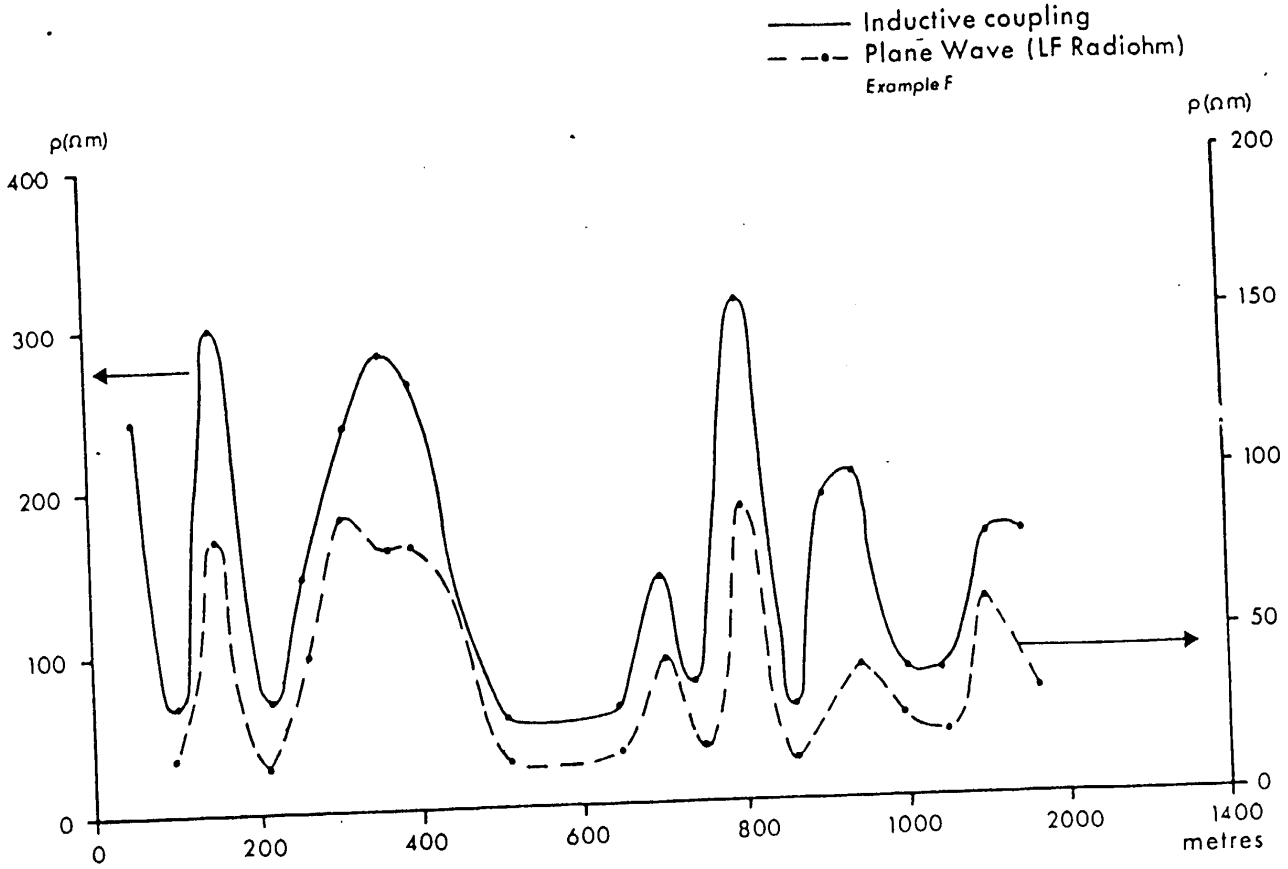
Example E

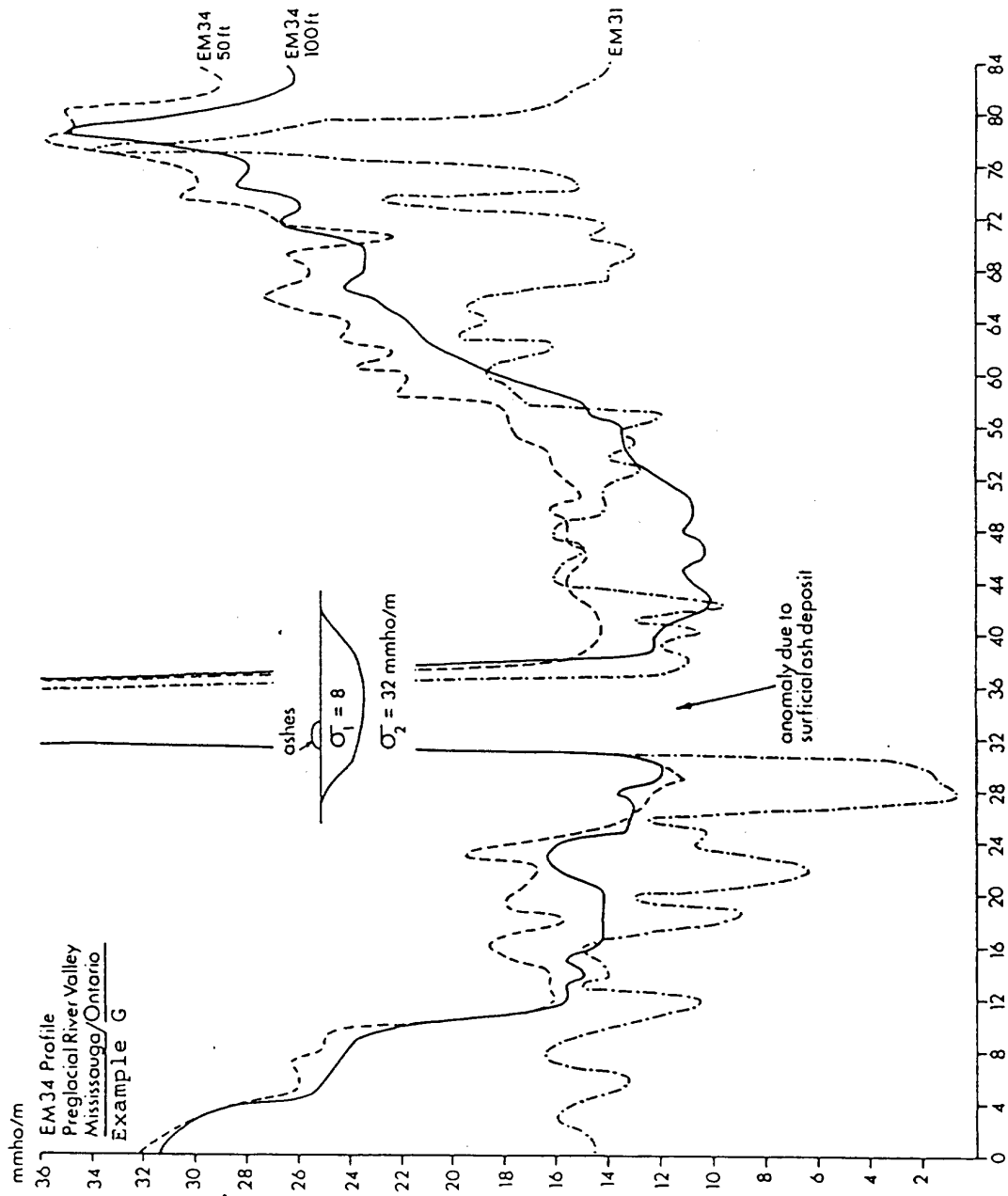
Contoured Depth



Model $\rho_1 = 200\ \Omega m$
$\rho_2 = 30\ \Omega m$


Inductive coupling
Plane Wave (LF Radiohm)
Example F
ρ(Ω m)
200
100
0
200
400
600
800
1000
1200
1400
1600
1800
metres


Inductive coupling
Plane Wave (LF Radiohm)
Example F
ρ(Ωm)
400
300
200
100
0
ρ(Ωm)
200
150
100
50
0
0
200
400
600
800
1000
2000
1400
metres


EM34 Profile
Preglacial River Valley
Mississauga/Ontario
Example G
mmho/m
EM34 50ft
EM34 100ft
EM31
ashes
σ1 = 8
σ2 = 32 mmho/m
anomaly due to surficial ash deposit

APPENDIX I - Formulae for Geometrical Sounding of Two-Layered Earth

In the case of a two-layered earth the apparent conductivity is given by:

$$\frac{\sigma_a}{\sigma_1} = 1 - R + kR = R(k - 1) + 1 \qquad (1)$$

with the instrument on the ground in its normal operating position, and

$$\frac{\sigma_a'}{\sigma_1} = 1 - R' + kR' = R'(k - 1) + 1 \quad , \qquad (2)$$

with the instrument on the ground on its side.

* Case 1. Therefore

$$\frac{R'}{R} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1} \qquad (3)$$

and from the first and/or second equations, with $k = \sigma_2 / \sigma_1$

$$\sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R} = \frac{\sigma_a' - \sigma_1 + R'\sigma_1}{R'} \qquad (4)$$

* Case 2. When the conductivity of the lower layer is known, from equations (1) and (2)

$$\sigma_a' = \sigma_1 - R'\sigma_1 + R'\sigma_2 \qquad (5)$$

APPENDIX I - Cont'd

Case 2. (cont'd)

$$\sigma_a = \sigma_1 - R\sigma_1 + R\sigma_2 \tag{6}$$

$$\sigma_a' - \sigma_2 = \sigma_1 - R'\sigma_1 + R'\sigma_2 - \sigma_2 \tag{7}$$

$$= (1 - R')(\sigma_1 - \sigma_2) \tag{8}$$

$$\sigma_a - \sigma_2 = (1 - R)(\sigma_1 - \sigma_2) \tag{9}$$

$$\frac{1 - R'}{1 - R} = \frac{\frac{\sigma_a'}{\sigma_2} - 1}{\frac{\sigma_a}{\sigma_2} - 1} \tag{10}$$

$$\sigma_1 = \frac{\sigma_a - \sigma_2 R}{1 - R} = \frac{\sigma_a' - \sigma_2 R'}{1 - R'} \tag{11}$$

Case 3. When the upper layer is resistive and σ_2 is much greater then σ_1, from equations (5) and (6)

$$\sigma_a \simeq \sigma_2 R \tag{12}$$

$$\sigma_a' \simeq \sigma_2 R' \tag{13}$$

$$\frac{\sigma_a'}{\sigma_2} = \frac{R'}{R} \tag{14}$$

and

$$\sigma_2 = \frac{\sigma_a}{R} = \frac{\sigma_a'}{R'} \tag{15}$$

APPENDIX I - Cont'd

*Case 4. When the upper layer is conductive and σ_2 is much less than σ_1, from equations (5) and (6)

$$\sigma_a \approx \sigma_1(1 - R) \tag{16}$$

$$\sigma_a' \approx \sigma_1(1 - R') \tag{17}$$

$$\frac{1 - R'}{1 - R} = \frac{\sigma_a'}{\sigma_a} \tag{18}$$

$$\sigma_1 = \frac{\sigma_a}{1 - R} = \frac{\sigma_a'}{1 - R'} \tag{19}$$

APPENDIX II - Determination of Two-Layered Earth Geometry by Varying Instrument Height

The electrical conductivity of the earth often exhibits horizontal layering and it is possible with the EM31 to determine how closely this layering can be approximated by a two-layered earth geometry.

Suppose measurements made with the instrument on the ground have yielded different values of apparent conductivity when the instrument meter is (a) face-up (i.e. the instrument is in normal position), and (b) vertical (i.e. the instrument is lying on its side). As discussed in Section 5.3 had the value of apparent conductivity been the same in both positions we would know that the earth was homogeneous, but the fact that the values are different for the two positions shows that the electrical conductivity varies with depth. We would now like to know whether the earth is two-layered.

To determine this we make measurements of the apparent conductivity with the instrument at various heights above the ground, as measured by the distance between the ground and the nearest point of the white tube. It is suggested that measurements be made at increments at least as small as one-half meter, for an example at 0, 0.5, 1, and 1.5 meters. Measurements made at smaller increments will yield greater accuracy, as will measurements made up to 2 meters height if possible. Measurements should be made both with the instrument in its normal position (meter face upright, i.e. vertical dipoles,) and with the instrument on its side (meter face vertical, horizontal dipoles) since it will be seen from the curves that the fall-off with height for these two configurations is quite different and that this difference is therefore of considerable diagnostic value in determining the two-layered earth characteristic. It is important to make the measurements of height and apparent conductivity as accurately as possible.

APPENDIX II - Cont'd

The curves in the two figures show the variation of apparent conductivity (divided by upper layer conductivity) for instrument height varying from 0 to 2 meters and for five values of upper layer thickness vis. 0.5, 1.0, 1.5, 2.0, and 3.0 meters. The relationship is shown for both vertical dipoles (meter face up, instrument in normal position) and horizontal dipoles (meter face vertical, instrument on its side) for various values of upper layer to lower layer conductivity contrast.

To use the curves one proceeds as follows:

(1) Place a piece of semi-transparent paper over either one of the graphs and on it plot the measured values of apparent conductivity versus instrument height to the scale used on the figures. Also sketch in on the transparent paper at any point a horizontal or vertical line to insure that when the transparent paper is translated over the figures in step 2 it does not rotate.

(2) Translate the transparent paper vertically and horizontally (i.e. without rotation) across the various plots shown on the figures to see whether the data points can be matched to one of the curves. In the event that no satisfactory match is achieved the earth cannot be represented by a two-layered case.

(3) If a satisfactory match can be achieved, the parameters of the two-layered earth are immediately calculated as follows: suppose that the measured values of apparent conductivity with height are

Height =	0.0	0.5	1.0	1.5	2.0 meters
σ_a (EM31 upright)	20.7	20.7	19.3	17.1	14.1 mmho/m
σ'_a (EM31 on side)	26.0	19.0	14.3	11.2	9.0 mmho/m

APPENDIX II - Cont'd

Upon plotting these data values to the scale of the figures and translating them we see that best agreement is achieved for an upper layer thickness of 1.5 meters and a conductivity contrast $k = 0.5$. We further observe that the value of 20.7 millimhos per meter for σ_a (instrument upright) corresponds to a value of σ_a / σ_1 of 0.62.

Since $\frac{\sigma_a}{\sigma_1} = 0.62$ when $\sigma_a = 20.7$

$$\therefore \sigma_1 = \frac{20.7}{0.62} = \underline{33.4} \text{ mmho/m}$$

Since $k = \frac{\sigma_2}{\sigma_1} = 0.5$

$$\therefore \sigma_2 = 0.5 \times 33.4 = \underline{16.7} \text{ mmho/m}$$

and $t = \underline{1.5\text{m}}$ from the figure.

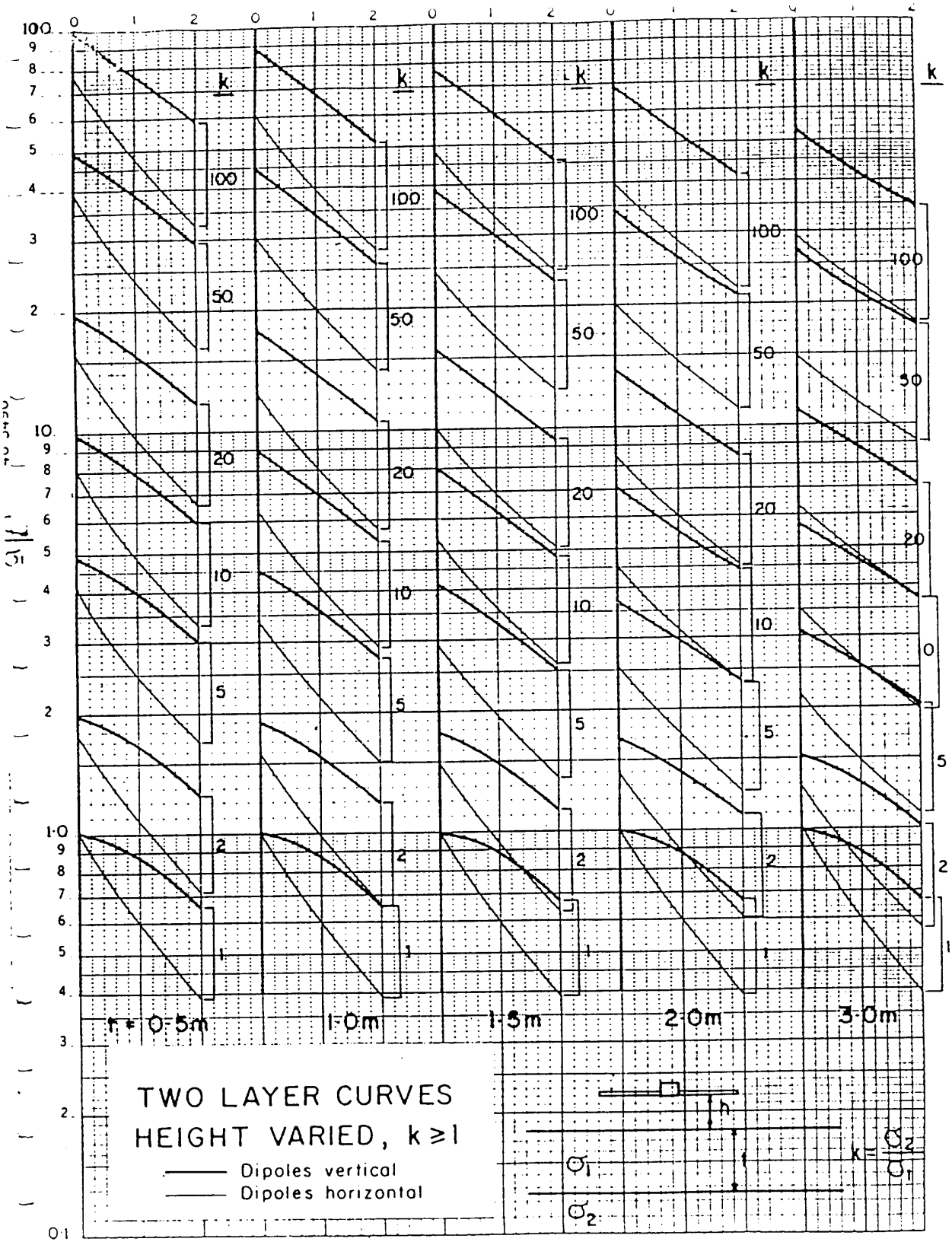
The two-layered earth is fully resolved.

It will be observed that in some regions on these two figures the resolution or differentiation between the various curves is not very great. For example if one examines the curves for $t = 1.0$ meters, and $k = 100$, 50, or 20, one sees that these curve pairs are almost identical. If the process described in step 3 is carried out for each of these conductivity contrasts the result of the calculations will show that the lower layer conductivity and the upper layer thickness are the same, regardless of which of the three curve pairs are used, and that the only calculated parameter that will vary will be the upper layer conductivity which is very small. This is an example of the well-known property of electromagnetic systems to accurately give both the distance to a good conductor and

APPENDIX II - Cont'd

the actual conductivity of the conductor but to poorly define the intervening resistive material. A problem in unambiguously matching the curves may also occur for larger values of upper layer thickness, where the thickness is sufficiently great to prevent accurate resolution of its value.

These features notwithstanding it will be found that these curves are useful in (a) deciding whether the ground resembles a two-layer case and if so (b) giving a reasonably accurate estimate of the electrical parameters.


TWO LAYER CURVES
HEIGHT VARIED, k ≥ 1
Dipoles vertical
Dipoles horizontal
t = 0·5m
1·0m
1·5m
2·0m
3·0m
k
h
t
σ1
σ2
k = σ2/σ1

Appendix A-A2

Dual Tube Reverse Circulation Air Rotary Drilling Methods

DUAL-WALL REVERSE CIRCULATION OFFERS.

HOW IT WORKS

The reverse circulation rotary drilling method utilizes dual-wall pipe, top drive rotation and a side inlet for injecting the drilling fluid, air or both.

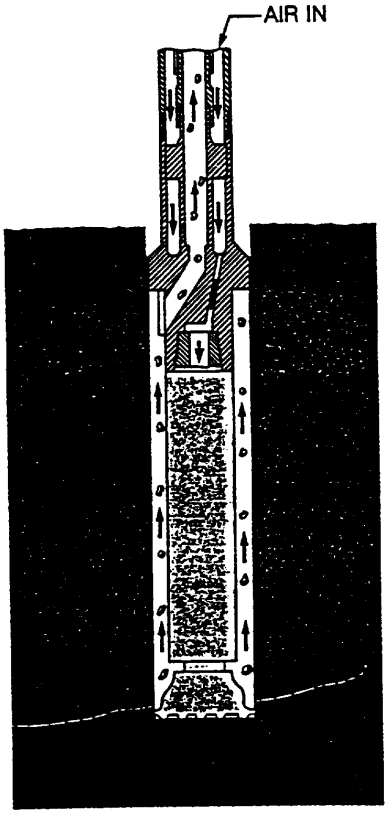
When drilling with this system, the drilling fluid is forced down the outer annulus of the dual-wall pipe to the drill bit where it is then directed to the center of the pipe. The air or drilling fluid returns the cuttings via the inner pipe at velocities in excess of 4,000 feet per minute.

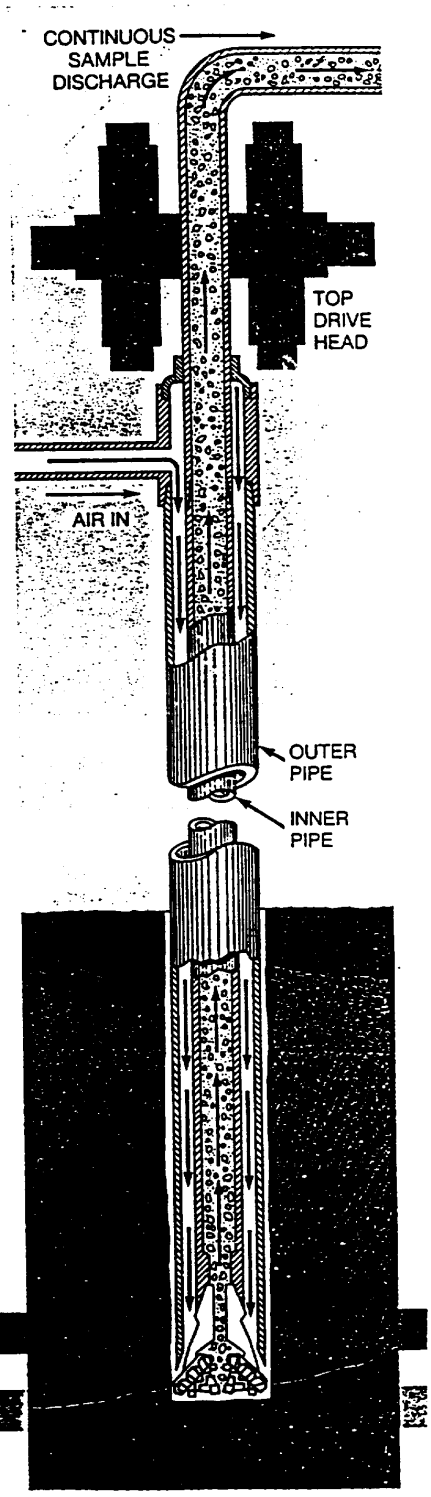
The reverse circulation rotary system uses flush-jointed drill pipe and a drill bit sub designed to fit snugly over the body of the drill bit. This unique design permits the bore hole to be cut with a minimum of clearance and so lessens the possibility of sample contamination. This configuration also minimizes the loss of air or misting in vuggy or fractured formations and will maximize the geological sample recovery in adverse conditions; conditions that are nearly impossible for rotary or diamond drills.

FOR HARD ROCK FORMATIONS

When it becomes uneconomical to drill in hard formations with either a tricone or roller button bit, the dual-wall method can still be used to advantage with a down-the-hole hammer drill.

An interchange sub is screwed onto the top of the hammer drill and then the complete unit is screwed onto the dual-wall pipe. In this configuration, the rotating/percussion action of the drill cuts the sample which travels between the wall of the hole and the outer barrel of the hammer drill. As the sample moves up the length of the hammer drill it is directed into the interchange sub openings where it is air lifted to the surface through the inner pipe.





EFFICIENCY, VERSATILITY AND ECONOMY

HOW LAYNE MAKES IT WORK FOR YOU

Whether you call it the double-wall pipe system, rotary continuous sample system or the dual-wall system, the results are the same . . . Layne can provide you with a faster, less costly way to obtain the purest exploration samples possible.

Uncontaminated Sampling
Since rock and water samples do not come in contact with the bore hole, there is no contamination from the formation or erosion. In extreme high water bearing formations, down hole erosion may occur due to fractures or gravel formations.

Faster Penetration
With top head drive rotary drills, exploration proceeds up to 15 times faster than with conventional core drills.

Lower Operating Costs
In some cases, costs have been reduced by 75%. Bit costs are generally lower and the job is completed in less drilling time. In addition, expensive drilling muds or additives are rarely used.

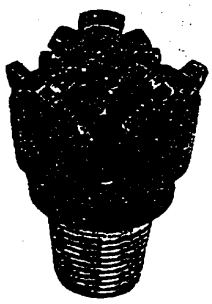
Straighter Holes
Flush-wall pipe with minimum clearance results in less deviation than with conventional drilling methods.

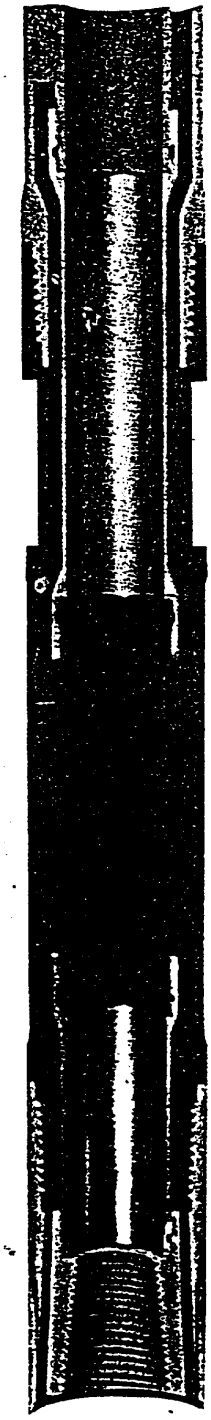
No Surface Casing
The outer pipe supports the bore hole while circulation is maintained internally.

No Lost Circulation
Circulation is maintained even when drilling in vugs, fractures, voids and joints thus avoiding expensive delays.

Improved Well Design
Better samples provide greater reliability in well design, enhancing results.

In-Place Water Sampling
Many geologic environments are suitable for obtaining reliable water samples from aquifer zones during drilling operations, eliminating the need for expensive packers or other methods of obtaining such samples.





TYPICAL SCENARIO
FOR DUAL-WALL REVERSE CIRCULATION
DRILLING AND BORING PROGRAM

The general purpose for completing the soil boring is to provide detailed characterization of subsurface hdyrogeologic conditions within the surficial unconsolidated aquifer materials (soils above bedrock) and, at some locations, characterization of the bedrock surface. Specific program objectives include the following:

1. Obtain continuous soil samples for purposes of characterizing and classifying the stratigraphy of the unconsolidated soil materials overlying bedrock.

2. Determine the depth to bedrock and obtain samples of the underlying bedrock.

3. Obtain additional stratigraphic information for comparative purposes by gamma logging.

4. Obtain representative ground water samples for contamination field screening (by headspace GC analysis) at specified depths within the saturated soil column.

The selection of the appropriate drilling method to meet these four objectives has been made with consideration of the following constraints:

1. The drilling method must permit the collection of representative ground water samples at specified depth intervals for field laboratory screening for selected compounds. Therefore, the method must NOT introduce foreign liquids into the soil near the cutting edge of the drill stem, and must provide sufficient working area within the drill casing to lower a small pump for purging ground water or a bailer or similar device for the collection of a representative ground water sample.

2. The method must be capable of installing temporary casing to isolate separate aquifers if encountered during the drilling process. To be cost and time efficient, it should not be necessary to change drilling rigs or get involved in long changeover times with one rig to install this temporary casing. Implied in this constraint is the ability of the drilling method to continue to drill through the temporary casing.

3. Upon reaching bedrock in the boring, the method must be capable of obtaining a core sample at least ten (10) feet in length of the bedrock. Again, cost and time efficiencies demand that one rig with a minimal changeover time be used.

4. Since there exists some possibility for encountering contaminated soils during drilling, the method must control any solids and fluids removed from the boring.

After long consideration and evaluation of various drilling methods, and other options for the collection of subsurface samples, a procedure for drilling these borings has been identified. This procedure meets all of the defined objectives and operates within the constraints imposed by those objectives.

The selected method is an air-rotary technique known as the Dual-Tube Reverse Circulation Method. This method consists of a water-tight steel casing or outer tube of approximately 4.5 inches in diameter. Within this outer tube there is an inner tube of 2.4 inches internal diameter. This inner tube is connected to the outer tube at specified intervals, leaving an annular space between the dual tubes. The lead casing of the inner tubes uses either a tri-cone rotary bit or an open-faced toothed bit for cutting soil materials. The relative positions of the two tubes are such that the leading edge of the bit is at the same depth or lags slightly behind the leading edge of the outer tube. High velocity compressed air, which is injected down the annulus formed by the two tubes and through the bit, carries cuttings to the surface via the inner tube. Cuttings are conveyed to a cyclone where the air velocity is reduced, the air is discharged out the top and the solids (soils) fall out the bottom. The high velocity of the air stream returns cuttings from depth to the surface with little discernable delay. When it is necessary to provide permanent casing within a boring to either isolate formations or to provide a cavity within which a monitoring well can be constructed, the dual-tube method requires the use of a separate casing, known as the overshot pipe.

The overshot pipe is steel with an inside diameter of approximately five (5) inches. To install the overshot pipe, high pressure water and/or air and hydraulic pressure are used to jet the overshot around the dual-tube. At some sites, it can be anticipated that a confining layer separating water bearing units will be found at some depth. If contamination exists in the overlying water-bearing unit, it will be necessary to isolate the upper portion of the boring from the contamination so that contamination cannot migrate through the boring to the lower water-bearing unit as drilling proceeds. This would be accomplished by first using the dual-tube to drill to the confining layer. The overshot pipe would be advanced to the depth of the confining layer and a seal would be made. Dual-tube methods would then be used to continue the boring through the confining layer and into the underlying formation(s).

The dual-tube string, as mentioned above, has an inside diameter of 2.4 inches. This cavity is continuous from the top of casing through the bit if the open-faced toothed bit is used as the cutting tool. If coring of bedrock is necessary, it can be accomplished through the dual-tube string (with open-faced bit) using AQ coring equipment. AQ coring equipment has an outside diameter of 1.89 inches and yields a core with an outside diameter of 1.062 inches.

This description of the dual-tube method and equipment makes it clear as to why it is the selected method for the soil borings. The dual-tube method provides continuous soil samples (although not for headspace screening purposes), formation isolation capabilities and bedrock coring all from the same drilling rig. Other, less obvious advantages can also be found in the dual-tube method. These include:

1. Air discharge from the cyclone, if contaminated, can be directed well away from the work area with the use of a hose connected to the top of the cyclone.

2. Soil discharge from the cyclone can be directed directly into a container, thereby controlling contamination (when present), and keeping the work area clean.

3. Representative ground water samples can be collected from within the dual-tube.

4. No foreign liquids are used during drilling.

5. The method is very fast; at other locations in similar soils, 100-foot soil borings have been completed in a period of three hours or less.

The disadvantages of the dual-tube method are no more restrictive than any other drilling method. Heaving liquified soils are always a problem when they enter the drill casing, and no drilling method can deal with them well. The only time soil heave is likely to occur is when the air-flow is shut off to allow sampling of ground water. Soil heave in this situation is not a problem provided that it is recognized that the first cuttings returning when the air-flow is resumed are those that have heaved into the casing. Another potential disadvantage of using the dual-tube as described above involves the need for the open-faced drill bit when bedrock coring will be required. Boulders approximately one-foot in diameter can "seat" themselves directly on the bit, making it virtually impossible to drill through. The tri-cone rotary bit will be used on all borings that will not be coring bedrock.

SAMPLING
WITH THE
DUAL-TUBE METHOD

Ground Water Sampling by Dual Tube Drilling

Using this method eliminates cross-contamination

By Lori Huntoon Pencak

One of the most challenging tasks facing the ground water professional is evaluating the vertical distribution of subsurface contamination. Dual tube drilling methods allow the collection of ground water samples with depth during drilling, and minimize cross-contamination while providing site-specific information within the borehole.

Collecting ground water samples with depth is useful not only in detecting the contamination, but also as a design tool for monitoring systems. Multiple monitoring nests are often installed in the initial phase of a hydrogeologic investigation in an attempt to evaluate the distribution of vertical contamination.

A common problem which arises is the improper vertical placement of screened intervals, due to lack of existing site information. One approach to designing a monitor well network is the collection of ground water samples with depth during well installation.

The use of screened hollow stem augers has proven to be very useful in this type of ground water sampling. However, the utility of the dual tube drilling method is quickly being understood as an extremely efficient and cost-effective method for determining specific aquifer information from the collection of ground water samples with depth.

Dual Tube Methods

Dual tube drilling, also called reverse circulation drilling and center sampling recovery, provides the collection of continuous representative samples from a borehole with virtually no cross-contamination of the samples. In addition to the collection of geological samples, ground water samples also can be collected from the borehole during drilling operations in order to determine differences in contaminant concentrations with depth.

This is possible due to the unique design of the drilling system. Dual tube drilling uses rotary drilling methods, including either a rotary bit or down-the-hole hammer to advance through the formation. The flush-threaded, dual-walled pipe is joined to the bit by a drill bit sub, and both are advanced simultaneously (see figure 1).

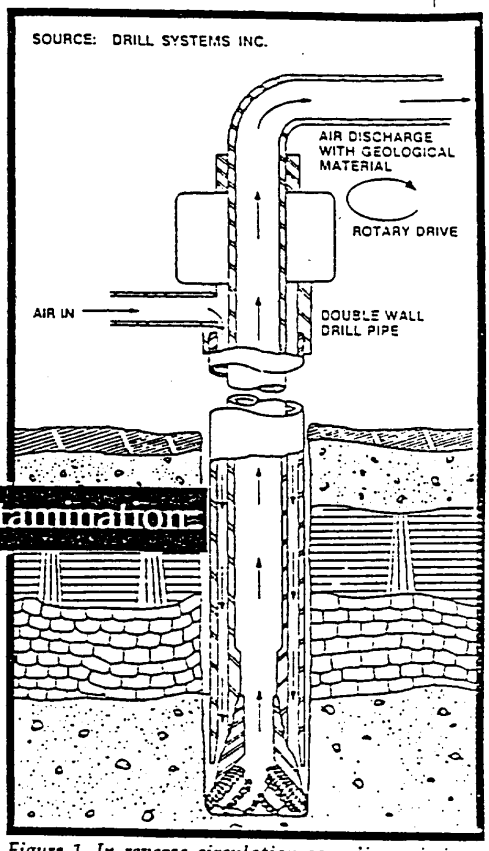


Figure 1. In reverse circulation sampling, air is injected down the outer annulus of dual-walled pipe, causing air and samples to flow up the inner pipe.

The drilling fluid, preferably air or mist, is forced down the outer annulus of the dual-walled pipe, and directed to the center of the pipe. The size of the borehole is such that there is a minimal clearance between the bit and the drilling pipe, which minimizes cross-contamination along the annulus of the borehole.

The cuttings are discharged through the inner annulus to the surface within fractions of a second, allowing immediate determination of the lithology. It also is important to note that formation

(Continued on next page)

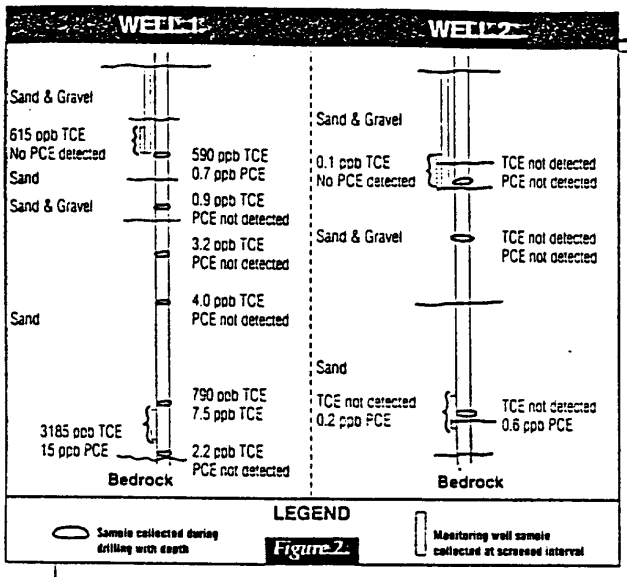


Figure 2.

cuttings and ground water samples don't come into contact with the overlying borehole materials.

During drilling, ground water samples can be collected at selected intervals as the borehole is advanced. Due to the design of the system, ground water is allowed to enter only through the open bottom of the borehole. Thus, the sample collected is representative of the actual ground water in the formation at the open interval.

Samples can be collected by lowering a pump intake line or a bailer through the inner tube of the drilling column. Several volumes of water may be removed prior to collection in order to insure a representative sample.

When using a bailer, lower and raise the bailer slowly with a smooth motion to avoid volatilization of potential contaminants. When using a pump, don't allow the intake line to extend to the bottom of the drilling tools — rather, place the line several feet above the bottom.

If possible, use a photoionization meter during drilling and sampling procedures, and screen the samples prior to submitting them to the laboratory. It's helpful to the lab if you can estimate whether the sample will have high or low detects of contaminants. High levels of contaminants when not expected can result in laboratory equipment being shut down for days in order to decontaminate the machines.

Samples can be collected in VOA vials and immediately analyzed by gas chromatography methods in the field, and/or transported to a laboratory for analysis. It's best to collect at least two vials for each sample, to allow for sample breakage and unexpected analytical duplicates. Additional samples can be collected for field screening, indicator parameters (pH, specific conductance, temperature) and additional laboratory analyses.

Properly label the sample bottles with the well number, sample depth, date and time. Samples should be immediately stored on ice and warmed to room temperature prior to analysis.

During the collection of ground water samples, stringent decontamination procedures should be followed. These include:

- Washing the sampling equipment with laboratory-grade detergent and several rinses with distilled water between each sample.
- Replacing the rope or tubing after each sample collection.
- Using gloves when handling the sampling equipment or bottles.

It's important to note that in fine-grained formations, bridging of sand in the drilling tools may not allow the collection of a ground water sample.

Case Study

A subsurface investigation was completed to determine the source

Do's and Dont's for Collecting Samples When Using Dual Tube Drilling

- DO decontaminate the sampling equipment thoroughly between each sample.
- DO replace the bailer rope or plastic tubing between each sample.
- DON'T surge the bailer up and down. Lower and raise the bailer into the well slowly with a smooth motion to ensure a representative sample and to avoid volatilization of potential contaminants.
- DO collect at least two vials for each sample to allow for sample bottle breakage and unexpected analytical duplicates.
- DO use a field ionization meter to screen the samples before submitting them to the laboratory. It will help if you can estimate if the sample will have high or low detects of contaminants.
- DO label the samples immediately after collection. Place the samples on ice, complete the necessary chain of custody records and transport the samples to the laboratory as soon as possible.
- DO use cross-section maps in addition to tables to evaluate the results received from the use of vertical sampling with depth during drilling. ❑

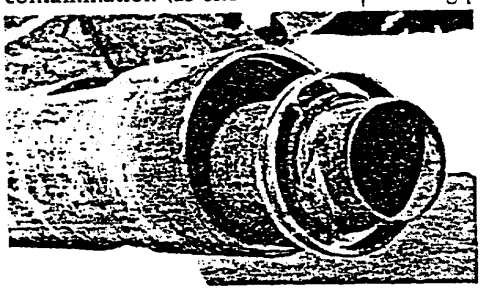
and extent of a trichloroethylene (TCE) ground water contaminant plume. Several deep monitoring wells were installed at the site, using the dual tube method.

Water quality samples were collected at 20 foot intervals to a depth of 150 feet in order to determine the variations in ground water quality with depth.

Concentrations of contaminants were detected at two distinct depths within the aquifer, which may have been missed during the installation of a standard monitoring network due to improper vertical placement of screened intervals. Figure 2 shows the results of the samples which were collected from two of these wells during drilling.

The contaminant plumes detected during the investigation may have been missed during the installation of a standard monitoring network due to improper placement of screened intervals. Figure 3 shows the hypothetical placement of a screen in coarse material overlying bedrock.

However, in this case, the contaminant plume wasn't located directly above the bedrock, but rather tens of feet above it. The installation of a screen in the coarse material would indicate a lack of contamination (as shown in the collection of the sample at depth in figure 2).



An end section of reverse circulation drill pipe.

In addition, concentrations of the contaminant in between these plume areas were minimal or nonexistent, insuring that cross-contamination between the samples was minimized.

Evaluating the vertical distribution of contamination with depth is critical in determining the appropriate screen depths of monitoring wells. The collection and analysis of ground water samples with selected depths during drilling provides the water professional with a definition of ground water quality with vertical distance.

And the most efficient and cost-effective method of collecting ground water samples with depth is through the dual tube drilling method, which allows collection of the samples at selected depths while minimizing the chance of cross-contamination. ❑

Lori Huntoon Pencak is staff hydrogeologist — project development for Geraghty & Miller Inc., Milwaukee, WI.

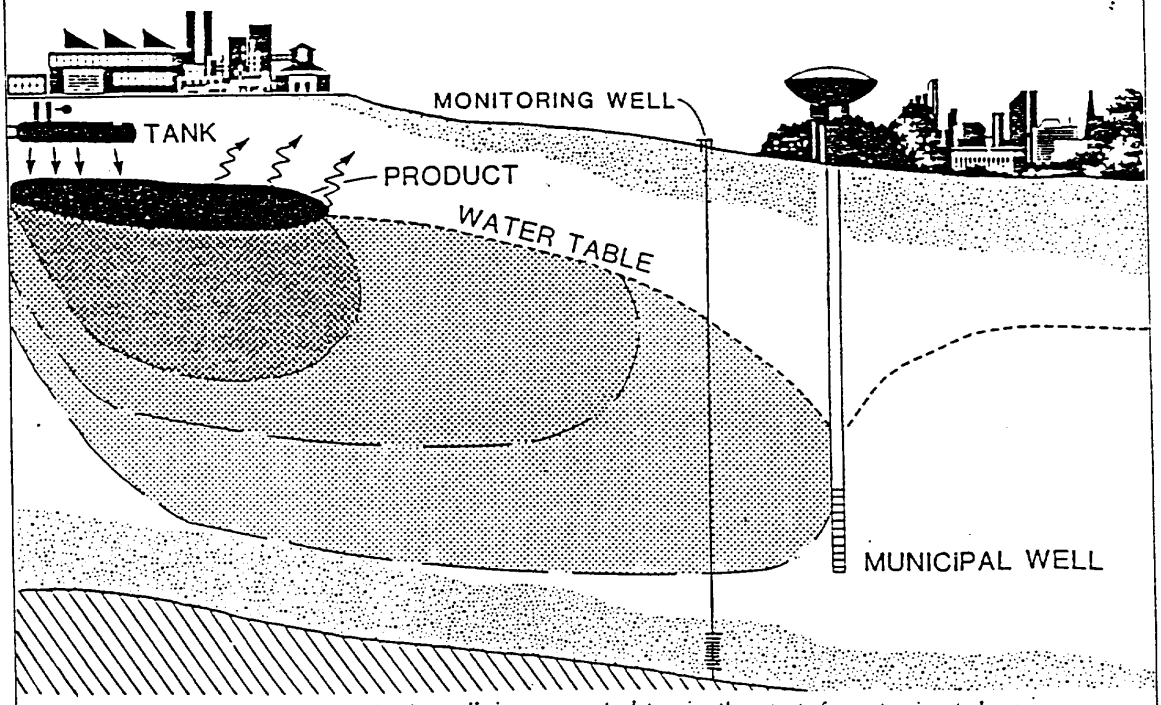


Figure 3. Specific placement of monitoring wells is necessary to determine the extent of a contaminant plume.

Appendix A-A3

Operator's Manual for the OMNI IV Magnetometer

EDA

OPERATION MANUAL

OMNI IV

TIE-LINE MAGNETOMETER

PPX-402 Printed in Canada 30.06.85

4123/31.03.85RO/M9A

WARRANTY

EDA Instruments Inc. warrants that at the time of shipment the goods shall be free from defect in material and workmanship. Notice of any defect must be given to EDA Instruments Inc. in writing immediately upon discovery of any such defect and must be within the time of the warranty stated in the face hereof or if none stated within 1 year from the date of shipment of the goods by the purchaser. EDA Instruments Inc. obligations under this warranty are limited to the repair or replacement of the parts or materials which upon examination by EDA Instruments Inc. are established to be defective within this warranty and in no event shall the corporation's obligations under this warranty exceed the cost of the goods sold. For the purposes of repairing or replacing parts or material under this warranty the purchaser shall forward at its own risk and freight prepaid the goods to EDA Instruments Inc. This warranty ceases to apply to any goods altered or repaired by anyone other than EDA Instruments Inc or its authorized representative.

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EDA Instruments Inc., reserves the right to null and void the warranty if and when it can be determined by specially trained personnel or an authorized agent that the instrument has been misused.

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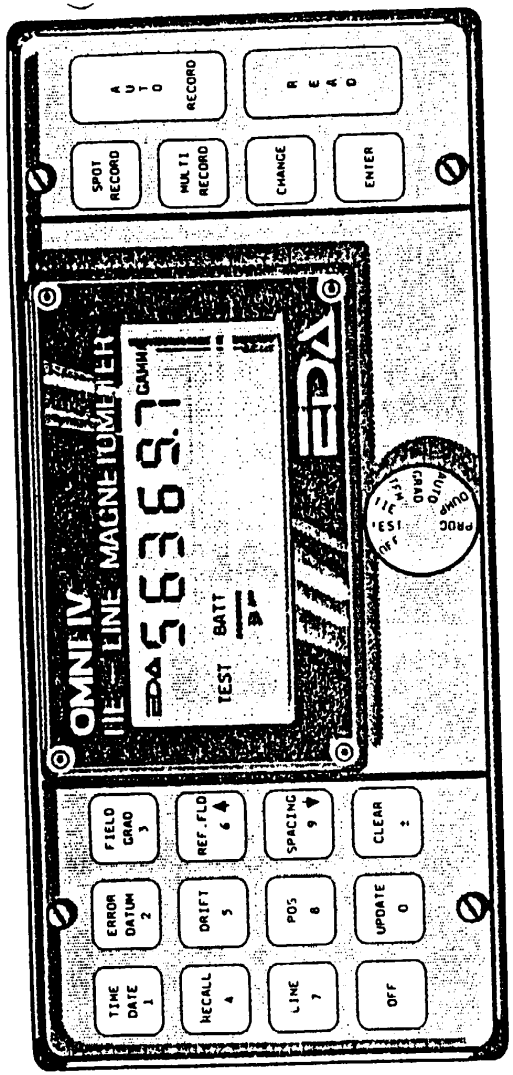


Figure 1-1 OMNI IV Tie-Line Magnetometer

SECTION 1

GENERAL INFORMATION

1.1 SCOPE OF MANUAL

This manual describes the OMNI IV Tie-Line Magnetometer designed and manufactured by EDA Instruments Inc., Toronto, Canada. The information is provided for personnel responsible for operation of the unit and should be read prior to operation and testing to obtain a full understanding of all of the features incorporated. Technical Specifications are given in Table 1-1.

1.2 PURPOSE OF INSTRUMENT

The OMNI IV is a portable, microprocessor-based total field magnetometer system which is capable of correcting its own field survey data for diurnal variations without the need of a base station. A measurement can be read and stored in only three seconds. The data is highly repeatable. A second reading is not usually required.

The OMNI IV is a multi-purpose instrument designed to operate in four different functions or modes:

a. Tie-line magnetometer (3).

b. Portable field magnetometer (3).

c. Recording base station magnetometer (4).

d. Gradiometer (5).

Software. The instrument function (mode) is predetermined by the software that is resident in EPROMs inside the instrument. The software cannot be changed by the operator. The level of the software installed by the manufacturer is identifiable on the instrument readout display as part of the serial number. The procedure for obtaining the software level display is described in the operation section. The software level identification number is shown in ( ) in a. through d. above. Software with a higher identification number also includes the software for the lower numbers.

The primary purpose of this microprocessor-based instrument is to:

- measure and store the total earth's magnetic field;
- identify the location of that measurement by recording either the grid coordinates or a record number;
- record the time that the measurement was made.

In addition, the OMNI IV can correct its own field measurements for diurnal variations without the need of a base station, by utilizing either the tie-line or looping point method of survey. However, the instrument stores only the raw data; it does not store corrected data. Corrected data is fed to the instrument output connector during the data dump mode for recording in a peripheral computer or printer. The raw data is retained until the instrument is re-initialized even after corrected data has been computed.

Data stored in memory is completely protected by a lithium battery. This battery also powers the real-time clock.

Field Measurement Accuracy. The following design features eliminate the need for taking multiple readings of poor instrument performance:

- Patented signal processing technique.
- Constant energy polarization that maintains equal energy to the sensor for each measurement.
- Processing sensitivity to within ±0.02 gamma.
- Automatic fine tuning which uses the previous reading as the basis for the next reading.

Choice of Function. The choices are categorized in four functions associated with the resident software. However in each category, there are also numerous feature choices, based on operator preference.

TIE-LINE MAGNETOMETER. In this mode, the OMNI IV is able to store raw data obtained using either the looping survey or tie-line survey method. This reference data is stored in a separate memory at the beginning of each survey. Then total field readings are taken and stored in another memory together with the repeat field readings at the tie-points. At the end of each survey day, the data in the two memories are merged so that the total field data is corrected for diurnal variations automatically. These corrected values are outputted from the instrument immediately (ie, not stored).

In the tie-line mode, the OMNI IV can store looping or tie-line data in any one of the following ways:

a. Using one base point for reference in the looping survey method.

b. Using one tie line consisting of a number of tie-points for reference in the tie-line survey method.

c. Using multiple tie lines for reference in the tie-line survey method.

d. Using a random scattering of tie-points as reference.

The flexibility offered allows the operator to choose the most appropriate tie-line method best suited for the survey, depending upon such factors as the size and character of the survey grid. For smaller survey areas the looping method is practical but for larger areas, tie-line references become necessary. The tie-line algorithms are based on a linear diurnal variation between adjacent tie points. Therefore the accuracy of the corrected data is a function of the time lapse between measurements at adjacent tie-points and any erratic behaviour of the diurnal variation during that same period. Typically, the time lapse should be less than 30 minutes. Using the multiple tie-line feature, the requirement for base station magnetometer recordings can be eliminated from some surveys.

Up to 100 tie points can be stored in one survey area. Alternatively, these 100 tie points can be divided into extensions of survey areas as needed. The tie point and tie-line measurements are stored for the duration of the survey. Tie-line data can be recalled even if stored on different days. However, raw field data can only be entered daily during a midnight-to-midnight period.

The OMNI IV can also calculate the drift between established tie points so that variations in the earth's magnetic field can be seen readily. Similarly, the OMNI IV can also calculate the desired diurnal drift measured between consecutive tie point readings.

In the OMNI IV software, there is a programmable datum. A datum (eg. 57,000 γ) can be entered into the instrument. Then, when a reading is taken, this datum, which is a coarse background value, is subtracted from the reading. This provides a small value of the differences to facilitate plotting and interpretation of data.

The tie-line capability is also applicable when the instrument is used as a gradiometer. The operator can therefore obtain 'corrected' total field data using the linear diurnal variation algorithm without relating to base station magnetometer readings.

PORTABLE FIELD MAGNETOMETER. In this mode, the OMNI IV measures and stores the total field magnitude; time of measurement; grid coordinates; and direction of travel in the survey grid. It also computes and stores the statistical reading error, the signal strength and the rate of decay. The memory can store up to 1400 data blocks thereby eliminating the need to record data manually. Accumulated data can be dumped to printers, to magnetic cassette recorders or in parallel with a base station magnetometer to a computer.

The operator has a choice of three data storage modes:

- Spot record. Each reading is assigned a record number automatically. This can be used to identify readings which are taken at locations which do not correspond to survey grid coordinates.
- Multi record. More than one reading can be taken at a survey grid coordinate without updating the current station or line number.
- Auto record. These readings are taken automatically based on a preprogrammed time interval.

The direction of travel is recorded using the primary cardinal points (N, S, E, W) and by increments in the position indication.

The statistical error is calculated for each measurement based on an internal algorithm. The result of the calculation is displayed to provide the operator with an accuracy factor. From this it may be determined whether or not an additional reading will be required.

Similar to the tie-line mode, the following features can be selected:

- If the last reading is unwanted, it can be cleared.
- The difference between the current reading and previous reading is calculated automatically and can be displayed upon push-button command.
- A preprogrammed datum or coarse magnetic field value can be subtracted from each field data measurement to simplify plotting of field survey results.
- Diurnal corrections are calculated automatically.

BASE STATION MAGNETOMETER. In the base station configuration, the OMNI IV measures and stores in its memory the diurnal variations in the earth's magnetic field. The instrument is designed specifically to meet base station specifications for airborne and ground surveys. Readings are taken automatically based on an interval which is programmed by the operator. The interval can be varied from 5 seconds to 60 minutes in 1 second increments. The memory has a capacity of 5000 data blocks. This provides a 7 hour capacity at the 5 second frequency.

The data that is being stored can be outputted simultaneously in a digital or ASCII format to a choice of data collection systems.

When the base station is connected in parallel with the compatible field magnetometer, it can correct and dump the total field measurements. The corrections made are for diurnal variations and reference field values.

The magnetic field activity is recorded in the following format:

- Time of measurement.
- Total field magnitude.
- Difference from the reference field value.
- Difference from the previous reading.
- Sequential record number.

The OMNI IV can also be programmed with a reference field value (eg, 57,000 γ). This 'bulk' value is then subtracted from each reading so that only a small but significant value (which is the variation between readings) is recorded.

GRADIOMETER. When the OMNI IV is programmed for use as a gradiometer, a dual sensor staff is connected. The sensors are incorporated in a staff with the sensors spaced vertically 0.5 m apart. The OMNI IV reads and stores the measurements of both sensors simultaneously, thereby providing a true measure of the total field and the gradient of the total field. Simultaneous readings remove the effects of diurnal variations and magnetic storm interferences from the data.

The stored data includes:

- The gradient of the total field.
- The total magnetic field measured by the upper sensor.
- The time of measurement.
- The survey grid coodinates where the measurement was made.
- The statistical error of total field reading made by the lower sensor.
- The signal strength and decay rate measurement of the lower sensor.

Using the tie-line method, or in conjunction with an OMNI IV base station, the total field measurements made by the upper sensor can be corrected for diurnal variation. Because the sensor readings are simultaneous, diurnal magnetic variations of the gradient measurements are cancelled.

In the gradient mode, the OMNI IV has improved resolution of total field anomalies. Therefore it defines more sharply the magnetic responses determined by total field data. Closely spaced anomalies are delineated individually rather than being identified collectively as one broad magnetic response. There is also direct delineation of vertical contacts.

Therefore, the OMNI IV is an ideal contact mapping tool especially in vertical or near vertical contacts or fault zones. These vertical contacts are depicted at the zero line of gradient contours or profile values. Vertical bodies analogous to a dyke can also be mapped effectively. The OMNI IV is also effective in enhancing anomalies near the surface. Any shallow source exhibits a higher frequency anomaly than a deeper low-frequency anomaly. This comparison can provide an on-the-spot approximation of the depth of the anomalous source. The capability of the OMNI IV to differentiate between higher and lower frequency responses removes background regional gradients from anomalous residual responses.

The OMNI IV can be used in a combined gradient and base station operation. It can be programmed to cycle automatically every 5 seconds in the gradient mode. This feature can be used in stationary or mobile applications.

The OMNI IV sensors are mounted in a sectional aluminum staff in which sections can be added or subtracted. Similarly, there are optional separations of the sensors of 0.5 m or 1.0 m. These can be used to obtain interpretive information especially for surface anomalous conditions (eg, determining if the field is linear or curved).

1.3 FUNCTIONAL DESCRIPTION

The OMNI IV Tie-Line Magnetometer is a ruggedized compact portable instrument designed specifically for field operation. The instrument is extremely accurate and flexible. It contains a microprocessor and associated circuitry (CPU) for monitoring, storing and processing data. Basically, the OMNI IV has two memories: One for storing data taken at a base reference point and along a tie line; the other for storing field measurements of the actual survey.

The actual survey points would comprise an imaginary grid covering the survey area. In the tie-line application, one or more of those grid lines (or even selected grid points) are established as the tie line (or tie point) references. Measurements of the earth's magnetic field are made, usually at the beginning of a survey, at each selected tie point. Then, field measurements are made at the survey grid intersection points including subsequent (ie, repeat) readings at the tie-line points. Variations in the readings taken at a tie-line point provides a reading of the diurnal variation experienced at that point. These are used as the basis for computing the corrections for readings at adjacent survey points. In this computation for correction of readings, the variation between two readings at a tie point (ie, the diurnal variation) is compared with the variation in the two readings at the previous tie point. The times that these variations were recorded are also recorded. Therefore, the diurnal variation associated with any intermediate survey point between two tie points can be computed with extreme accuracy confidence and the correction can be applied to the reading taken. This computation is carried out within the instrument automatically without operator assistance.

The algorithm assumes that the diurnal variation is linear with respect to time. This is usually valid if the interval between two readings at the same point is short (ie, less than 30 minutes). It may be less accurate during periods of increased activity. For critical measurements, the base station method provides greater accuracy due to the time interval factor; however, the inaccuracy is a function of the distance from the base station to the point of measurement.

The instrument performs all of the following functions initiated by pressure-sensitive push-button commands:

a. It provides a visual readout of the total magnetic field amplitude. When the instrument is used as a gradiometer, it also provides a visual readout of the gradient of the total field.

b. It provides a visual readout of the time and date of a measurement upon push-button command.

c. It provides a visual readout of the statistical error associated with the total field measurement upon push-button command.

d. It assigns the grid coordinates of a measurement in terms of line and position numbers in the survey grid.

e. It provides incremental update of the position and line numbers along a line including changes to the line and position spacing increments at any time.

f. It provides the drift or difference in gammas between the previous and current field reading.

g. It records all of the parameters, a through g, in memory by selecting one of three record keys.

h. It assigns a record number to each total magnetic field measurement. This can be used instead of grid coordinates.

Physical Dimensions	Wt(kg):	w × h × d(mm)
Instrument console only	2.8:	238 × 150 × 250
Battery belt (Alkaline)	1.2:	540 × 100 × 40
Battery cartridge (Lead acid)	1.8:	235 × 105 × 90
Battery belt (Lead acid)	1.8:	540 × 100 × 40
Sensors		
Remote sensor	1.2:	56 dia × 220
Gradient sensor		
0.5 m separation (standard)	2.1:	56 dia × 790
1.0 m separation (optional)	2.2:	56 dia × 1290

Environment

Electronics	
Operating temperature range	−40°C to +55°C
Relative humidity	0 to 100% (weather-proof)
Sensor	
Temperature range	−45°C to +55°C
Relative humidity	0 to 100% (weather-proof)

Standard Memory Capacity

Total field or gradient	1400 data blocks or sets of readings
Tie-line points	100 data blocks or sets of readings
Base station	5000 data blocks or sets of readings

Electronics

RS-232C serial I/O	2400 baud; 8 data bits; 2 stop bits; no parity
Electronic console	Enclosure contains electronics and battery pack (if not contained in separate belt). Front panel includes liquid crystal display (LCD), keypad and MODE selector.
Power supply	Internal battery pack or external battery belt; or +12 V car battery if used as a base station.

Table 1-1 Technical Summary

Specifications

Dynamic range	18,000 to 110,000 γ. Roll-over display suppresses first significant digit upon exceeding 100,000 γ.
Processing sensitivity (total field)	±0.02 γ
Statistical error resolution	0.01 γ
Absolute accuracy	±1 γ at 50,000 γ at 23°C. ±2 γ over total temperature range.
Display resolution (total field)	0.1 γ
Tuning method	Microprocessor calculates precise tuning. Value utilizing a specially-developed timing algorithm.
Automatic fine tuning	±15% relative to ambient field of last stored total field value.
Tracking range	18,000 to 110,000 γ
Gradient tolerance	6,000 γ per metre (field proven)
Tuning indicator	Descriptor on display (TUNE) commences to flash on and off. Audio alarm activated also. (New field value must be entered into system.)
Display	Custom designed, ruggedized liquid crystal display with an operating temperature range of −40°C to +55°C at 100% RH. The display contains six numeric digits, two decimal points, battery monitor, signal decay rate and signal amplitude monitor and function descriptors.

Table 1-1 Technical Summary (Cont)

SECTION 2

PHYSICAL DESCRIPTION

2.1 SYSTEM COMPLEMENT

The instrument can be assembled in three configurations, with variations depending upon the type of sensor selected, the preferred power package and peripherals.

The configuration complements are given in Table 2-1.

STANDARD SYSTEM (Software Code 3). The basic configuration is for use in total field, tie-line and looping applications. It comprises:

a. A console which is carried on the chest and operated in that position. (The console includes a 1400 data block memory for field magnetometer applications.)

b. A shoulder harness to support the console comfortably so that both hands are free.

c. A remote sensor which is complete with a 3 m cable for attachment to the console.

d. A sectionalized pole assembly (four, 60 cm sections) which supports the sensor.

e. A rechargeable battery cartridge.

f. This system also includes a plug-in battery charger for use with 110 Vac or 220 Vac circuits, and an operation manual.

BASE STATION CAPABILITY (Software Code 4). This option comprises a standard system plus the following:

a. A standard console with a 1400 data block memory for field magnetometer applications and an enhancement of a 5000 data block memory for base station applications.

b. A 28 m base station cable extension so that the sensor can be located remote from the console (eg, console located in a shelter).

c. A rope joiner. This joiner provides attachment points for securing the sensor and pole upright using non-metallic guy lines.

BASE STATION AND GRADIENT CAPABILITIES (Software Code 5). This option is the same as the standard configuration except for the following enhancements:

a. A standard configuration with a 1400 data block memory for field readings and an enhancement of a 5000 data block memory when in the base station mode.

b. The remote sensor is replaced by a dual sensor with the sensors spaced 0.5 m apart. Optionally, the sensors can be spaced 1.0 m apart.

System Peripherals and Accessories. Each of the configurations can be used with the peripherals listed in Table 2-1. An optional transit case is also available as an accessory for the console, sensor, sensor pole, battery pack and battery charger.

2.2 COMPONENT DESCRIPTION

Standard Instrument with Remote Sensor. The electronics console is carried on the chest with the instrument panel facing upwards. The console is supported in a dual shoulder-type harness. The sensor is mounted on a sectional pole, 2.5 m long and connected to the console through a flexible cable. It is hand carried similar to a staff.

Instrument with Gradient Sensor. The electronics console is carried on the chest with the instrument panel facing upwards. The console is supported in a dual shoulder-type harness. The gradient sensor comprises two sensors mounted vertically on the hand-held pole and separated by a distance of 0.5 m (standard) or 1 m (optional). This permits simultaneous measurements from each sensor.

INSTRUMENTS CONSOLE. The primary electronics, data acquisition circuit, microprocessor and memories are built into a rectangular, aluminum, weather-proof case with the instrument panel facing upwards.

Display. Data and information is displayed on a custom-designed, ruggedized, liquid crystal display (LCD) which operates in temperatures ranging from −40°C to +55°C. The display includes a six-numeric digit readout, decimal point, battery status monitor, signal decay rate and signal amplitude monitor, and function descriptors. The internal heater is activated automatically at −25°C during the survey. Make sure that the console MODE selector is set to OFF overnight if the temperature is expected to be below −25°C. The OFF selection turns off the display heater.

Operator Keys. The operator keys are grouped into two keypads located on each side of the LCD. The 12 keys on the left hand side are for programming the instrument. The 6 keys on the right hand side are for taking measurements and recording them. The key functions are described in Section 3.

	Standard OMNI IV	OMNI IV with Base Station Capability	OMNI IV with Base Station & Gradiometer Capabilities
OMNI IV Console	✓	✓	✓
Memory (1400 data blocks)	✓	✓	✓
Memory (5000 data blocks)		✓	✓
Remote Sensor PPX-326R	✓	✓	
Gradient Sensor (0.5 m separation) PPX-528			✓
Gradient Sensor (1.0 m separation) PPX-			Optional
Pole Assembly (4 × 60 cm sections) PPX-360	✓	✓	✓
Cable Extension, 28 m PPX-436		Optional	
Rope Joiner (guy rope attachment) PPX-457		✓	
Battery Belt PPX-	✓	✓	
Battery Cartridge (rechargeable, sealed, lead acid) PPX-365	Optional	Optional	✓
Battery Charger 110/220 Vac PPX-255	Optional	Optional	✓
Operation Manual PPX-402	✓	✓	✓

Table 2-1 OMNI IV System Complements

Options and Accessories:

Thermal Printer (AC/DC)
40 character
DCU-400

Thermal Printer (AC only)
40 character
DCU-040

Dot Matrix Printer (AC only)
80 character
DCU-080

Paper, Thermal Printer
DCX-441

Paper, 80 column fan fold
DCX-445

Communications Interface Adapter
DCA-100

Interconnect cable (data dump) for single OMNI IV
DCX-442

Interconnect cable (data correction to TRS80/100), male
(1-020-00335)

Interconnect cable (data correction to printer), male
DCX-443

Interconnect cable (data correction to Heathkit H-14 printer), female
(1-020-00330)

Interconnect cable (data correction to HP-85 option 0), male
DCX-446 (1-020-00335)
Note: Female RS-232 serial interface connector.

Interconnect cable (data correction to HP-85 option 1), female
DCX-446 (1-020-00330)
Note: Male RS-232 serial interface connector.

Interconnect cable (data correction to IBM PC), female
DCX-447 (1-020-00330)

Table 2-1 OMNI IV System Complements (Cont)

Cable Connectors. There are two cable connectors located on the rear of the instrument (ie, remote from the control panel). When the console is being used (ie, chest mounted):

- The one on the operator's left side connects to the sensor.
- The one on the operator's right side is for data dumping. It is an RS-232C serial input-output connector. The interface parameters are: 2400 baud; 8 data bits; 2 stop bits; no parity.

REMOTE SENSOR. The sensor consists of two helical coils of copper wire connected in series in a noise-cancelling mode with at least 50 dB attenuation of external noise. The coils are immersed in a hydrocarbon-rich liquid inside a light-weight, leak-proof cylinder. The sensor cylinder is mounted inside a thin-wall fiberglas tube. The coils are positioned with their axes parallel to each other. The interconnections are carried through a cable, 3 m long and terminated in a connector which interfaces with a connector on the rear of the OMNI IV.

Remote Sensor Pole. This sensor pole consists of a sensing head mounted on a pole. The pole consists of four short rods which engage end to end so that the sensor is approximately 2.5 m above the ground.

Dual Gradient Sensors. For the gradiometer application, two identical sensors are mounted vertically at the ends of a rigid fiberglas tube. In the standard configuration, the centres of the coils are spaced 0.5 m apart. An optional configuration separates the coils by 1.0 m. Interconnections are carried through a cable, 3 m long and terminated in a connector which interfaces with a connector on the rear of the OMNI IV. The sensor pole is the same as that used with the remote sensor.

Base Station Sensor. The base station application can use either a standard remote sensor or a dual gradient sensor. The interconnection cable between the sensor(s) and the OMNI IV can be 28 m long. This allows the instrument to be located in an enclosure while the sensor is located outside.

Base Station Sensor Pole. The sensor support pole is fitted with a rope joiner. This is a collar with anchor tie points for attaching three non-metallic guy ropes.

Battery Cartridge Power Supply. Three types of power supply are available (a. and b. are optional; C. is standard):

a. A non-magnetic rechargeable battery pack with eight sealed lead acid cells.

b. A non-magnetic rechargeable battery belt with eight sealed lead acid cells.

c. An alkaline battery belt with 12 D size alkaline disposable power cells.

Battery Belt. This is a webbed belt with a zip enclosure enclosed pouch. For the standard configuration, each system includes a battery belt for alkaline 'D' size batteries. Optionally, the belt comes with sealed, lead-acid rechargeable batteries. The batteries are placed in protective packing inside the pouch. Interconnections between batteries are made internally so that there are two outputs: One is the power output; the other is the battery charging and data dump connection. The power output cable is connected to a base plate which is similar in appearance to the battery pack which mounts on the base of the instrument. Therefore, when the base plate is attached to the instrument, the battery connections to the instrument are completed.

Battery Charger. Each system with the optional rechargeable batteries, includes a battery charger, MBC-100 (Part No. PPX-255) for use with the sealed lead acid battery pack. The batteries are recharged from a 110 Vac or 220 Vac source while the cartridge is located on the console, or in the belt.

Base Station Power Supply. In the base station mode, the OMNI IV can also be operated from an external 12 Vdc car battery.

NOTE

The characteristics of alkaline batteries require a program variation. For this reason, the second digit of the operator code is entered as a '9' (eg, OP39NN) for alkaline batteries and any other digit for rechargeable batteries.

Alkaline Batteries. The use of alkaline batteries is now possible. Previously, they could not be used because the unit sees a low battery voltage during polarization. Until now this was interpreted as sealed lead-acid batteries which require charging to prevent damage to the batteries. To bypass this protection monitor, it is necessary to enter an OP-code that indicates alkaline batteries (which have much lower voltage during load). Enter a '9' as the second digit of the code, ie, OP39NN for the total field unit; OP49NN for base station unit. The second digit of the operator code must be a digit other than 9 if normal rechargeable batteries are used. This prevents damage to the rechargeable batteries.

NOTE

Alkaline batteries cannot be used when the unit is operated as a gradiometer.

SECTION 3

PRINCIPLES OF OPERATION

3.1 INTRODUCTION

This section is confined to the principles of operation of the OMNI IV instrument. An explanation of the design concept and the derivation of constants used in the design concept are beyond the scope of this manual.

3.2 PRINCIPLES

The instrument includes a signal processing method which, for the first time, produces records which are meaningful statistically.

The instrument performs the following functions:

a. It makes an accurate measurement of the total magnetic field strength and processes the information to verify its accuracy.

b. It stores the measurement together with various parameters including signal strength and rate of decay.

c. It records location data so that each measurement can be identified in terms of geographical coordinates (or by record number) related to a precise ground location.

d. It records the date and time at which each reading was taken.

e. It records the type of data recorded, whether it is corrected or uncorrected data, operator identification number, number of readings taken, and voltages of battery.

f. It provides visual readouts of parameters based on keypad commands, ie, datum to be subtracted.

g. It produces data block header information which identifies both the operator and instrument used.

h. It provides three data output formats, for hard copy print-out and digital data transmission.

j. It performs built-in test programs which provide a high confidence level in field measurements.

Circuit operations, computer programs and data logging requirements which fulfill these functions are described in the following paragraphs.

3.3 OPERATOR CONTROLS

Upper Panel. Visual readouts are provided on a custom designed liquid crystal display (LCD) which is capable of operating in ambient temperatures from +55°C to −40°C. The display utilizes a special low temperature fluid which allows the display to be operated down to −35°C. However, at this temperature, the response time is slow. A heater is built into the display. The heater circuit is controlled by a thermal relay so that it is turned on automatically when the ambient temperature is below −25°C. This is to ensure a specific response time. When the unit is turned off (ie, MODE selector to OFF), this heater circuit is disconnected to conserve battery power and the automatic temperature control is overridden. Therefore, in extreme temperatures, when the unit is not being used, it is recommended that it be stored inside, in the usual personnel accommodation environment to ensure that the display becomes operational with a minimum warm-up period.

Mode Selector. The instrument operating mode is established by the setting of the MODE selector. The measurements used in the self-correcting computations for survey measurements are recorded in the TIE mode. Survey measurements are made in the total field (TFM) mode. Survey gradient measurements, using dual (gradient) sensors, are made in the GRAD mode. Sequential measurements taken at the same time interval and at the same location, (ie, base station application), are made in the AUTO mode. Data block output occurs in the data dump (DUMP) mode. Data output in an analog profile plot format occurs in the program (PROG) mode. In the TEST mode, simulated field measurements are made and software diagnostics are performed. The OFF mode is used to power down the display heaters and electronics when the OMNI IV is not being used or during shipping. The memory and real-time clock is still intact in this mode.

Keypads. There are two keypads; the left hand pad is for programming the instrument; the right hand pad is for recording and modifying measurement and reference data. The keys are pressure sensitive push-buttons. The primary key functions which are parameter and command functions, are identified in black lettering (eg, TIME).

Note

The individual key functions are described in Part 4.

3.4 Battery Power

Field operations are carried out under a rigid computer-controlled program of conservation which maximizes the operational period between battery charging or replacement. This is accomplished during a brief, two second reading

sequence. Residual battery power is monitored for every reading. The OMNI IV also has a unique feature called 'constant energy polarization'. The same amount of power is used to polarize the sensor for each reading regardless of the state of depletion of the batteries. When the required amount of power is not available, a reading is prevented.

3.5 Reading Sequence

A reading sequence is started by operator command. This turns on the power and initiates the selected computer program. First, the computer is initialized, then the setting of the MODE selector is interpreted. If a total field measurement is being taken, the following programmed sequence occurs.

a. Polarizing Cycle.

b. Signal Analysis Cycle.

c. Tuning Sequence.

d. Display Cycle.

Polarizing Cycle. During this period the sensor or sensors are energized.

The OMNI IV magnetometers utilize the principle of constant energy polarization. Most instruments utilize a fixed polarization time regardless of the battery voltage. It can be demonstrated that with such a method, a 40% deterioration occurs in sensor signal strength rendering the instrument to be more and more inaccurate (scatter phenomena).

The microprocessor within the instrument computes the battery voltage during polarization and at a specific point determines whether or not the polarization time should be increased. This method ensures that the sensor always receives the same amount of energy per unit of time. Although the performance is not linear, a significant improvement results even when the battery is approaching the depletion state.

Signal Analysis Cycle. At the end of the sensor energizing period, the signal decay cycle occurs. The precession signal is monitored and the decay rate, analyzed. This precession signal is compared statistically to an optimized decay curve at 400 points along the curve. Computations are made and the results of the computations displayed as error.

Tuning Sequence. After the signal analysis period, this new tuning value becomes the basis for the next reading. The measurement portion of the reading sequence occupies typically 2 seconds.

Display Cycle. The selected parameter is displayed on the liquid crystal display (LCD) for approximately 8.5 seconds. At that time, the audio circuits provide a sequence of beeps which indicate that the instrument will be shut down after another 8.5 seconds unless the same command or another command is keyed.

Note

During this cycle, valid readings, location and time values are entered into scratch-pad memory as a data block.

To summarize, a typical reading sequence terminates after about 20 seconds. Then the computer turns off the power.

3.6 Audio Circuits

The instrument provides both visual and audio feedback. Every time that a key is pressed, an audio beep will be heard. Parameters are displayed for 8.5 seconds only. The moment that this visual period has elapsed, the audio is activated to give repeated beeps for another 8.5 second period to indicate the end of the data block display period. If the operator fails to press any key during this warning period, the instrument turns off automatically. Every time that a key is pressed, a new 8.5 second period is initiated. This allows the operator to have enough time to evaluate and program data. The display period may be extended by pressing the same key or another key. However, selection of a different key initiates a different display response.

3.7 Statistical Error Alarm

Following each reading, the instrument computes the true statistical error of the total field reading. This value represents the confidence level of the data. A visual alarm may be triggered based on the following criteria:

a. When the statistical error of a particular reading is equal to or larger than 0.2 gamma, the least significant digit of the displayed total field flashes on and off. For example, assume that the statistical error is 0.23 gamma and the displayed total field is: 57936.4. In this case, the 4 flashes on and off. Flashing the least signficant digit on and off indicates that the accuracy is impaired and the operator should be warned.

b. When the statistical error of a particular reading is equal to or larger than 2.0 gammas, the two least significant digits of the displayed total field flash on and off. For example, assume that the statistical error is 2.3 gamma and the displayed total field is: 57936.4. In this case, the 6 and 4 flash on and off. Flashing the two least significant digits on and off indicates that the accuracy is badly impaired and an audio warning sounds to alert the operator.

NOTE

If the error is larger than 2.0 gammas, take another reading.

3.8 Automatic Fine Tracking (Tuning) of Total Field

The OMNI IV contains high technology circuits which allow for automatic fine tracking (tuning) over the entire field strength range from 18,000 to 110,000 gammas, under computer control. An optimized tuning algorithm ensures that the system is tuned for optimum performance.

Under normal operating conditions, a ± 15% capture tuning range from reading to reading is achieved. The capture tuning range is defined as the difference between the previous and current readings relative to the previous reading. However, if a larger difference is computed, the instrument will warn the operator of this and further measurements are inhibited automatically. The warning is both visual and audible. The TUNE descriptor located on the left hand side of the display flashes on and off and the audio alarm is enabled as well. The displayed total field reading also flashes on and off to inform the operator that the displayed value is invalid. The value displayed, however, is the previous total field value, and should be changed manually to reflect the new tuning field.

3.9 SURVEY GRID

When a total magnetic field survey is conducted, it is necessary to locate and record the ground position where each measurement is made. In the grid method, the operator usually follows a predetermined straight track and takes measurements at fixed intervals along that track. When the arbitrary end of the track is reached, the operator moves a fixed distance to the left or right of this track, then measurements are taken along a parallel track but moving in the opposite direction.

The OMNI IV is designed to operate on a rigidly controlled grid similar to this. See Figure 3-1. The tracks which are followed are designated as lines and the locations along each line where a measurement is taken is designated as a position (station). The fixed distance between each position is defined as the position spacing and the distance between parallel lines is defined as the line spacing.

Preparing the Grid Matrix. When a field measurement is taken, the line number and the position number along that line must be recorded together with the measurement to produce a meaningful record. This recording procedure is simplified by the computer. First the grid coordinates of the starting point are entered. These entries are in the form of a line number and a position number. Then the spacing intervals between lines and between positions are entered by the operator. Therefore, when a reading is taken, the line and position numbers are recorded automatically. At the next position along the line, the computer can update the position number by adding one position spacing increment to the previous position number. This can be accomplished automatically so that the new measurement record is identified by the original line number plus the updated position number.

When the end of a survey line has been reached, the line spacing interval is activated, (either positive or negative as shown in Figure 3-1), to set up the next line number. By the fact that the operator will now be proceeding in the opposite direction, the sign of the position spacing increment must be changed (toggled).

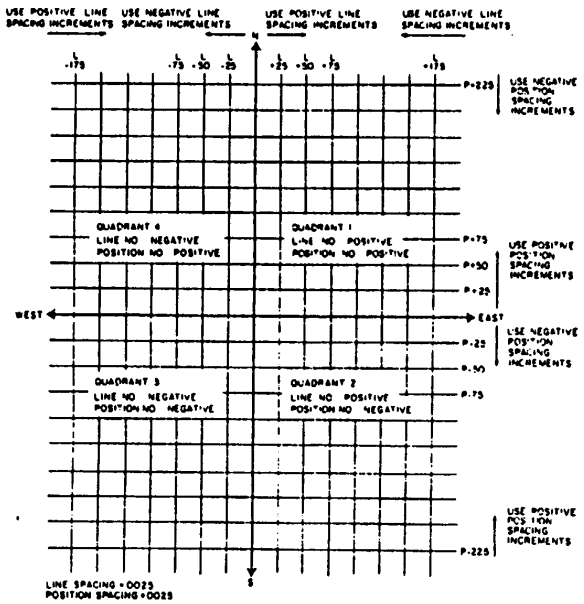


Figure 3-1 Grid Configuration

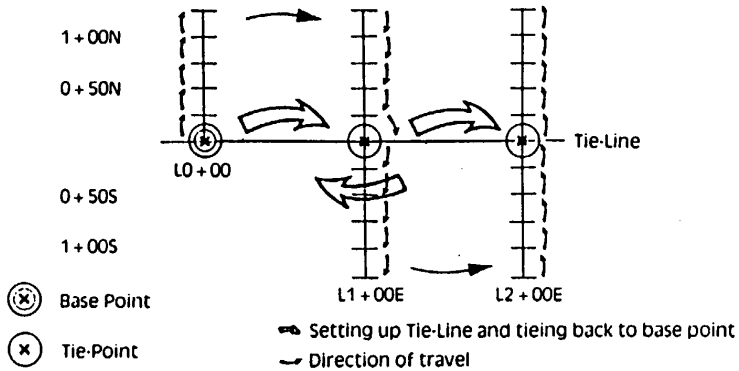


Figure 3-2 Diagram Showing Tie-Line Survey Method

Correction of Survey Data. The self-correcting feature of the OMNI IV is based on using reference measurements obtained by a tie-line or looping method of correcting data. The correction process is accomplished rapidly and accurately using the internal microprocessor, a unique program algorithm and two separate memories.

The procedure is straightforward.

a. At the beginning of the survey day, a total field reading is taken by the operator at a known and identified base reference point in the survey grid. This is the data and grid reference for either the tie-line or looping method for diurnal correction. The value, which is identified by grid coordinates and the time of the reading, is stored as a data block in a special memory. This special memory is accessed when the MODE selector is set to TIE.

b. **Tie-Line Method.** See Figure 3-2. In this method, several coordinates in the survey grid are identified as tie points. Frequently, these tie points are selected along a grid line which passes through the base reference point. However, multiple tie lines or even a random scattering of tie points can be selected.

c. As soon as the base reference recording has been made, a total field reading is taken at each selected tie point in sequence. At the end of the sequence, a second recording is taken at the base reference point to close the loop. This step must be carried out in the shortest time possible to improve the correction accuracy. Each of these data blocks is stored in the tie memory file.

d. Next, the tie point values are corrected diurnally to the base reference point. To achieve proper diurnal correction of each tie point value, the microprocessor computes by linear interpolation, the drift in the earth's magnetic field with respect to the time that each reading was taken. By distributing the drift in the earth's magnetic field and applying it to the times that the tie-point readings were taken, extremely accurate field results are computed for each tie point.

e. Now the remainder of the survey can be conducted by collecting total field values at each coordinate in the survey grid. Each reading is stored as a data block in a separate memory called the field data memory file. This memory is accessed when the MODE selector is set to TFM (for total field magnetometers) or GRAD (for gradiometers). Each time that the operator arrives at a coordinate which has been designated as a tie point, the new reading is flagged in the memory for calculating diurnal drift. The first and last reading of the survey during the day should be on any tie point, in the TFM or GRAD mode; or on the base reference point when the TIE mode is selected.

f. At the end of the survey day, the data stored in the tie-line memory file is merged with the field measurements stored in the field data memory file. The microprocessor then computes the variation in the earth's magnetic field for each grid coordinate and corrects the field data file using the tie-line file. No manual computations are required.

g. **Looping Method.** See Figure 3-3. This method is similar to the tie-line method. However, instead of selecting numerous tie point references, only the base reference point is used. At the start of the survey, a data block is recorded at the base reference point with the MODE selector set to TIE, and stored in the tie-line memory file.

h. During the survey, total field values are collected at each grid reference. Each reading is stored as a data block in the field data memory file. Then, at intervals within 30 to 60 minutes, the operator returns (loops back) to the base reference point and takes another reading. Each of the total field readings taken at the base reference point is stored as a data block in the tie-line memory file.

j. At the end of the survey day, the base reference data stored in the tie-line memory file is merged with the field measurements stored in the field data memory file.

k. The microprocessor computes by linear interpolation, the drift in the earth's magnetic field with respect to the time that each base reference point reading was taken. Then these corrections are applied to each reading in the field data file.

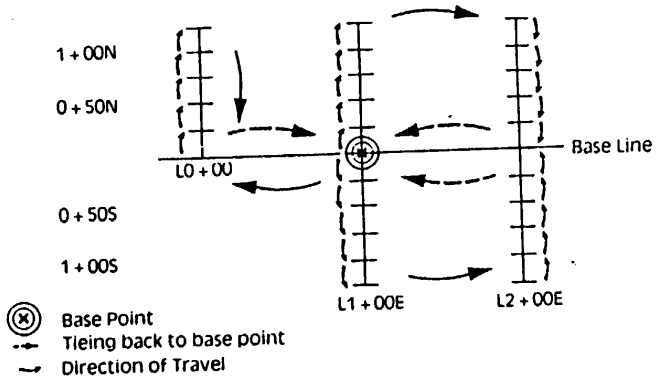


Figure 3-3 Diagram Showing Looping Survey Method

3.10 BASE STATION (AUTO-CYCLING) OPTION

In addition to the self-correcting feature of its data using the tie-line or looping method, the OMNI IV can also include a base station or auto-cycling option. The option enables the instrument to perform as a self-recording base station. This is most useful in locations such as Northern latitudes where more detailed monitoring of the earth's magnetic field is required.

Auto Cycle. The base station OMNI IV is set up and remains stationary at the base reference station. When the MODE selector is set to AUTO, the instrument takes readings at a preprogrammed time interval (adjustable from 5 seconds to 60 minutes in 1 second increments).

Survey Data Correction. In order to correct the total field data recorded in a separate OMNI IV, the total field magnetometer and the base station are interconnected through a cable. This cable is also connected to the external computer or printer. The data from the field unit is then read by the base station and the diurnal variation corrections computed within the base station magnetometer. The base station unit then sends the corrected field data to a printer or a computer.

SECTION 4

OPERATION

4.1 ARRANGEMENT

The electronic console includes:

a. An operating-mode selector with eight positions.

b. A programming keypad on the left side, with 12 pressure sensitive keys.

c. A recording keypad on the right side, with 6 pressure sensitive keys.

d. An LCD multi-function display with digital readout.

These are all that is necessary for operating and checking the instrument and for making field measurements.

NOTE

(Reference: 1 γ (gamma) = 10^{-5} gauss = 1nT (nanoTessla).)

4.2 DESCRIPTION OF SWITCHES AND INDICATORS

The locations of the MODE selector positions, the programming and recording keypad push-buttons, are shown in Figures 4-1, 4-2 and 4-3 respectively. Their functions during normal operation are given in Tables 4-1, 4-2 and 4-3 respectively. The liquid crystal display (LCD) is shown in Figure 4-4. The descriptions are explained in Table 4-4.

4.3 MODE SELECTOR

At the lower edge of the electronic console is an eight position mode selector. The selections permit field measurements, data retrieval, functional testing and battery power conservation.

Figure 4-1 Mode Selector

Description	Function
MODE selector	This eight-position selector allows the instrument to be operated in several different modes (ie, as a field mag, base station, or gradiometer).
OFF	The display heater circuit is disabled when the MODE selector is set to OFF. This prevents unnecessary power drain when the ambient temperature drops below −25°C and the instrument is not scheduled for use for a period of time. NOTE **The display heater circuit becomes enabled automatically when the temperature is below −25°C and the MODE selector is not set to OFF.**
TEST	In this mode, the unit may be tested by pressing the READ key. The instrument conducts a synthetic test to verify proper operation of the electronics and program software. The test result, which is a total test field expressed in gammas, appears on the display, (ie, **56369.7 GAMMA**). The program memory is also subjected to a test. In addition, when the ERROR key is pressed, proper operation of the mathematical statistical error routine is verified, (ie, LCD displays .00 GAMMA).
TIE	In this mode, tie-line or looping data is taken and recorded in a separate memory. This becomes the baseline data which is recalled automatically and used by the microprocessor to correct survey readings taken later during the day of survey and in the TFM mode. For TIE, each data block stored in the separate memory contains: a. Time of reading (HH:MM:SS). b. Total field intensity. c. Statistical error analysis. d. Grid coordinates (line number, position number). e. Decay rate. f. Signal strength.

Table 4-1 Mode Selector

Description	Function
	NOTE Tie-line data can be recalled even if stored on different days.
TFM (Total Field Mode)	This is the selection for normal operation during a survey. In this mode, the instrument may be operated normally to measure and store the total field intensity. For TFM, each data block stored in memory contains: a. Time of reading (HH:MM:SS). b. Total field intensity. c. Statistical error analysis. d. Grid coordinates (line number, position number). e. Decay rate. f. Signal strength.
GRAD (Vertical gradient)	This is the gradiometer mode. In this position, two measurements are made: a. The total field intensity is measured using the top sensor. b. The gradient of the total field is calculated by taking simultaneous readings with both sensors separated by a distance of 0.5 m or 1.0 m. For GRAD, each data block stored in memory contains: a. Time of reading (HH:MM:SS). b. Total field intensity (top sensor). c. Gradient measurement. d. Statistical error analysis. e. Grid coordinates (line number, position number). f. Decay rate. g. Signal strength. In the grad position, the instrument measures, calculates, displays and stores the vertical gradient, expressed in gammas gradient/metre.

Table 4-1 Mode Selector (Cont)

Description	Function
AUTO	This is the base station mode. In this mode, data is taken and accumulated in memory to monitor the earth's magnetic field at the base station location. Readings are taken at a preprogrammed interval. This interval can be varied from 5 seconds to 60 minutes in 1 second increments. Up to 5000 data blocks can be stored in memory. Each data block contains: a. Time of reading (HH:MM:SS). b. Total field intensity. c. Difference from the programmed reference field value. d. Difference from the previous reading (ie, drift). e. Date of day.
DUMP	This position is applicable to all modes. In this mode, the data is retrieved from the protected memory using various access codes. The access codes format the data output required (eg, field data, corrected data, gradient data, etc). In the data print-out, the grid coordinates can also be represented by compass directions (ie, N, S, E and W).
PROG	This position is applicable to all modes. In this mode, data can be output in an analog profile plot format. Scaling of the profile plot is programmed through the keypads. This selection is ideal for presenting base station data to monitor the activity of the earth's magnetic field. This selection also outputs data in a fixed ASCII format for computer processing.

Table 4-1 Mode Selector (Cont)

4.4 KEYPADS

The console is equipped with two custom-designed keypads: One for programming the instrument; the other for recording and modifying measurement and reference data. The keys are of the touch sensitive type, however, to activate a key some pressure must be applied. An audio tactile feedback system is incorporated so that when a key is pressed, an audio device produces a single short-period beep. The keypads are completely waterproof.

Programming Keypad. This keypad contains 12 keys arranged in a matrix. Keys 1, 2, 3, 5, 6, 9 and CLEAR have three levels; the OFF key has one level; and the remainder have two levels.

a. The first level represents function parameters. These functions are printed in black lettering on a white background (eg, TIME, ERROR, etc).

b. The second level which consists primarily of numerics is printed in red on a white background. The CHANGE key on the recording keypad is the shift key from functions to numerics for the programming keypad.

c. The third level also represents function parameters. These are also shown in black lettering on a white background between the function descriptor and numeric (eg, DATE, DATUM and GRAD).

The OFF key is single level with one function only.

NOTE

The first and third levels form a toggle type push-button. Consecutive operations of the push-button activates the alternate selection.

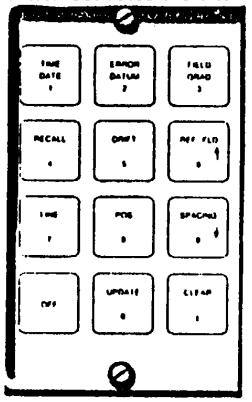


Figure 4-2 Programming Keypad

Key	Function
TIME DATE 1	When this key is pressed once, the actual time of day (ie, 24 hour real-time clock) will be displayed in hours, minutes and seconds (HH:MM:SS) (eg, 13:24:42). The time is not updated automatically on the display. The displayed time relates to the time of a measurement. When the key is pressed a second time, the date will be displayed in year, month, day (YY:MM:DD) (eg, 85:03:31). (The clock does not have leap year compensation. The year must be updated manually on Jan 1st.) When changing the date, the time must also be re-entered in order to enter the new date in the real-time clock. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the TIME key becomes a numeric key with a value of 1.
ERROR DATUM 2	When the key is pressed once, the statistical error will be displayed in gammas, with a resolution of 0.01 γ. NOTE The statistical error can only be displayed after the total field value has been displayed. Refer to FIELD key operation. To display the statistical error, the ERROR key may be pressed at any time to obtain a first time display or for display recall purposes. When the key is pressed a second time, a datum value can be entered. The datum is then subtracted from the raw or corrected total field data when it is being dumped. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the ERROR key becomes a numeric key with a value of 2.

Table 4-2 Programming Keypad

Key	Function
FIELD GRAD 3	The FIELD and GRAD selections may be considered as a toggle switch. When this key is pressed once, the total magnetic field reading will be displayed, expressed in gammas. NOTE This key is used primarily for total field recall, because, in normal operation, the total field value will be displayed first automatically. This key may also be used for displaying the total field value when it requires changing whenever the instrument needs to be tuned. When the GRAD mode is selected and this key is pressed, the display first indicates total magnetic field reading taken by the upper sensor. When the key is pressed again, it toggles to the GRAD selection and displays the vertical gradient automatically in gammas gradient/metre. The gradient display cannot be changed. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the FIELD key becomes a numeric key with a value of 3.
RECALL 4	This selection accesses data in memory to recall the total field readings entered previously. With RECALL selected keys 6 and 9 can be used to scroll the memory in order to display previous records. Key 9 decrements the record location; key 6 increments it. In recall, any stored parameter (ie, date, time, line, position, error, total field, or gradient) can be displayed by pressing the appropriate function key. NOTE During recall, the decay and sensor descriptors will all be flashing to distinguish between parameters displayed in normal operation and those displayed in recall.

Table 4-2 Programming Keypad (Cont)

Key	Function
	When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the RECALL key becomes a numeric key with a value of 4.
DRIFT 5	When this key is pressed once, the change in reading between the two most recent readings is displayed. When the key is pressed again, it indicates the field variation between the two most recent tie points. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the DRIFT key becomes a numeric key with a value of 5.
REF.FLD ↑ 6	When this key is pressed in the base station or tie-line mode, a programmable magnetic field value can be entered. This value is then subtracted from the raw base station data to obtain the drift correction which is to be applied to the field data. The reference field is also used for the drift correction if the unit is used as a tie-line magnetometer. When RECALL has been selected, this key (↑) increments the memory location. When the key is held, it scrolls the memory in the fast forward direction.
LINE 7	When this key is pressed once, the current line number (longitude) will be displayed. This is a four digit number and can be either positive or negative. For example, a line to the west of the quadrant reference might display L – 0525. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the LINE key becomes a numeric key with a value of 7.
POS 8	When this key is pressed once, the current position or station number (latitude) will be displayed. This is a four digit number and can be either positive or negative. For example, a position to the south of the quadrant reference might display P – 0085.

Table 4-2 Programming Keypad (Cont)

Key	Function
	When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the POS key becomes a numeric key with a value of 8.
SPACING ↓ 9	In field survey applications, (ie, TIE, TFM or GRAD), this key initiates a display of the spacing between lines or positions. a. To obtain line spacing: (i) The LINE key is pressed first. This causes the current line number to be displayed. (ii) Next, the SPACING key is pressed to display the spacing increment between adjacent lines which has been set into the instrument memory. An example of a spacing display is INTV L – 0025. NOTE When line spacing is being displayed, the descriptor INTV (interval) appears on the left of the display. This enables the operator to differentiate between a line number display and a line spacing display. b. To obtain the position (station) spacing: (i) The POS key is pressed first. This causes the current position number to be displayed. (ii) Next, the SPACING key is pressed to display the spacing increment between adjacent positions which has been set into the instrument memory. An example of a spacing display is INTV P – 0025. This means that for each sequential field measurement, the grid position reading will change by – 0025. Station spacing of 12.5 can be entered by keying in 0012. The display will show 00:12 with both the decimal point and colon flashing to indicate that the .5 is not being displayed.

Table 4-2 Programming Keypad (Cont)

Key	Function
	NOTE When position spacing is being displayed, the descriptor INTV (interval) appears on the left. This enables the operator to differentiate between a position number display and a position spacing display. In base station applications, (ie. AUTO mode), after first pressing the TIME key, the SPACING key initiates display of the programmed sampling interval (ie, set between 5 seconds and 60 minutes). Time spacing is also used for the field magnetometer if synchronization is required between the base station and field units. If it is not required, this feature is disabled by entering 0000 for the time spacing in the field unit. When RECALL has been selected, this key (1) decrements the memory location. When the key is held, it scrolls the memory in the fast backwards direction. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the SPACING key becomes a numeric key with a value of 9.
UPDATE 0	The function of this key is to update the displayed position number in the survey grid. When the UPDATE key is pressed and no parameter related to grid coordinates is displayed, as a default, the position number will be incremented or decremented automatically defined by the position spacing and sign. To update the previous position number, the operator may also press the POS key first. The display will indicate the last stored position number. When the UPDATE key is pressed, the displayed position number is updated by the programmed position spacing increment. Consider the example in which the current position number in the grid is P – 0350; and the position spacing increment is INTV P – 0015.

Table 4-2 Programming Keypad (Cont)

Key	Function
	NOTE In terms of the grid reference, this implies that the operator is south of the zero position reference and is proceeding southwards along a grid line. If the POS key is pressed, P – 0350 will be displayed. Then, when the UPDATE key is pressed, the spacing increment will be added to this: (ie, – 0350 + (– 0015) = – 0365). The display then changes to P – 0365. This is the position number of the ordinate which is adjacent to the updated ordinate along the track. Consider another example with P 0575 (ie, north of the zero reference line) and INTV P – 00:12 (ie, proceeding southwards). The updated position number becomes +0575 + (– 0012.5) = P 0562.5. This is displayed as P 05:62. The procedure for updating the line number by adding the line spacing is the same provided that the current line number is displayed first, (ie, press LINE prior to pressing the UPDATE key). It is evident that all four quadrants on the survey grid can be described accurately provided that the origin of the grid is in the centre. NOTE If the display is not indicating a position number or the position spacing when the UPDATE key is pressed once, the display will indicate automatically the updated position number on the grid as a default condition. When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the UPDATE key becomes a numeric key with a value of 0.

Table 4-2 Programming Keypad (Cont)

Key	Function
CLEAR ±	In any of the manual measurement modes, (ie, TIE, TFM or GRAD), if the RECALL key is pressed to display the most recent reading, then the CLEAR key is pressed, that reading is deleted from the memory. No other reading can be deleted. In the DATA DUMP mode, if the CLEAR key is pressed it clears the tie-line memory of all tie-line data if HP8888 is entered. When the instrument is in the change mode (ie, when the CHANGE key is pressed), the CLEAR key becomes a mathematical key with a ± toggle application to change the sign. NOTE Only the negative sign is displayed. The positive sign is replaced by a space or blank.
OFF	When this key is pressed, it commands the instrument to turn off power. The power-down sequence is under computer control to ensure an organized shut-down. NOTE The OMNI IV shuts down automatically after approximately 20 seconds if no keys are pressed. The OFF may be used to override the normal 20 second display time-out period to conserve battery power any time.

Table 4-2 Programming Keypad (Cont)

Recording Keypad. This keypad contains 6 keys in a matrix. The READ and AUTO-RECORD keys occupy the space of two square keypads. The READ key has black lettering on a bright red background. These keys are single level. The SPOT, MULTI and AUTO RECORD keys enable the operator to take a total field reading and record the data in a choice of three different ways depending upon the survey grid configuration established:

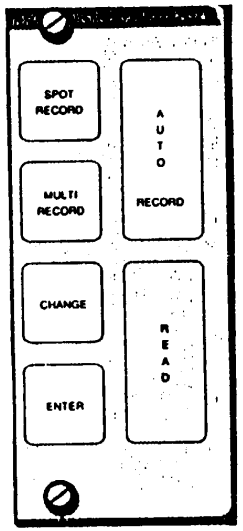


Figure 4-3 Recording Keypad

Key	Function
	NOTE The SPOT RECORD, MULTI RECORD and AUTO RECORD keys are single level keys with a built-in safety mode. Each key has to be pressed twice before an action can be initiated. Following a data dump sequence, and when the operator attempts to store the very first reading again using the SPOT RECORD, MULTI RECORD or AUTO RECORD keys, the instrument will refuse to store the data block unless the software code plus an operator code number has been entered.
R E A D	When this key is pressed, it commands the instrument to turn on its power. First, the computer initializes itself, then the setting of the MODE selector is read. The computer program then proceeds with routines on the basis of the MODE selected. Then the OMNI IV takes a reading of the magnetic field and displays it on the LCD. This reading can then be stored using one of the three RECORD methods.
SPOT RECORD	In this record, the information is stored together with a record pointer number. There is no indication of the grid coordinates (ie, line and position number). Following a data dump, the first spot record becomes 0001. This is incremented with each new reading. When SPOT RECORD is pressed once, the next record number is displayed. A typical display could be RP0052. (The prefix designator RP means record pointer.) This RP number becomes assigned automatically to the data to be stored in memory. When the number is displayed, it allows the operator to record this number, on a map or chart for example, for cross-reference later when required. The new data will not be stored in memory yet. To store the data under the displayed record pointer, the SPOT RECORD key must be pressed a second time. In response, the instrument stores the data, then it switches itself off immediately.

Table 4-3 Recording Keypad

Key	Function
MULTI RECORD	This key is used to store many readings at one station without updating the station coordinates. The multiple entries are similar to the SPOT RECORD entry except that the coordinates are repeated. At the appropriate grid coordinates, the READ key must be pressed to obtain the total field reading. This reading is displayed on the LCD. When the MULTI RECORD key is pressed once, the position number will be displayed and an audio beep is repeated until a follow-on key operation occurs, or the wait period times out. To store the data under the current grid coordinates, the MULTI RECORD key must be pressed a second time. In response, the instrument stores the data, then it switches itself off immediately. Repeat readings can be taken and stored by pressing READ, then MULTI RECORD twice. However, the data printout will show these readings keyed to the same grid coordinates (ie, the coordinates do not increment in this multi record mode). With the MODE selector set to TIE, this key functions in the same way, except that the default parameter displayed is the LINE instead of the POSITION.
A U T O RECORD	This key is used in two modes: Tie line measurements; and field survey measurements. a. When TIE is selected and a reading is taken, the line number is incremented automatically by the programmed position spacing increment. b. When TFM or GRAD is selected and a reading is taken, the position number is incremented automatically by the programmed line spacing increment. At the appropriate grid coordinates, the READ key must be pressed to obtain the total field reading. This reading is displayed on the LCD. When the AUTO RECORD key is pressed once, the line number will be displayed (in the TIE mode) and the position number will be displayed in the TFM or GRAD modes. At the same time an audio beep is repeated until a follow-on key operation occurs, or the wait period times out. To store the data under the current grid coordinates, the AUTO RECORD key must be pressed a second time. In response, the instrument stores the data, the position number (TFM or GRAD) or the line number (TIE) increments by the spacing increment, then it switches itself off immediately.

Table 4-3 Recording Keypad (Cont)

Key	Function
	NOTE If interaction is required to change the displayed coordinate, this can occur before the key is pressed for the second time because the data will not have been stored in memory yet.
CHANGE	When the CHANGE key is pressed, two functions are initiated: (i) The keypad function keys are changed to the numeric value mode. (ii) The values of the following parameters can be selected, displayed and changed individually: Time; date; total field, (for the purpose of tuning only); line number and line spacing; position number and position spacing; operator number; and confidential number.
ENTER	This key enters the desired numeric data into memory and returns the selected function key to the function (black) level. This key can be pressed at any time, even while some digits are still flashing. When the audio warning is sounding and all digits are flashing, this key is disabled until after the CHANGE key is pressed first to turn off the audio warning.

Table 4-3 Recording Keypad (Cont)

4.5 LIQUID CRYSTAL DISPLAY (LCD)

The display is a custom-designed, ruggedized, high-quality, liquid crystal display. It utilizes a fluid which allows the display to be operated in temperatures as low as −35°C without any heating. However, at −30°C, there is already a significant segment turnover slow-down. A safety margin is provided by incorporating a heater system which becomes enabled at about −25°C. During prolonged low temperature operation, the heater circuitry remains enabled even when the power to the instrument is switched off, provided that the MODE selector is not set to OFF.

NOTE

When the ambient temperature is lower than −25°C and the magnetometer is not in use for a while, it is recommended that the MODE selector be set to the OFF position. This action prevents continuous battery power drain. The display comprises ten individual descriptors; three bar and wedge graphic indicators; and the primary numeric readout. The numeric readout consists of six digits; two colons which separate the digits into three pairs; and two individual decimal points which indicate tenths or hundredths. (Examples of displays are given throughout Tables 4-1 and 4-2.) Refer to Figure 4-4 and Table 4-4.

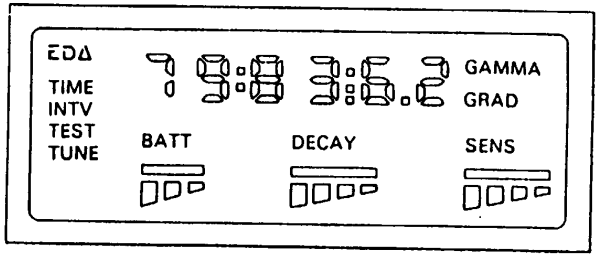


Figure 4-4 Liquid Crystal Display

Description	Function
EDA logo	The letters **EDA** are displayed whenever the instrument is on. The logo will commence to flash on and off during data dumping when the internal lithium battery system which is for emergency battery back-up, is depleted. When the logo starts to flash, the lithium battery must be replaced. NOTE EDA recommends that the internal lithium battery be replaced by EDA personnel. If this is not possible, consult EDA first, prior to changing the battery, to obtain instructions. The life of the lithium battery is estimated to be five years under normal conditions.
TIME	The word **TIME** will be displayed when the TIME key is pressed once. This is accompanied by the six-digit local time display. When the TIME key is pressed a second time, the word TIME disappears and the date is displayed.
INTV	The descriptor **INTV** (interval or spacing) will be displayed when the time interval, line or position spacing is displayed.
TEST	The word **TEST** will be displayed when the MODE selector is set to the TEST position and the READ key is pressed.
TUNE	The word **TUNE** will be displayed in a flashing manner automatically when the instrument detects that the system requires tuning. Tuning is achieved by pressing the CHANGE key and entering a new value of total field which is close to the actual ambient total field value.
GAMMA	The word **GAMMA** will be displayed when the actual total field measurement or the test field reading is displayed. The word also appears when the ERROR key is pressed.
GRAD	The word **GRAD** will be displayed when the MODE selector is set to the GRAD position, and the gradient is displayed by pressing the FIELD/GRAD key. The word **GRAD** will also be displayed if the error of the gradient (lower) sensor is displayed.

Table 4-4 Liquid Crystal Display

Description	Function
BATT	The word **BATT** will be displayed during normal operation. It indicates that adequate power remains in the battery system. When **BATT** commences to flash on and off, the operator is warned that the battery is approaching the discharged condition rapidly. When the audio alarm is activated, the battery must be changed or charged. No further readings are possible. The battery monitor comprises the **BATT** descriptor, a rectangle and three wedge-shaped bars. With a fully charged battery system, all three bars are displayed. Gradually, after many readings, the residual battery power decreases. This is indicated when the left hand bar disappears. With continued battery discharge, the second bar disappears, followed eventually by the third bar. When this condition occurs, the battery is almost depleted. Therefore, it is recommended that the battery system be recharged or replaced when the **BATT** descriptor commences to flash on and off. The instrument inhibits any further readings when the **BATT** descriptor flashes and the audio alarm is actuated.
DECAY	The word **DECAY** will be displayed during normal operation. When the word commences to flash on and off, it indicates that the decay rate of the precession signal is too fast due to large local gradients. NOTE A possible way to reduce the effect of the large local gradient is to raise the sensor. The precession signal decay rate monitor comprises the **DECAY** descriptor, a rectangle and four wedge-shaped bars. Every time that a reading is taken, the computer calculates the actual decay rate. This is an important parameter because it reveals the degree of gradient tolerance. When the decay rate is too fast due to large local gradients, the accuracy of the measurement is impaired to some degree. The statistical error value reveals this immediately. All four

Table 4-4 Liquid Crystal Display (Cont)

Description	Function
	wedge shaped bars will be displayed when a normal decay rate is computed. When the operator gradually approaches a large local gradient, the computed decay rate increases, and the wedge-shaped bars may gradually disappear in sequence, from the left until the decay rate is too fast for proper signal processing. When this occurs, the **DECAY** descriptor commences to flash on and off and completion of the reading is inhibited. The reading is aborted and the **TUNE** descriptor flashes on and off.
SENS	The word **SENS** will be displayed during normal operation. When the word commences to flash on and off, it indicates that the starting amplitude of the precession signal is below the minimum acceptable threshold. This condition may occur when: a. The sensor is damaged (fluid in sensor is gone). b. A very large local gradient is encountered. c. Sensor is not properly aligned, especially in areas where the total field vector inclination angle is less than 45°. The sensor signal monitor comprises the **SENS** descriptors, a rectangle and four wedge-shaped bars. Every time that a reading is taken, the computer calculates the actual starting amplitude of the precession signal derived from the sensor. This monitor may be used to ensure that the sensor is aligned properly with magnetic North. When the signal amplitude is normal and above a pre-defined threshold, all four wedge-shaped bars are displayed. If the calculated amplitude decreases, the bars disappear in sequence from the left until the amplitude does not meet the minimum threshold level. When this occurs, the **SENS** descriptor commences to flash on and off.

Table 4-4 Liquid Crystal Display (Cont)

4.6 PREPARATION FOR USE

Unpacking. Remove all compontents from the shipping container(s) and check the contents against the Packing Slip/Release Note. Report any shortages or damage immediately to EDA Instruments Inc.

Battery Packs. There are two battery configurations with various battery types. Refer to Table 2-1. One battery pack attaches directly to the underside of the instrument so that it is an integral part when the instrument is chest mounted. Another battery arrangement has the batteries in a wide web belt which is attached around the waist. The connecting cable for the belt pack terminates in a base plate which is similar in appearance to the integral battery pack.

a. Secure the battery pack (or the belt connector base plate) to the base of the console. Align the contact springs in the pack with the conductive discs in the base of the console. (Note: The plate can be installed with the attaching screws at either end.)

b. Tighten the four captive screws in the plate finger tight.

c. Buckle the pack around the waist and adjust by tightening the strap.

Console. The instrument console is carried on the chest and secured by a dual shoulder harness.

a. Attach the harness to the instrument then adjust the straps so that the console is at a suitable viewing and operating height.

Sensor. There are two connectors on the base of the instrument: The one adjacent to the left hand when the instrument is chest mounted is for the sensor cable; the one adjacent to the right hand is for the computer or printer interface cable used to dump data.

a. Attach the cable to the left hand connector. The connector is keyed (ie, centre pin blocked) so that the sensor will only mate with one connector.

b. Mount the sensor on the top section of the pole or staff. The end terminates in a tapered, threaded adapter.

c. Connect the other pole sections in series with the white plastic end tip at the base of the pole.

Operating Stance. To take field measurements, hold the sensor pole vertically at arm's length away from the console. Then operate the appropriate keys.

4.7 OPERATING PRECAUTIONS AND GUIDELINES

☐ **Metallic Objects.** Prior to using the OMNI IV, remove all magnetic material from the body. (To check for the presence of such materials, take two sample readings without moving the sensor. Press the READ key, then press the FIELD key. Take one reading while standing near the sensor, and the other reading at some distance from the sensor. If there is no change in the readings, there is no magnetic material present. Do not store the readings.)

☐ **Survey Log.** Maintain a notebook record during the survey. Annotate all errors and deviations from the usual numbering sequence, SPOT records, etc. Record the locations of roads, fences, pipes, houses. Without clear records, numbering errors are nearly impossible to recover.

☐ **Survey Grid.** Maintain a constant check on the surveyed line and station locations. It is very easy to update position numbers inadvertently, especially when taking multiple readings at a station. It is much more difficult to correct these errors once they are stored in memory. Do not let errors propogate.

☐ Ensure that four-digit line numbers and position numbers describe all station coordinates adequately including fill-ins.

☐ **Spot Records.** Use spot records to delimit erroneous records. For example, if several station recordings have been taken erroneously, take a spot record before resuming the proper sequence. Annotate this reading in the log notebook together with a description of the problem.

☐ **Time Clock.** Synchronize the instrument clocks daily. Drift is ± 1 sec per 24 hours. Make sure that the date is the same on both instruments.

☐ **Base Stations.** When a base station intrument is being used, revisit the base station once a day to verify that it is cycling. Wait for the period to time out to verify that it is functioning. If it has stopped, it should restart by pressing the READ key. If the fault repeats, re-initialize the unit.

☐ When an operator returns to the base station at the end of the day, if the base station instrument has shut down, be sure to take some readings as follows:

a. Wait for cycling interval to time out before deciding that the base station unit has shut down.

b. Set the MODE selector to TFM.

c. Take several readings and store them.

☐ **Initialization.** Read and understand the initialization procedure for field operations.

CAUTION

Never initialize the OMNI IV when it contains valid stored data. This will clear the memory.

☐ **Performance Monitoring.** Observe the DECAY and SENS wedged-shaped bars on the LCD continually, because these give a quick indication of a malfunction. It is possible to observe factors which are degraded severely in areas of high magnetic gradient; so much so, that it may be impossible to store the reading.

☐ **Battery Duration.** A fully charged belt pack will provide power all day (2.5 amp-hours) if total field data is collected, or 6 hours if gradient data is collected. Twelve disposable D cells will provide 5 amp-hours of power which is good for up to 3 days of survey based on 1500 to 2000 readings per day. If a battery does not have sufficient power to achieve constant sensor polarization, the OMNI IV shuts down. If the OMNI IV completes a reading, then that reading can be considered good regardless of the indication of the BATT wedge-shaped bars.

4.8 OPERATING PROCEDURES

CAUTION

At all times, ensure that no device other than the sensor cable is connected to the sensor connector on the OMNI IV. The data dump connector is not capped, therefore, ensure that dirt or contaminant does not damage it.

Similar to most microprocessor-oriented systems, operating procedures are primarily at the discretion of the operator. Keypad control provides the flexibility feature which is only limited by operator ingenuity.

An example grid survey is summarized in Appendix A. In the following paragraphs, the following operating topics are discussed:

General Procedures (para 4.8.1).
Starting the Unit (para 4.8.2).
Changing a Parameter (para 4.8.3).
TEST Mode (para 4.8.4).
Total Field Test (para 4.8.5).
Error Calculation Test (para 4.8.6).
Software Diagnostics (para 4.8.7).

Making Initial Total Field Measurements (para 4.8.8).
Statistical Error Evaluation (para 4.8.9).
PRE-SURVEY PROCEDURES (para 4.9).
Identifying the Instrument (para 4.10).
Erasing the Previous Day's Survey Data (para 4.11).
Initializing the OMNI IV (para 4.12).
Tie-Line Memory (para 4.12.1).
Field Memory (para 4.12.2).
Initializing the Field Memory (para 4.12.3).
Initializing the Tie-Line Memory (para 4.12.4).
Starting the Base Station Magnetometer (para 4.12.5).
Starting the Gradiometer (para 4.12.6).
Pre-Survey Programming (para 4.13).
Real-Time Clock (para 4.13.1).
Reading Synchronization (para 4.13.2).
SYSTEM TUNING (Prior to Survey) (para 4.14).
Retrieval of Last Previously Stored Total Field Value (para 4.14.1).
Tuning the OMNI IV (para 4.14.2).
Incomprehensible Displays (para 4.14.3).
SYSTEM TUNING (During a Survey) (para 4.15).
Large Gradients (para 4.15.1).
PREPARING THE SURVEY GRID PROFILE (para 4.16).
Survey Track (para 4.16.1).
SETTING UP A TIE LINE (para 4.17).
Extending the Tie Point Grid (para 4.17.1).
Displaying the Tie Point Drift (para 4.17.2).
SETTING UP A LOOP SURVEY (para 4.18).
FIELD MEASUREMENTS (para 4.19).
Erasing Field Data Records (para 4.20).
Choosing Recording Format (para 4.21).
Skipping Stations (para 4.22).
Intermediate Points (para 4.23).

4.8.1 General Procedures. The following procedures apply to keying operations which can be carried out at any time, and do not necessarily relate to specific survey procedures. These procedures also apply to the OMNI IV regardless of the software level (ie, field mag, base station and gradiometer).

4.8.2 Starting the Unit. When a charged battery is connected, the OMNI IV is ready to operate.

a. Set the MODE selector to the required position.

b. Press the READ key. This turns on the power to the system.

4.8.3 Changing a Parameter. Whenever a parameter key is pressed, the stored value of that parameter will apppear on the display. To change a parameter value (such as TIME, DATE, LINE, POS, LINE SPACING, POS SPACING, or TUNING):

a. Press the parameter key (eg, TIME).

b. Then press the CHANGE key. This causes the displayed value to flash on and off, indicating that the value may now be changed.

c. If it is applicable to the value, press the +/− key.

d. Press the required numeric keys in sequence from left to right to make up the new number.

NOTE

As each digit is entered, the numeric stops flashing.

e. When the new parameter value is being displayed, verify its accuracy, then press the ENTER key. The new value then enters the OMNI IV memory.

f. Press the parameter key again and verify on the display that the correct value has been entered and stored.

NOTE

If a keying error occurs when entering the numerics, press the CHANGE key again.

4.8.4 TEST Mode. The OMNI IV circuitry and software includes a built-in test capability which confirms the validity of field measurements, data acquisition and record processing. The following tests are available:

a. Total field test.

b. Error calculation test.

c. Software diagnostics.

NOTE

1. Repeated test readings performed under c. allow for hard-copy verification that the OMNI IV is operating properly.

2. About 85% of the OMNI IV electronics is tested in the TEST mode.

3. These tests may be repeated several times with the OMNI IV in any position to verify repeatability. Once repeatability has been confirmed, the OMNI IV is now ready to be used for a survey provided that certain survey grid parameters (or base station recording interval) are pre-programmed into the memory. At random intervals during a survey, the total field test and error calculation test may be repeated to verify that the OMNI IV is operating properly during the actual survey.

4.8.5 Total Field Test. Proceed as follows:

a. Set the MODE selector to TEST, then press the READ key. On the display, the EDA logo and GAMMA descriptor will appear. Also the word TEST will flash on and off.

b. The OMNI IV will take a synthetic reading and complete a total field test. The display will indicate a six-digit numeric value which represents the test total field. The displayed field value is 56369.7 GAMMA.

NOTE

As a result of this test, there is an 80% probability that the OMNI IV will operate satisfactorily.

4.8.6 Error Calculation Test. This is a total field (or gradient) error calculation test. Proceed as follows:

a. Leave the MODE selector on TEST.

b. Press the ERROR key once. The display must indicate a value of .00 GAMMA.

c. For gradiometers, (ie, software 5), press the GRAD key once. The display must indicate a value of .0 GAMMA GRAD.

d. Press the ERROR key once. The display must indicate a value of .00 GAMMA GRAD.

4.8.7 Software Diagnostics. A feature is included whereby the OMNI IV executes a complete verification program of the stored data by computing and storing the check sum pertaining to a data block. This feature enhances stored data validity. The check sum verification program checks if there are any sudden problems in the stored program memory.

The program memory diagnostics program is initiated in the DUMP mode. Proceed as follows for the DUMP mode:

a. Set the MODE selector to DUMP.

b. Press the READ key. Observe the display. The display indicates: NNNNNN (ie, a six digit number. Refer to para 4.10 for interpretation).

Note

For this test, it is necessary to know the unit serial number.

c. If the value during the display is correct, the program memory is satisfactory.

d. If the value during the display is alphanumeric or incomprehensible, record this because there is a problem in the program memory.

4.8.8 Making Initial Total Field Measurements. Proceed as follows:

a. Refer to the cardinal point decal on the end of the sensor to establish the North direction of the sensor.

b. When a remote sensor is used, ensure that the sensor is connected to the OMNI IV and keep the sensor as far away as possible from the OMNI IV.

CAUTION

Ensure that the sensor pole is not in the vicinity of magnetic objects or power lines.

c. Rotate the sensor to align it with magnetic North.

NOTE

The OMNI IV is very sensitive and capable of measuring variations of 0.05 gamma.

d. Set the MODE selector to TFM.

e. Press the READ key once. The OMNI IV will power-up automatically, then it commences to energize the sensors for a specific polarization period. During this period, the display will indicate a six digit total field value with the two most significant digits flashing on and off. For example, the display may indicate **58315.6 GAMMA** and digits **5** and **8** flash on and off. The six-digit value (58315.6 γ) is the last previously-stored value. The flashing **58** indicates that the OMNI IV is tuned to 58 × 1000 γ. This is also applicable when the MODE selector is set to the GRAD position.

f. After about 2 seconds, the display will be updated automatically. During the brief display update, an audio beep will be activated. The new numeric value on the display will be the actual total field reading obtained, eg, **58186.2 GAMMA**, when in the TFM mode, or eg, **2.7 GAMMA GRAD** when in the GRAD mode.

g. The actual total field or gradient value will remain on the display for about 8.5 seconds. After this, the audio beep is enabled for a period of 8 seconds to warn the operator that within another 8.5 seconds, the OMNI IV will shut itself off automatically.

h. During the entire display period, other parameter keys may be pressed to display the value for that parameter. Every time another parameter key is pressed, a new 17 second display period will be activated. The display periods for each parameter should be minimized to conserve battery energy.

4.8.9 Statistical Error Evaluation. When in the GRAD mode and after sensor polarization, the display is updated to indicate the value of the gradient. In relatively low noise and gradient free areas, errors of .03 to .07 gamma are typical for the total field value.

a. To display the error value of the gradient (lower) sensor, press the ERROR key. Both the **GAMMA** and **GRAD** descriptors will be displayed.

b. To display the error of the total field (upper) sensor, it is required to display the total field first by pressing the FIELD key.

c. Once the total field is displayed, the error of the total field can be displayed by pressing the ERROR key. Only the **GAMMA** descriptor will be displayed when displaying the total field error.

NOTE

In the gradient mode, only the error of the gradient (lower) sensor is stored.

4.9 PRE-SURVEY PROCEDURES

At the start of a survey day, prior to commencing operations, it is necessary for the operator to perform a start-up procedure. This is to ensure that stored data is not destroyed; that accurate measurements can be taken; and new data will be stored properly for retrieval later. Carry out the following procedures:

a. Identify the OMNI IV correctly by identifying the software that is resident in the instrument (ie, what modes of operation can be initiated and performed).

b. Capture Stored Data. Ensure that accumulated data stored from the previous day is dumped prior to initlalizing the OMNI IV.

c. Initialize the OMNI IV memories for the current day's survey activity.

CAUTION

1. Initialization of the OMNI IV clears any stored data from the memories, except tie-line data.

2. All OMNI IV magnetometers have a software flag at 12:00 midnight. Data cannot be collected and stored past 12:00 midnight without first dumping (or clearing) the stored data which was collected prior to midnight. This ensures that data which has been collected on a specific day can only be corrected for diurnal variations which have been measured (or computed) during the same specific day.

4.10 Identifying the Instrument

If there is more than one instrument being used in a survey, (eg, base station and total field magnetometers), it will be necessary to identify the capabilities of the unit by determining the software that is resident in the unit.

a. Turn the MODE selector to DUMP.

b. Press READ key. A six-digit number will be displayed on the LCD.

eg. 0 6 3 1 0 8

- Unit serial No.
- Software code
  - 3 = Total field or tie-line magnetometer
  - 4 = Base Station
  - 5 = Gradiometer
- Software revision and options code (For EDA use)

4.11 Erasing the Previous Day's Survey Data

At the beginning of each day's survey, it is necessary to initialize the OMNI IV by entering an operator's code 'OPNNNN' where N = a numeric. If data is still in the memory from the previous day, it will be erased when the operator's code is entered.

4.12 Initializing the OMNI IV

The OMNI IV has two memories for storing data: The tie-line memory and the field memory.

4.12.1 Tie-Line Memory. This memory stores data for up to 100 tie points and the base reference. This data in memory is not destroyed until the operator initializes the tie-line memory. The data is retained for tie-line survey use until it is initialized regardless of the number of days the survey might take. This memory is entered in the TIE mode.

4.12.2 Field Memory. This second memory stores the field data. It is initialized when the operator's code number has been entered and the first total field reading has been stored. This memory is entered in the TFM, GRAD, or AUTO modes.

CAUTION

After this point, the prior day's field data is lost and cannot be dumped.

4.12.3 Initializing the Field Memory. This procedure is carried out at the beginning of each survey day, (and after valid data has been dumped correctly).

To enter the operator's code (ie, to clear and initialize the field data memory), proceed as follows:

a. Turn MODE selector to TFM (for total field (3)); to GRAD (for gradiometer (5)); to AUTO (for base station (4)); or to TIE (for tie-line (3 or 5)).

b. Press one of the following keys: SPOT RECORD, MULTI RECORD or AUTO RECORD. The display will flash OPNNNN accompanied by an audible beep.

c. Press the CHANGE key, then press the following numerics, where M = 9 for alkaline batteries and any other digit for rechargeable batteries; and the Ns represent a number which identifies the operator.

3MNN for total field
4NNN for base station
5MNN for gradiometer

NOTE

1. The gradient operator code can be used for the tie-line survey.

2. If a keying error occurs when entering the numerics, press the CHANGE key again and re-enter the digits.

3. At any time that it is necessary to enter a new operator's code, the accumulated field data must be saved by dumping first. Once the new operator's code has been entered and a reading taken, recovery of the data stored prior to entry of the new operator code will not be possible.

4. The second digit of the operator code must be a 9 if alkaline batteries are used.

4.12.4 Initializing the Tie-Line Memory. This procedure is only carried out after a survey has been completed. It is required in order to enter new tie-line data for the basis of a new survey. Proceed as follows:

a. Turn the MODE selector to DUMP.

b. Press the READ key, then press the AUTO RECORD key. The display will flash HPNNNN accompanied by an audible beep.

c. Press the CHANGE key, then the digits 8888, followed by ENTER and CLEAR.

d. The tie-line data will be cleared if the OMNI IV turns off.

4.12.5 Starting the Base Station Magnetometer. Proceed as follows:

a. Set the MODE selector to AUTO.

b. Press the READ key. A reading of the total magnetic field will be taken while the displayed digits flash on and off. When the reading is completed, a beep sounds and the reading is displayed.

c. When a reading has been attempted, if the magnetometer is not tuned within the acceptable tolerance, (referred to as detuned), there will be a beeping sound and a flashing field reading with the TUNE descriptor flashing. (Refer to para 4.14.2 for the manual tuning procedure.)

d. Press the SPOT RECORD key. The base station magnetometer will beep and the display will read OPNNNN. Enter the correct operator's code (commencing with the numeric 4) as described in para 4.12.3 (ie, initializing the field memory).

CAUTION

If the data stored in the base memory of the previous day has not been dumped, the OPNNNN will not be requested. Entering an OP code will erase the data.

e. After the OP number has been entered, press SPOT RECORD again.

f. If the base station recording time interval has not been entered since memory initialization took place, it will commence at this point. The OMNI IV will beep and the previous time interval will be displayed in flashing digits. Change and enter value as necessary (between 5 seconds and 60 minutes).

g. Press SPOT RECORD again. The display will show RP001 signifying that the memory is ready to store the first reading.

h. Press SPOT RECORD again. The first reading will be stored in memory and identified as RP001 and the OMNI IV will be turned off automatically.

j. Press the READ key to start the base station again.

NOTE

From this point, the base station will function automatically. After the preprogrammed recording interval has elapsed, the OMNI IV turns itself on; displays the previous total magnetic field reading with flashing digits until the polarization sequence is complete; then, after storing the reading and incrementing the RP number, it shuts itself off. This is the cycling sequence.

4.12.6 Starting the Gradiometer. Proceed as follows:

a. Set the MODE selector to GRAD.

b. Press the READ key. The OMNI IV turns on and a total field reading is being processed while the first two displayed digits are flashing. When the reading is completed, there is a beep and the reading is displayed.

NOTE

If the OMNI IV is detuned, it will indicate this by a sequence of beeps accompanied by a flashing display of the previous reading. Refer to para 4.14.2 for the manual tuning procedure.

c. Press SPOT RECORD. This will cause the OMNI IV to beep and OPNNNN will be displayed. For the gradiometer, enter 5 followed by a 9 (if alkaline batteries are being used) or another digit if not; followed by the two operator digits.

NOTE

As soon as the new OP number is entered, the field memory will be initialized, and any stored data in memory is cleared (ie, not retrievable).

d. Press MULTI RECORD. The position number will be displayed. If correct, press MULTI RECORD again to store the data.

4.13 Pre-Survey Programming

At the base station, the following programming tasks may be accomplished with the respective MODE selectors on the base station magnetometer and total field (or gradient) magnetometer both set to TEST.

a. Clock synchronization (including TIME and DATE setting).

b. Reading synchronization (if desired).

c. For the field magnetometer (or gradiometer), programming the initial LINE, LINE SPACING, POS, and POS SPACING.

d. On the base station, the base LINE and POS readings can be entered as a reference record, then the time SPACING interval can be reprogrammed for the desired automatic recording interval cycle.

e. Then memory initialization can be completed with the entry of the OP number.

4.13.1 Real-Time Clock. The OMNI IV includes a programmable real-time clock for synchronizing readings and for recording the times of field readings. The clock also includes the date. However, this does not include leap year compensation. It is suggested that a digital wrist watch be used to set the time at the start of a survey and to synchronize the clocks within the base station magnetometer.

NOTE

If the date is changed, the time has to be re-entered as well.

To set the time, the following procedure must be followed:

a. The time can be set in any position of the MODE selector.

NOTE

Consider an example in which the display indicates the following time: **14:26:13**. It is the intent to set the clock at **14:28:00**. Therefore, it will be necessary to enter this set time (ie, 14:28:00) when the digital wrist watch displays 14:27:59. **For convenience, the real-time clock can only be programmed for hours and minutes; not seconds.** Therefore, allow at least one minute prior to the time that the entry will be made.

b. Press the CHANGE key and observe the display. Note that the numerics 1 and 3 in the seconds column have been set to zero automatically, ie, **14:26:00**.

c. The display flashes on and off indicating that it is ready to accept new values. Key in the following numerics in sequence: 1, 4, 2, 8. Observe the display to confirm that the new value is entered correctly. Do not enter any digits for seconds.

NOTE

Depending upon the time difference between steps b. and c., it is most probable that the display period will time out and the audio alarm will commence beeping. To extend the time display period, press the ENTER key followed by the CHANGE key again. It may be necessary to repeat this procedure several times during the time-setting process.

d. Now monitor the wrist watch until it displays **14:27:59** then, immediately, press the ENTER key. With some experience, the setting accuracy can be less than 1 second.

e. To confirm that the real-time clock has been set properly, press the TIME key twice and compare the time on the wrist watch with the time on the display.

NOTE

Synchronization of the base station and field magnetometers requires two operators to ensure synchronization accuracies of less than one second. Every time that a reading is taken, the computer reads the real-time clock and stores the time in memory. The time at which the real-time clock is read is defined as the time immediately after the processing of the sensor signal. The same procedure is used in the base station magnetometer. This method improves the record accuracy of the actual time that the total magnetic field was measured.

To synchronize OMNI IV clocks, it will be necessary to carry out this procedure simultaneously; or to set one **TIME** display, then use that display as the reference to set another **TIME** display.

To enter or change the date:

a. Press the TIME key. The time will appear on the display.

b. Press the TIME key again immediately. The time display will convert to a date display.

c. Consider, for example, that the displayed date is **85:08:19** (ie, 19th August 1985) and that the required setting is **86:04:12** (ie, 12th April 1986).

d. Press the CHANGE key. Then, in sequence, press the digits 8, 6, 0, 4, 1, 2.

e. Verify that the date is correct, then press the ENTER key.

f. Press the TIME key two times to recall the entered date to the display.

4.13.2 Reading Synchronization. If it is required to synchronize the field unit with the base station unit, the time spacing for the field unit must be re-entered.

a. Press the TIME key on the field unit.

b. Press the SPACING key to display the time interval.

c. If the time spacing is the same as in the base station unit, press the ENTER key.

d. If the time spacing is different from the base station unit, press the CHANGE key, then enter the correct time interval.

e. If no synchronization is required, enter a time interval of 00:00 in the field unit.

4.14 SYSTEM TUNING (Prior to Survey)

Whenever a total field measurement is to be taken for the first time, it is necessary to confirm that the predicted measurement will approximate the total field reading which has been stored previously.

If the difference between the actual total field strength and the last total field reading stored previously in memory is too large, the OMNI IV may not be capable of taking a proper reading. In such a case, the display will indicate that OMNI IV tuning is required. The **TUNE** descriptor will flash on and off and the audible alarm will be activated.

4.14.1 Retrieval of Last Previously Stored Total Field Value. To obtain the previous value of the total field, proceed as follows:

a. Press the RECALL key.

b. The display will indicate the record number of the last reading. The **DECAY** and **SENS** descriptors will flash to indicate that the unit is in recall.

c. Press the FIELD key and observe the display. The total field value which is the last stored total field value, will be displayed.

d. Press RECALL again to toggle back to the normal function.

4.14.2 Tuning the OMNI IV. Proceed as follows:

NOTE

As a general rule, when the difference between the local ambient field strength and the last stored total field strength is greater than ±15% relative to the local ambient field strength, the OMNI IV must be tuned manually.

a. Consider the example when the display indicates a last previously stored total field strength of **50500.7 GAMMA.**

b. Furthermore, in the example, it is estimated that the local ambient field strength is 59000 γ. By calculation; ±15% of 59000 γ = ±8850 γ.

NOTE

The OMNI IV might not be capable of tuning automatically from 50500 to 59000 gammas. Therefore, a closer value must be inserted in the two most significant digits that are used for tuning.

c. Press the CHANGE key. Observe the flashing total field value; 50500.7 indicating that the value may be changed now.

d. In sequence, press the following numeric keys: 5, 9. Observe that every time a numeric key is pressed, the associated corrected digit on the display stops flashing. This procedure is equivalent to a flashing cursor which travels from left to right.

e. After the changed numerics have been entered, confirm visually that the new entry is accurate, then press the ENTER key once. The OMNI IV is now tuned to 59 kγ.

NOTE

If an incorrect numeric is entered in step d., it is not possible to cancel or edit the entry while in the process of entering. The procedure for correcting an entry is the same as for entering a new value.

f. Assume that the numerics 5, 3 have been entered and suddenly, it is evident that a 3 has been entered erroneously instead of a 0. Press the ENTER key to enter the incorrect value. Then press the CHANGE key again and repeat step d.

4.14.3 Incomprehensible Displays. Assuming that the operator has no historical knowledge regarding the OMNI IV when the FIELD key is pressed in the TEST mode, it is possible that the display may also indicate an incomprehensible number which flashes on and off. When this occurs, the OMNI IV does not have a valid total field value in memory, (ie, the check sum for the total field is bad). In this event, the OMNI IV must be tuned to the approximate ambient local field.

a. Carry out the tuning procedure.

4.15 SYSTEM TUNING (During a Survey)

Consider the condition where there is a warning that the OMNI IV requires tuning during an actual survey. Although this is very unlikely, it is possible if a sudden large local gradient is encountered between one station on the grid and the next station. To cause this, the local gradient must induce a change which will be larger than ± 15% of the last stored total field strength value.

If this occurs, the total field value displayed will flash on and off, the **TUNE** descriptor will appear, and the audio alarm will beep.

Although there is no indication how large the gradient and the sign of the gradient is, it is suggested that the tuned value be increased by + 10% of the last value.

The flashing total field value on the display is the last stored value which is valid. Therefore, it must be used as the reference to calculate and add + 10%.

a. As an example, assume that the flashing total field value is **62350.3 GAMMA.**

b. By calculation, 10% of 62350 γ is approximately 6000 γ.

c. Add 6000 γ to 62000 γ. The new tuning value shall be 68 kγ, (ie, 68 × 1000γ).

d. Press the CHANGE key. In sequence, enter the following numeric digits: 6, 8.

e. After the changed numerics have been entered, confirm visually that the new entry is accurate, then press the ENTER key once.

NOTE

When the OMNI IV is operated in either the GRAD or TFM mode and the OMNI IV requires tuning during an actual survey, a new reading will be taken automatically following tuning when the FIELD key is pressed. This is a feature of convenience for the operator.

f. To obtain a reading immediately after tuning, press the FIELD key.

g. Observe the display for about 3 seconds. The display will indicate 68000.0 and the two most significant digits **6** and **8** will be flashing on and off. Flashing of the two most significant digits is confirmation that the OMNI IV is tuned to 68 × 1000 γ.

h. After this, a new total field reading will appear on the display, eg, **66100.8 GAMMA.** For this example, the OMNI IV was capable of tuning to the new total field value. The OMNI IV is now tuned to 66100 γ for the next reading.

j. In the event that the OMNI IV indicates that it cannot tune even after it was tuned to 68000 γ, the operator might re-tune the OMNI IV to 72000 γ using the same procedures as outlined previously.

k. If no results are obtained after two or three attempts, tune the OMNI IV to − 10% of 62350 γ or 62000 − 6000 = 56000 γ. The reason for this is that the OMNI IV may be subject to a large negative gradient relative to the local ambient field.

NOTE

In general, the operator might have to re-tune the OMNI IV several times in order to discover the new total field value. Field experience has indicated that usually the OMNI IV needs re-tuning only once.

4.15.1 Large Gradients. In the tuning procedures, it was indicated that the range of automatic tuning that the OMNI IV is capable of handling is approximately ± 15%. It must be noted that this is not an instrument fault or design limitation, but an indication of large gradient variations and this is a significant observation in the survey. One useful procedure is to monitor the DRIFT function (ie, the change in gammas between the previous total field reading and the present reading). To display the drift:

a. Press the DRIFT key following any reading. This option remains in effect until changed by pressing the FIELD key.

NOTE

Monitoring drift makes it easy for the operator to detect large horizontal gradients, which may require additional readings (SPOT RECORDS) at intermediate fill-in stations along the survey line. The drift indication also checks data repeatability at any given station.

4.16 PREPARING THE SURVEY GRID PROFILE

A sample grid configuration is shown in Figure 3-1.

Initially, to set up the imaginary grid pattern, a definition is required regarding the direction (or ground track) that the operator must travel. Consider an example where the operator starts from a specific reference position and proceeds to the South along a specific line. See Figure 4-5. The starting position will have a reference of P 0000. If the operator proceeds initially to the South, this requires a minus (−) sign notation for the position spacing value according to the example grid. The linear units of measurement for spacing are arbitrary since they merely represent a means of identifying magnetometer measurements geographically by coordinates when the data is printed out. It is essential, however, that the spacing keyed into the OMNI IV be identical to the actual spacing established in the field survey.

As an example, consider the line spacing of 25 metres shown in Figure 4-5.

To set the initial line reference and the grid spacing between adjacent lines into the OMNI IV:

a. Set the MODE selector to TEST.

b. Press the READ key and observe the displayed total test field (ie, 56369.7 GAMMA).

c. Press the LINE key. The actual display is irrelevant. Then press the CHANGE key. The displayed value will commence flashing on and off.

d. Press the ± key once or twice until the first two positions on the display indicate L and blank.

NOTE

The display does not indicate a + sign. Instead a blank will be observed.

e. Press the 0 key four times to give a display of L 0000. This is the primary reference line.

f. Press the ENTER key to enter the new value.

g. Press the LINE key again. Confirm that the entry is correct.

h. Press the SPACING key. The INTV descriptor will appear to indicate that this is the line spacing.

j. Press the CHANGE key. The displayed value will commence flashing on and off.

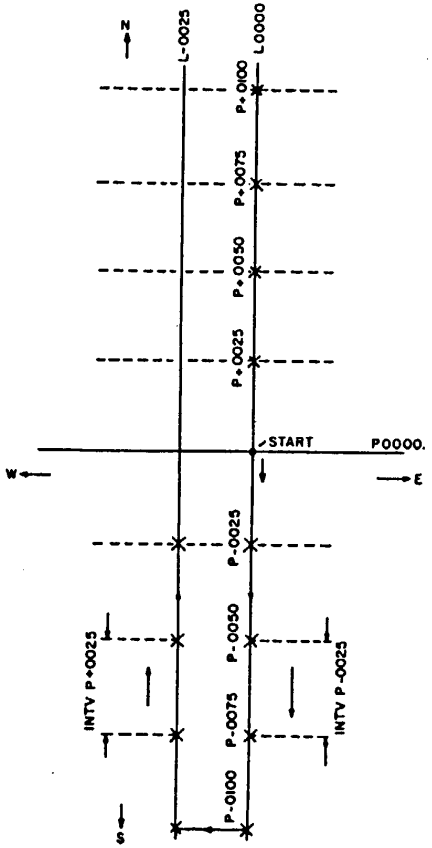


Figure 4-5 Start of Survey Profile

k. Press the ± key once so that the first two positions on the display indicate L–.

m. Press the following numeric keys in sequence 0, 0, 2, 5 to give a display of INTV L–0025 (line spacing).

n. Press the ENTER key to enter the new line spacing value.

p. Press the SPACING key to recall the line spacing display. Confirm that the entry is correct.

A similar procedure applies for position (station) numbering and spacing. The position ordinates are set up using the same procedure but operating the POS key instead of the LINE key. In this example, the position spacing is also set at –0025 intervals and the zero reference is P 0000.

NOTE

To summarize the quadrant sign, tracking to the North and East are considered positive.

4.16.1 Survey Track. A typical survey track is shown in Figure 4-5. Consider the path traversed during the example survey. The origin is identified on the figure as 'start' and has the grid coordinates L 0000, P 0000.

a. When the operator starts at the origin, if the LINE key is pressed, the value L 0000 will be displayed. If the POS key is pressed, the value P 0000 will be displayed. When a reading is taken at this location, the reading must be stored in the TFM mode under MULTI RECORD key control.

b. When the first position south of the origin is reached, a position update is required. Press the UPDATE key. The computer combines the P 0000 position entry with the INTV P–0025 spacing entry to give the new position entry of P–0025.

c. When a total field reading is taken at this location, the position entry P–0025 is recorded together with the line entry L 0000.

NOTE

Alternatively, if the AUTO RECORD key is used, the position is updated automatically.

d. As the survey continues Southward, this procedure decrements the position number from P–0025 to P–0050, then from P–0050 to P–0075 by using the UPDATE key, when the data is to be stored.

e. In the example, when the operator reaches P–0100, the track turns Westward to the next line, then it continues Northward. Therefore, the position number remains unchanged, but the line must be updated. When the next station Westward is reached, press the LINE key, followed by the UPDATE key. The computer combines the L 0000 line entry with the INTV L–0025 spacing entry to give the new line entry of L–0025. The position is not updated here.

f. When the total field reading is taken at this location, the entries L–0025 and P–0100 will also be recorded in the manual record mode. The reading must be stored under MULTI RECORD control because only the line number is updated.

g. The track now turns Northward, therefore the position interval must be changed from –0025 to +0025. Press the POS key, then press the SPACING key. Press the CHANGE key, then press the ± key. Press the ENTER key. The position spacing display should read INTV P 0025.

h. As the track progresses Northward, the negative position numbers will change in increments of 0025 until P 0000 is reached. Then they will become positive, eg, P 0025, P 0050, etc.

4.17 SETTING UP A TIE LINE

For this survey method, there is no base station. Therefore, in order to utilize the data correction algorithms in the OMNI IV microprocessor, it is necessary to establish a base station (or base point) reference as well as a series of tie points (usually along the same tie line). Tie line(s) are usually located along the strike length of the survey grid, and either along the baseline or at the ends of grid lines.

NOTE

The tie-point readings form a bench mark for computing diurnal variations so that the survey readings can be corrected. Therefore, at each reading, the correct survey grid coordinates (ie, line and position) must be recorded simultaneously. Erroneous data must be deleted or readings must be repeated. Readings at the base reference point can be stored in the tie-line memory by selecting the TIE mode and entering recordings using the SPOT RECORD key. Check the reading quality display (ie, SENS and DECAY) for each reading. Discard a poor reading by pressing the OFF key. Then take a repeat reading by using the SPOT RECORD for the base reference point or the MULTI RECORD for the tie points. If the quality is poor for multiple readings, move the tie point to a location where good quality and repeatability can be obtained. The location of the base reference point should be selected in an area with low gradient.

Refer to Figure 3-2 and proceed as follows:

CAUTION

Prior to taking any reading, ensure that the sensor is oriented with its North mark (engraved on the sensor) facing North. Ensure that the sensor interconnect cable is attached securely to the OMNI IV. Keep the sensor at arm's length when taking a reading.

a. At the base reference point:

(i) Set the MODE selector to TIE.

(ii) Press the READ key. The display shows the total field reading at the base point.

(iii) Press the SPOT RECORD key once. The audio beep will commence with the display flashing OPNNNN, if this is the first reading to be stored for the day.

NOTE

The first digit designates the application in which the OMNI IV was last used (eg, 3 = field magnetometer, etc).

(iv) For the total field magnetometer application, press the CHANGE key. The beeping will stop.

(v) Enter the OP number (ie, 3 followed by three additional digits).

NOTE

These can be any three digits but are usually assigned to identify the operator with the data stored.

(vi) Press the ENTER key.

(vii) Press the SPOT RECORD key. The display should show LP 9999 to indicate a base reference point.

(viii) Press the SPOT RECORD key a second time to enter the reading into the special tie-line memory.

b. Proceed to the first tie-point position, then:

(i) Keep the MODE selector set to TIE. Press the READ key. After the polarization, the display shows the field reading at the tie point.

(ii) Press the MULTI RECORD key. This does not advance the tie-line number.

(iii) The display will show the tie-line number. The flashing L denotes that this is a tie-line reading.

(iv) Verify that this is the correct line number. If not, refer to para 4.16 to program the grid coordinates. Change the reading to the correct line and position number as required.

(v) Press the MULTI RECORD key again to store the reading and grid coordinates in the special tie-line memory.

c. Proceed quickly to the next tie-point position along the tie line.

(i) Press the READ key. The display should show the field reading at the previous tie point during polarization, followed by the new field reading.

(ii) Press the AUTO RECORD key. This advances the line number.

(iii) The display will show the next sequential line number. The flashing L denotes a tie-line reading.

(iv) Verify that this is the correct line number for the tie point.

(v) Press the AUTO RECORD key again to store the reading and grid coordinates in the special tie-line memory.

d. Repeat step c. along the entire length of the tie line or at the designated tie points.

CAUTION

Proceed as quickly as possible to minimize the effects of diurnal variations. If scattered tie-point locations are selected, it will be necessary to correct the line and position displays. Refer to para b. and especially step (iv).

e. When all tie points have been recorded, return to the base reference point, then:

(i) Press the READ key in the TIE mode. The display will show the total field reading at the base reference point.

(ii) Press the SPOT RECORD key. The display will show LP 9999, denoting a base reference point.

(iii) Press the SPOT RECORD key again. The reading and grid coordinates will be stored in the special tie-line memory. Therefore, there will be two readings entered for the base reference point: One at the beginning and one at the end of the tie-point reference measurements. These two readings will indicate the diurnal variation between them and the elapsed time between the two readings.

NOTE

The tie-line loop is now complete and stored in memory for the entire survey. On subsequent survey days, the first and last reading of the day must be recorded at the base reference point or at any given tie point stored in memory. These must be stored in the TIE mode.

4.17.1 Extending the Tie-Point Grid. If the survey grid is extended, it is possible to increase the number of tie-line references in the tie-line memory at any time or day during the survey. Repeat steps a. through e. at the new tie-point positions. Previous tie-line readings will remain in memory until they are erased by the HP8888 dump code. (See para 4.12.4.)

4.17.2 Displaying the Tie-Point Drift. The diurnal drift between the two most recent measurements at the tie points can be monitored as follows:

a. Ensure that the MODE selector is set to TIE.

b. Press the DRIFT key twice. The display will show the GRAD descriptor and the value will be the drift between the last two consecutive tie points.

CAUTION

Do not initialize the tie-line memory again until the survey is complete.

4.18 SETTING UP A LOOP SURVEY

This method is almost the same as the tie-line method except that, instead of selecting and recording numerous tie point references, only the base reference point is used as a bench mark. This method is applicable to smaller survey areas in which it is practical to return to the base reference point frequently to take subsequent readings. The looping survey diagram is shown in Figure 3-3.

CAUTION

Before the looping method can be used, the tie-line memory must be initialized. (See para 4.12.4.) Then the base reference point only will be stored. If it is not initialized, existing tie-point information will be picked up and used in computations.

4.19 FIELD MEASUREMENTS

If this is the first day of the survey, the OP number will not have been programmed. On subsequent days, the OP number will have to be programmed at the start of each day after the survey data from the previous day has been dumped.

NOTE

When measuring the total field (TFM mode), use the OP3NNN number. When measuring gradient (GRAD mode), use the OP5NNN number.

CAUTION

At each station, check the reading quality and repeatability by observing the **DELAY** and **SENS** displays. Also monitor the drift between consecutive stations. Discard any bad or doubtful readings by pressing the OFF key. Then take a new reading.

To take field measurements, proceed as follows:

CAUTION

It is not possible to correct erroneous coordinates once the data is stored in memory except by erasing the previous reading. (See para 4.20.) This also applies to computations which utilize values obtained from an address identified by LINE and POS coordinates.

a. Upon arriving at a station, check the surveyed line and station number and compare them with the LINE and POS numbers programmed into the OMNI IV.

b. Position the sensor correctly. Set the MODE selector to TFM.

c. Press the READ key. The previous total field reading will appear in flashing digits while the reading cycle is being completed.

NOTE

If the reading cycle has been synchronized with the base station unit, (ie, the readings occur at the same time that the base station takes a reading), a count-down to the next read cycle will appear first. (See para 4.13.2.)

d. Press the MULTI RECORD key. The display shows the position number. Verify that this position or station number is correct for where the reading is to be stored. Then press the LINE key. Verify the correct line number. (If not, reprogram these values.) If the line and position numbers are correct, press the MULTI RECORD key twice to store the first total field reading of the survey.

e. Proceed to the next survey station.

f. Press the READ key. The flashing display shows the previous total field reading until the new reading cycle is completed. Then it shows the reading for the current location.

g. Press the AUTO RECORD key. The display shows the updated station number.

h. Press the AUTO RECORD key again to store the new data.

j. Repeat steps e. through h. at each position along the survey grid line.

NOTE

If the line and position of a survey location correspond to the tie-line point, the P or P– on the display will flash to indicate a tie-point station.

k. When the end of a survey grid line is reached, it is necessary to transfer to the next line without changing the position number, then to change the spacing sign. Refer to para 4.16.1 for this procedure.

4.20 Erasing Field Data Records

To erase data from memory, one record at a time, proceed as follows:

a. To erase tie-line readings, set the MODE selector to TIE. To erase field readings, set to TFM, or to GRAD (for readings in gradiometer mode).

b. Press the READ key and wait 2 seconds until the cycle is complete.

c. Press the RECALL key, then check the field, time, line or pos to verify that this is the reading to be erased.

d. Press the CLEAR key.

e. Repeat the sequence in a. through d.

NOTE

The block erase feature works in the data-dump mode also. Similarly, it is necessary to select RECALL.

4.21 Choosing Recording Format

Depending upon the character of the survey grid and the survey conditions, the operator can choose from three recording formats.

a. SPOT RECORD. This key is used where grid coordinates are not required. The data block is identified by an RP number. Do not use this key in the TIE mode as this will indicate a base reference station.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the SPOT RECORD key once. The display assigns the next sequential record number (RP) to the reading.

(iii) Press the SPOT RECORD key again. The reading is stored in the memory data block.

b. MULTI RECORD. This key is used to store many readings at the same position (station) number.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the MULTI RECORD key once. The display shows the previous position number.

(iii) Press the MULTI RECORD key again. The reading is stored in the memory data block.

c. AUTO RECORD. This key is used to update the position (station) number at pre-programmed spacing increments.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the AUTO RECORD key once. The display updates the position (station) number to the next position by adding (or subtracting) the spacing interval.

(iii) Press the AUTO RECORD key again. The reading is stored in the memory data block.

4.22 Skipping Stations

Where it is not possible to collect data at a given position (eg, because of water or topographical obstruction), the position (station) number can be updated without storing a field reading. Proceed as follows:

a. Proceed around obstruction to where a field reading can be taken.

b. Press the READ key. The total field reading will be displayed.

c. Press the POS key.

d. Press the UPDATE key once, or for as many times needed to arrive at the new position number.

e. At the correct position number, press the MULTI RECORD key twice to store the reading in the memory data block.

4.23 Intermediate Points

On a survey grid with 25 m spacing increments, it is desirable to record data at the mid points (eg, 12.5 m, 37.5 m, etc). The display is only capable of indicating the two digits, therefore, when a space of 12.5 m is required, only the digits 12 are entered. Then the display shows 00:12 with the colon and decimal point together and flashing. This readout actually means 12.5 m even though the .5 is not displayed.

SECTION 5

DATA RETRIEVAL

5.1 RETRIEVAL CONCEPTS

Data stored in the OMNI IV memories can be processed and dumped using various program directives. These directives (access codes) are entered through the keypad.

The data is dumped by connecting the OMNI IV to an applicable peripheral device such as a printer or computer. The connection is made through a cable which is attached to the connector on the rear of the console. (When the OMNI IV is chest mounted, the data dump connector is on the underside of the console adjacent to the operator's right hand.)

5.1.1 Diurnal Correction of Data. When the OMNI IV is used in the tie-line or looping mode, the data stored in the special tie-line memory file is merged with the field measurements stored in the field data memory file. The internal microprocessor then computes the diurnal variation in the earth's magnetic field for each survey grid coordinate and corrects the field data file using the tie-line file. No manual computations are required. This correction process takes place during the data dump.

In addition to the self-correcting feature for data using the tie-line or looping method, the OMNI IV can also include a base station (or autocycling) option. In this method, the base station records the total field reading at a single location but at precise time intervals. During the data dump, the raw data from the field magnetometer is dumped to the base station magnetometer where it is merged. The internal microprocessor then computes the diurnal variation in the earth's magnetic field for each survey grid coordinate by comparing the times at which readings were taken and computing any mid-interval values. This is most useful in Northern latitudes where more detailed monitoring of the diurnal variations is required. This correction process takes place during the data dump.

Another correction which can be applied during the data dump is the programmable datum or the programmable reference field. In this application, the OMNI IV is programmed to remove a designated datum (or value) from the field measurements. Removal of this coarse background value facilitates plotting and interpretation of data.

5.1.2 Data Dump Configurations. There are basically two equipment interconnection configurations:

a. The OMNI IV can be connected directly to the printer or computer.

b. An OMNI IV field magnetometer can be connected to an OMNI IV base station magnetometer and then to a printer or computer.

NOTE

In order to hook up an OMNI IV to a computer, the user must be able to control the DTR (data terminal ready) line and the CTS (clear to send) line on that computer.

5.1.3 Interface Cables. EDA produces a series of optional data cables for interconnecting the OMNI IV to various printers and computers. The list is given in Table 2-1. Each data cable includes a communications buffer board custom designed to suit the interface requirements. See Figures 5-5 thru 5-10.

5.2 Dump Set-Up

Using the applicable interface cable interconnect the OMNI IV to the printer or computer. (Computer handshaking procedures are beyond the scope of this manual.)

5.2.1 Access Code. Data stored in memory in the OMNI IV is retrieved by entering a four-digit access code. The composition of the code is as follows:

2 3 0 0

Always zeroes.

First Digit: Type of data to be retrieved.

1 = Raw data dump. The contents in the memory are dumped directly to the recording device.

2 = Corrected data where:

a. The OMNI IV can correct its own total field data when used as a tie-line magnetometer.

b. The OMNI IV is used as a base station to read raw data from the field magnetometer, then to correct it by linear interpolation.

3 = Verification. The field data is dumped through the base station without correction being applied. Rerouting of the data dump cable is not required.

4 = Tie-point drift. The diurnal drift for the individual tie points is retrieved by subtracting the programmable reference field.

5 = Corrected tie-line data. The tie-line data file is dumped directly.

Second Digit: Formats the line number with a compass bearing.

0 = Standard +/− data presentation.

1 = North (+) or South (−).

2 = East (+) or West (−).

3 = This is a fixed format for additional computer processing. It is operable only in PROG mode.

5.3 DATA DUMP PROCEDURES

Proceed as follows:

a. Interconnect the OMNI IV(s) with the applicable peripheral device.

b. Carry out the appropriate procedures related to the peripheral device.

5.3.1 Initiating a Raw Data Dump. Figures 5-1 and 5-2 show sample printouts of uncorrected total field data and base station data. Figure 5-3 shows tie-point drift and tie-line data. Proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

(i) Key in the number 1 to dump the raw data.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0 if required.

(iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key to begin the new data transfer.

```
EDA  OMNI IV "Tie-line" Mag  Ser #14015
TOTAL FIELD DATA (uncorrected)
Date:  4 AUG 83
Operator:  5012
Datum Subtracted:        0.0
Bat: 15.6 Volt    Lithium Bat: 3.3 Volt
Last time update:    1 AUG 83  15:22:00
Start of print:      4 AUG 83  18:29:43

  LINE 54+00 N    DATE  4 AUG 83      #1
POSITION  FIELD  ERR   DRIFT   TIME    DS
 31+00 E 57508.4 .10     0.0 15:00:35 00
 31+50 E 57506.3 .08     0.0 15:00:43 00
 32+00 E 57505.0 .08     0.0 15:00:47 00
 32+50 E 57501.9 .10     0.0 15:00:51 00
 33+00 E 57504.1 .08     0.0 15:00:56 00
 33+50 E 57503.1 .07     0.0 15:01:00 00
 34+00 E 57511.1 .07     0.0 15:01:05 00
 34+50 E 57514.9 .09     0.0 15:01:09 00
 35+00 E 57511.1 .08     0.0 15:01:17 00
```

Figure 5-1 Uncorrected Total Field Data

```
EDA  OMNI IV "Tie-line" Mag  Ser #31005
BASE STATION DATA
Date:  4 AUG 83
Operator:  4007
Reference Field: 57500.0
Line 20+00 N     Position 30+25 E
Bat: 12.1 Volt   Lithium Bat: 3.2 Volt
Last time update:   1 AUG 83  15:22:00
Start of print:     4 AUG 83  18:59:53

  TIME     FIELD    CHANGE    DRIFT   RECORD
15:00:33 57517.8     0.4      17.8       1
15:00:38 57508.6    -9.2       8.6       2
15:00:43 57511.7     3.1      11.7       3
15:00:48 57510.5    -1.2      10.5       4
15:00:53 57518.0     7.5      18.0       5
15:00:58 57525.9     7.9      25.9       6
15:01:03 57514.9   -11.1      14.9       7
15:01:08 57508.4    -6.4       8.4       8
15:01:13 57512.9     4.5      12.9       9
15:01:18 57512.7    -0.2      12.7      10
```

Figure 5-2 Base Station Data

```
EDA  OMNI IV "Tie-line" Mag  Ser #14015
TOTAL FIELD DATA (uncorrected)
Date:  4 AUG 83
Operator:  5012
Datum Subtracted:        0.0
Bat: 15.6 Volt   Lithium Bat: 3.3 Volt
Last time update:   1 AUG 83  15:22:00
Start of print:     4 AUG 83  18:29:43

  LINE 54+00 N    DATE  4 AUG 83      #1
POSITION  FIELD   ERR   DRIFT    TIME    DS
 31+00 E 57508.4 .10     0.0 15:00:35 00
 31+50 E 57504.3 .08     0.0 15:00:43 00
 32+00 E 57505.0 .08     0.0 15:00:47 00
 32+50 E 57501.9 .10     0.0 15:00:51 00
 33+00 E 57504.1 .08     0.0 15:00:56 00
 33+50 E 57503.1 .07     0.0 15:01:00 00
 34+00 E 57511.1 .07     0.0 15:01:05 00
 34+50 E 57514.9 .09     0.0 15:01:09 00
 35+00 E 57511.1 .08     0.0 15:01:17 00
```

Total Field Data (Uncorrected)

Figure 5-3 Tie-Point Drift and Tie-Line Data

5.3.2 **Correcting Total Field Data.** Figure 5-4 shows a sample print-out of OMNI IV corrected total field data. To correct total field data using the tie-line or looping method, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

(i) Key in the number 2 to dump corrected data.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0.

(iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key. The tie-line memory data is then merged with the field data and self correction begins.

```
EDA  OMNI IV "Tie-line" Mag  Ser #14015
TOTAL FIELD DATA (base stn. corrected)
Date:  4 AUG 83
Operator:  5012
Datum Subtracted:  57000.0
Bat: 15.6 Volt   Lithium Bat: 3.3 Volt
Last time update:   1 AUG 83  15:22:00
Start of print:     4 AUG 83  18:39:53

BASE STATION  Ser #31005
Date:  4 AUG 84
Operator:  4012
Reference Field: 57500.0
Line 20+00 N     Position 30+25 E
Bat: 12.1 Volt   Lithium Bat: 3.2 Volt
Last time update:   1 AUG 83  15:22:00
Start of print:     4 AUG 83  18:39:53

  LINE 54+00 N    DATE  4 AUG 83      #1
POSITION  FIELD   ERR   DRIFT    TIME    DS
 31+00 E  494.3 .10     14.1 15:00:35 00
 31+50 E  494.6 .08     11.7 15:00:43 00
 32+00 E  494.3 .08     10.7 15:00:47 00
 32+50 E  486.9 .10     15.0 15:00:51 00
 33+00 E  481.4 .08     22.7 15:00:56 00
 33+50 E  481.6 .07     21.5 15:01:00 00
 34+00 E  498.9 .07     12.2 15:01:05 00
 34+50 E  505.6 .09      9.3 15:01:09 00
 35+00 E  498.4 .08     12.7 15:01:17 00
```

Figure 5-4 OMNI IV Corrected Total Field Data

5.3.3 Correcting Total Field Data Using a Base Station. Figure 5-2 shows a sample print-out of base station data.

On both the base station OMNI IV and the field OMNI IV, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

On the OMNI IV used as a field magnetometer or gradiometer:

a. Press the CHANGE key, then enter the required access code:

(i) Key in the number 1 to send raw field data to the base station unit.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0 if digits were not 0, 0.

(iv) Press the ENTER key.

On the OMNI IV used as a base station:

a. Press the CHANGE key, then enter the required access code:

(i) Key in the number 2 to correct the field unit data.

(ii) Key in the same second digit as in a.(ii) above.

(iii) Key in 0, 0 if required.

(iv) Press the ENTER key.

For both OMNI IV units:

a. Power up the peripheral device.

b. Press both AUTO RECORD keys simultaneously. Data transfer will commence and data correction will occur in the base station OMNI IV.

5.3.4 Verification of Raw Field Data. On both the base station OMNI IV and the field OMNI IV, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

On the OMNI IV used as a field magnetometer or gradiometer:

a. Press the CHANGE key, then enter the required access code:

(i) Key in the number 1 to dump raw field data.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0.

(iv) Press the ENTER key.

On the OMNI IV used as a base station:

a. Press the CHANGE key, then enter the required access code:

(i) Key in the number 3 to verify the field unit data.

(ii) Key in the same second digit as in a.(ii) above.

(iii) Key in 0, 0.

(iv) Press the ENTER key.

For both OMNI IV units:

a. Power up the peripheral device.

b. Press both AUTO RECORD keys simultaneously. Data transfer will commence and data correction will occur in the base station OMNI IV.

5.3.5 Acquisition of Drift Measurements Between Tie Points. To dump the diurnal drift for the tie-point measurements in the tie-line unit, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

(i) Key in the number 4 to dump the tie-point drift.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0.

(iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key to begin the transfer of tie-point drift data. An 'R' denotes that the diurnal drift calculation is derived from a base reference point reading; a 'T' denotes that the drift correction is derived from a tie-point measurement.

5.3.6 Retrieval of Corrected Tie-Line Data. On the OMNI IV used as a tie-line field magnetometer or gradiometer;

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

(i) Key in the number 5 to dump corrected tie-line data.

(ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.

(iii) Key in 0, 0.

(iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key. Data transfer will commence and data correction will occur in the tie-line OMNI IV.

5.3.7 Programmable Datum. By first subtracting a coarse field value from each field measurement collected on a survey grid, it is considerably more accurate to plot the differences between readings. (For example, if adjacent readings of 57,500 and 57,700 gammas are recorded, then a coarse value of 57,000 is subtracted from each, the resultant plot would be values of 500 and 700.) To remove the preset datum from the field data, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the ERROR key twice (ie, DATUM is activated). If datum displayed is not as required, press the CHANGE key, then key in numerics to construct desired datum.

d. Press the ENTER key.

e. Proceed with any one of the data dump procedures in paras 5.3.1 through 5.3.4.

5.3.8 Programmable Reference. By first subtracting a coarse field value from each base station measurement collected on a time-interval basis, it is considerably more accurate to plot the drift in the magnetic field. To remove the preset reference field, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the REF.FLD key once. If the reference field datum displayed is not as required, press the CHANGE key, then key in the numerics to construct the desired reference value.

d. Press the ENTER key.

e. Proceed with the data dump from the base station or tie-line unit using the procedure in para 5.3.1 or 5.3.6.

5.3.9 Field Dumping of Data. It may be necessary to dump unwanted data during a field survey without having a printer connected so that the field data memory can be initialized (ie, entering the OP number). Proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key, then press the AUTO RECORD key. The display shows a flashing number HPNNNN.

c. Press the CHANGE key, then key in 1000. Press ENTER.

d. Press the AUTO RECORD key. No key will respond because the unit is waiting for a ready signal from the printer.

e. Press the OFF key.

5.4 Profile Plots

Profile plots can be developed using the data collected by the OMNI IV. Choose any printer with a width between 40 and 190 characters. If the printer width is 70 characters or less, only the profile plot is printed. Printers wider than 70 characters can accommodate the profile plot and the data. When the paper width is less than 70 characters, the data can be printed out in digital format first using the normal data dump procedures. Then the plot can be performed using the PROG mode. The paper plot can be performed using the PROG mode. The paper strips can be matched side by side to give the overall format. A sample profile plot showing total field and gradient is given in Figure 5-5. Proceed as follows:

a. Interconnect the line printer and turn on the power.

b. Set the MODE selector to PROG.

c. Press the READ key. The serial number of the OMNI IV will appear on the display.

d. Press the AUTO RECORD key. The display will show 1PNNNN. This denotes the first parameter which is the printer paper width to be used. If the parameter is correct, press ENTER.

e. If the parameter requires changing, press the CHANGE key, then key in the new parameter. (For example, for a 40 character printer, key in 0040; for 190 characters, 0190.) Press ENTER. The printer will verify that the parameter is entered.

f. Press the AUTO RECORD key again. The display will show 2PNNNN. This denotes the second parameter which is the total field full scale shown as X on the print-out. If the parameter is correct, press ENTER.

g. If the parameter requires changing, press the CHANGE key, then key in the new parameter. (The largest field scale is 9999 gammas.) Press ENTER. The printer will verify the new full scale.

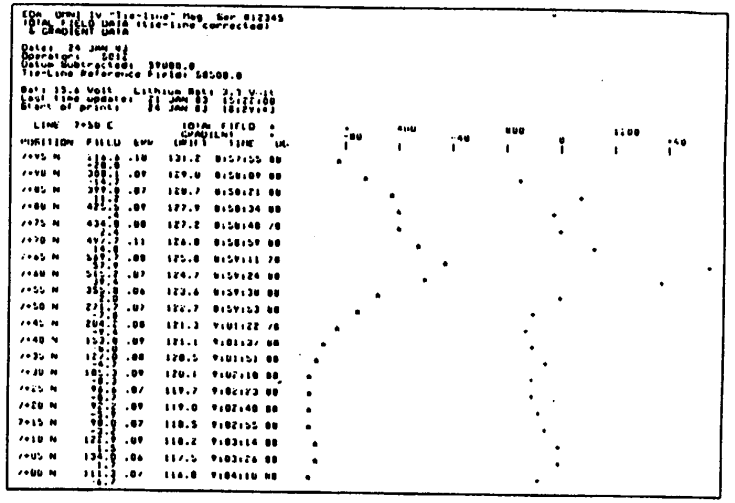


Figure 5-5 Profile Plot of Total Field and Gradient

h. Press the AUTO RECORD key again. The display will show 3PNNNN. This denotes the third parameter which represents the full scale of the gradient data being presented.

j. If the OMNI IV does not have a gradient capability, press the CHANGE key, then key in 0000. Press ENTER.

k. If the OMNI IV has gradient capability and the parameter is correct, press ENTER. If the parameter requires changing, press the CHANGE key, then enter the new parameter. (The largest field scale is 9999 gammas.) Press ENTER. The printer will verify the new full scale.

NOTE

If the gradient scale is set to 3P0000, a fine total field scale (denoted by +) will also be plotted at a 10 times finer scale than the total field scale (ie, 2PNNNN ÷ 10, full scale).

CAUTION

If any parameter has been entered incorrectly, all three entries must be repeated.

m. After all three parameters have been entered, press the AUTO RECORD key to start the profile plot of data.

NOTE

If the paper width is less than 70 characters, the data blocks can be dumped first, then the profile plot underneath it. Then the two printed outputs can be placed side by side for the overall picture.

5.5 Specifications of the ASCII Fixed Format CPU Dump

In the ASCII CPU dump, the fields are rigidly defined.

First, there are a few empty lines with: 'cr,lf,nul,nul'. All the lines with data start with a three character alphanumeric field, not belonging to the data, but defining what type of data/header it is:

'H0'	first line of header data.
'H1'	second line of header data.
'H2'	third line of header data.
'H3'	fourth line of header data.
'H4'	fifth line of header data.
'D'	magnetometer data, preceding each line of measurement data.

DATA FORMAT FOR FIELDMAG or GRADIOMETER DATA (corrected and uncorrected). (BASE-STATION (uncorrected only) DATA EXITS OUT OF THE FIRST 26 FIELDS, including 'C' for the checksum.

```
         1         2         3         4         5         6
123456789012345678901234567890123456789012345678901234567890 1

D  1107080114 561234 1123* 561234  42000 -55125 250078 561234
xxxMMDDHHMMSSFFFFFFFRRRRRCBBBBBBBLLLLLLLPPPPPPP EEEEDSGGGGGG
```


Format	Column	Description
xxx	1-3	3 character alphanumeric preamble for measurement data (always 'D').
MMDDHHMMSS	4-13	10 character numeric field for date and time.
FFFFFFF	14-20	7 character numeric field for raw upper sensor data, with implied decimal point before the last digit, ie, 56123.4 gamma.
RRRRR	21-25	5 character numeric field for the reading number (or record pointer 'RPxxxx' on display).
C	26	1 alphanumeric character checksum error (blank if all OK).
BBBBBBB	27-33	7 character numeric field for corresponding base station reading to be used for diurnal correction.
LLLLLLL	34-40	7 character numeric field for line number with implied decimal point before the last digit, and preceded by a '–' sign if either S or W '–121250' = 121S+25 or 12125 S.
PPPPPPP	41-47	7 character signed numeric field for station or position number in the same way as the LINE number.
EEEE	49-52	4 character numeric field for error with implied decimal point after the second digit, ie, xx.xx.
D	53	1 character numeric field for the decay rate (1 to 8).
S	54	1 character numeric field for the sensor strength (1 to 8).
GGGGGGG	55-61	7 character numeric field for the raw field strength of the bottom sensor with implied decimal point before the last digit. This value minus the upper sensor strength gives the gradient.

Examples:

```
D  1107080114 561234 1123* 561234  42000 -55125 250078 561234
D  8131401361095000123 00 1011234 -42000  35250     7881084000
```

NOTE

A blank should be regarded as a zero. The whole string has leading zero blanking.

Header Formats:

'H0 IISSSSSSXXYYRRRRRROOOOHHhhMMMMVVVV'

'H0'	3 character alphanumeric field for first header line.
II	2 character numeric field for instrument code.
SSSSSS	6 character numeric field for serial number.
XX	2 character numeric field (00 if uncorrected, 99 if corrected).
YY	2 character numeric field (internal record length).
RRRRRR	6 character numeric field for the number of records or readings.
0000	4 character numeric field for the operator number.
HH	2 character numeric field for HP-CODE used in field mag.
hh	2 character numeric field for HP-CODE used in base station (correcting mag).
MMMM	4 character numeric field for main battery voltage.
VVVV	4 character numeric field for lithium battery voltage.

'H1 PPPPPPPLLLLLLLRRRRRRRDDDDDDD'

'H1'	3 character alphanumeric preamble for 2nd header line.
PPPPPPP	7 character numeric field for position or station of base mag (if used).
LLLLLLL	7 character numeric field for line of base station mag (if used).
RRRRRRR	7 character numeric field for the base-reference field.
DDDDDDD	7 character numeric field for the datum field of grid.

'H2 MMDDHHMMSSmmddhhmmssMMDDHHMMSS'

'H2'	3 character alphanumeric preamble for 2nd header line.
MMDDHHMMSS	10 character numeric field for time of field mag time update.
mmddhhmmss	10 character numeric field for time of first reading of survey.
MMDDHHMMSS	10 character numeric field for time of data dump. All times are in month/day, hour:minute:seconds format.

H3 — 3 character alphanumeric preamble for 3rd header line.

All data fields are the same as for 'H2', except that the times apply to the base station mag use in the correction mode (if applicable).

H4 — 3 character alphanumeric preamble for 4th header line.

No data fields presently specified (for future expansion only).

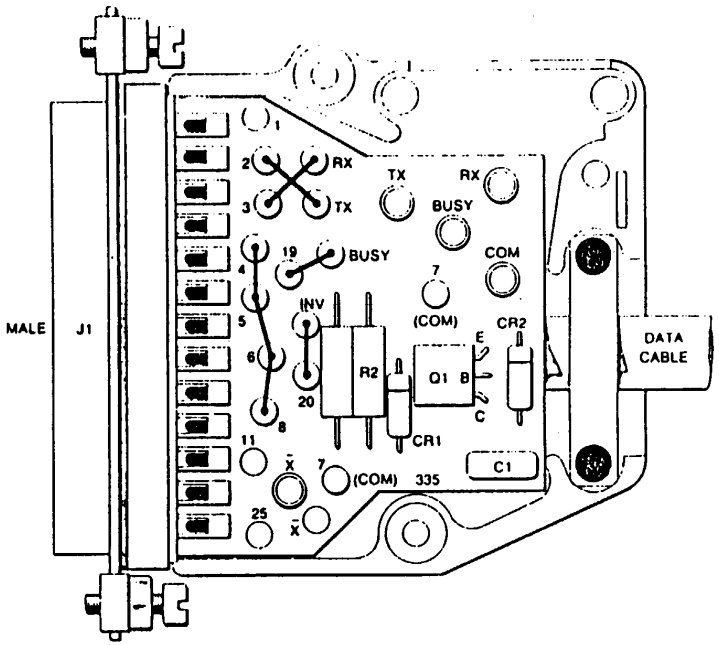


Figure 5-6 Interconnect Cable (Data Correction to TRS80/100), Male, (1-020-00335)

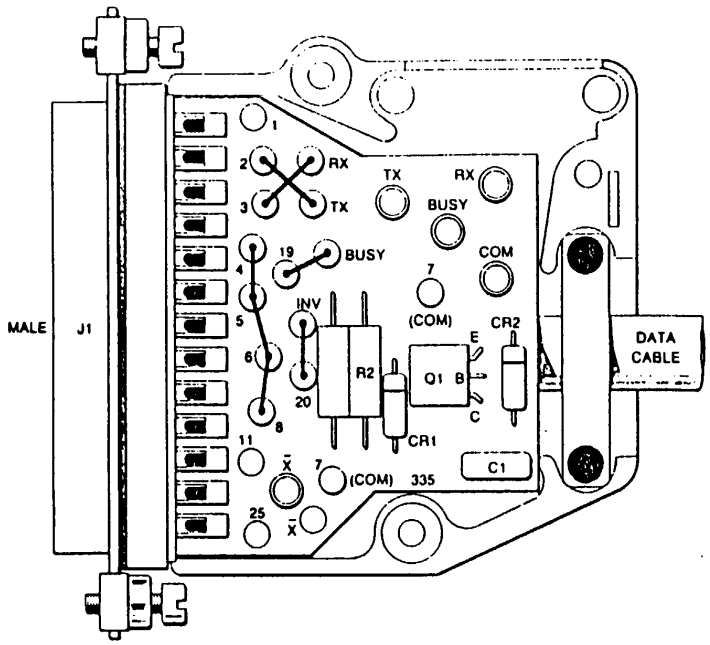


Figure 5-7 Interconnect Cable (Data Correction to Printer), Male, (1-020-00335) DCX-443

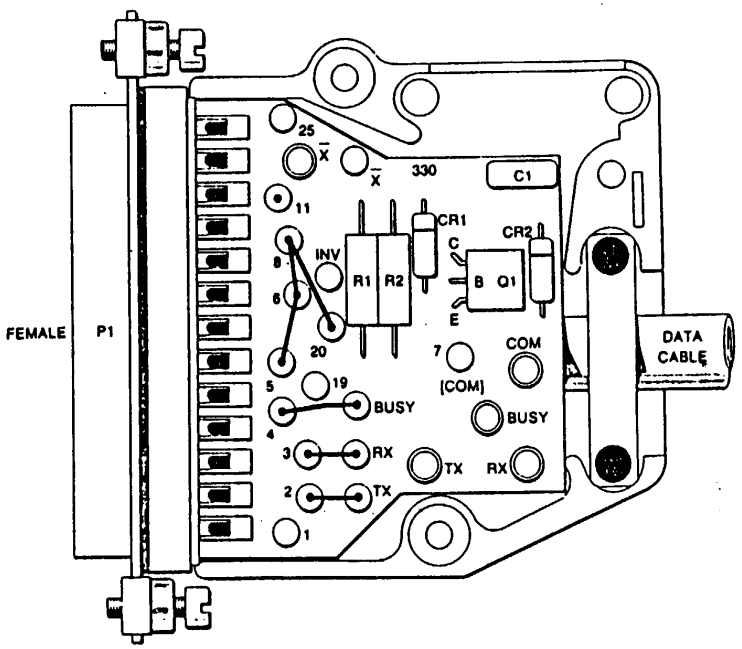


Figure 5-8 Interconnect Cable (Data Correction to Heathkit H-14 Printer), Female, (1-020-00330)

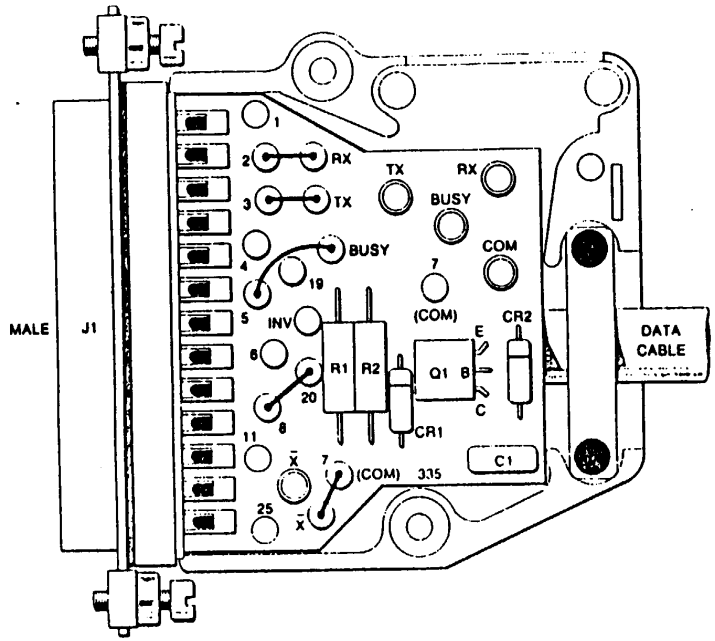


Figure 5-9 Interconnect Cable (Data Correction to HP-85 Option 0), Male, (1-020-00335) DCX-446

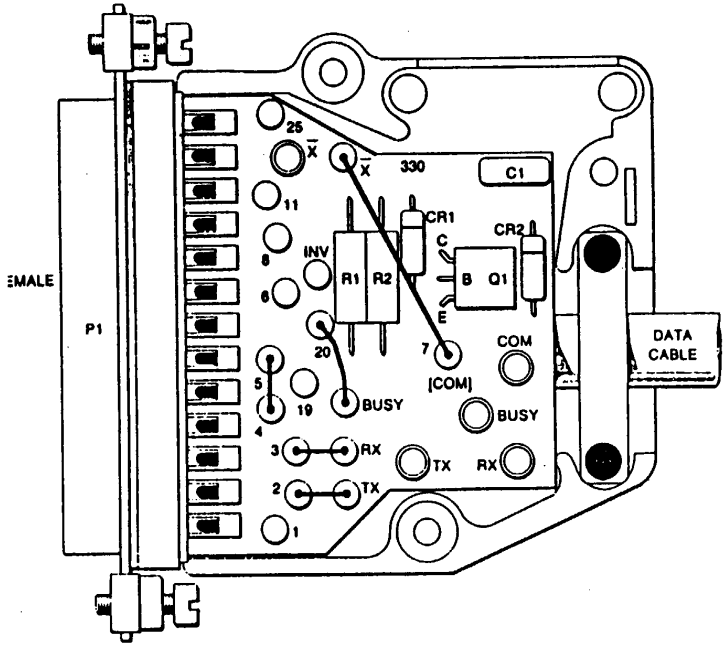


Figure 5-10 Interconnect Cable (Data Correction to HP-85 Option 1), Female, (1-020-00330) DCX-446

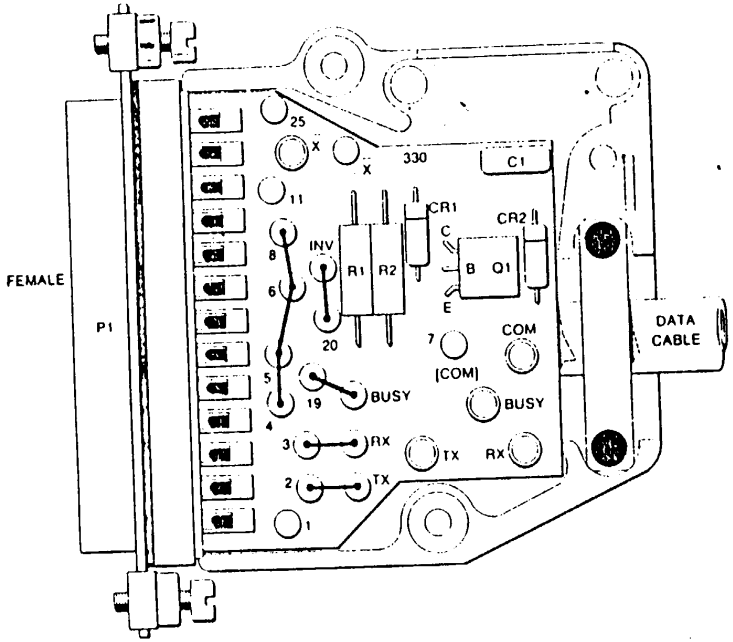


Figure 5-11 Interconnect Cable (Data Correction to IBM PC), Female, (1-020-00330) DCX-447

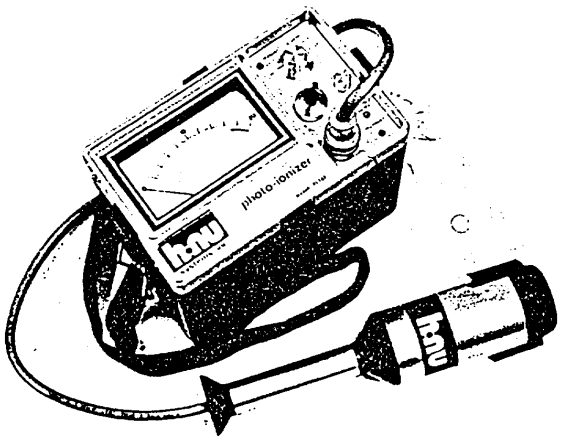
h·nu systems inc.

INSTRUCTION MANUAL

FOR

MODEL PI 101

PHOTOIONIZATION ANALYZER



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SECTION 2

OPERATION

2.1 Unpacking

Unpack the instrument carefully and remove the housing, the probe and any spare parts from the shipping carton. Place the instrument on a table or bench with the label upright. Remove the top section of the instrument by opening the two fasteners on the cover (see figure 1). The inner panel of the top section can be removed by pulling up on the fasteners. The top section of the instrument contains the battery charger and a waist strap. The waist strap clips on to the strap brackets of the instrument when needed.

Before attaching the probe, check the function switch on the control panel to make sure it is in the off position. The 12 pin interface connector for the probe is located just below the span adjustment on the face of the instrument (see figure 2). Carefully match the Alignment Key in the probe connector to the 12 pin connector on the control panel, and then twist the probe connector until a distinct snap and lock is felt.

Attached to the instrument is a warranty card which should be filled out completely and returned to HNU Systems.

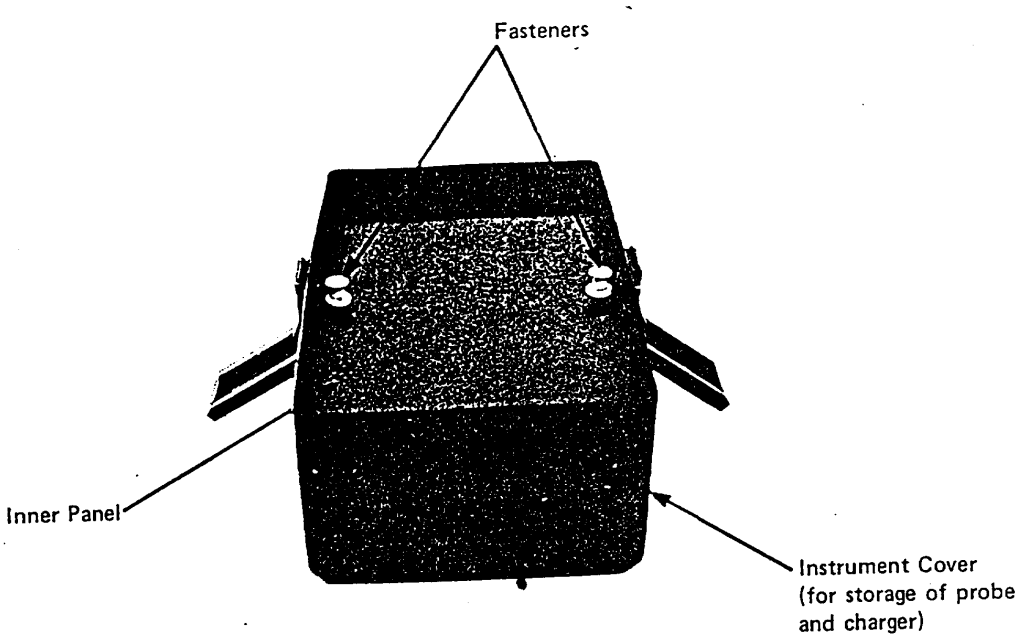
2.2 Operation

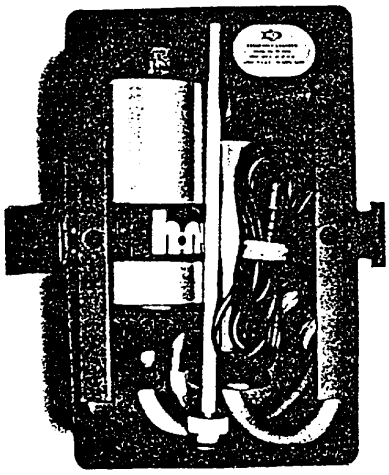
Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scaleplate. If the needle is in the lower portion of the battery arc, the instrument should be recharged prior to making any measurements. If red LED comes on, the battery should be recharged.

Next, turn the function switch to the on position. In this position the UV light source should be on. Look into the end of the probe to see the purple glow of the lamp.

A brief description of the instrument controls and functions is shown in Figure 2.

Figure 1 (Continued)





Top View

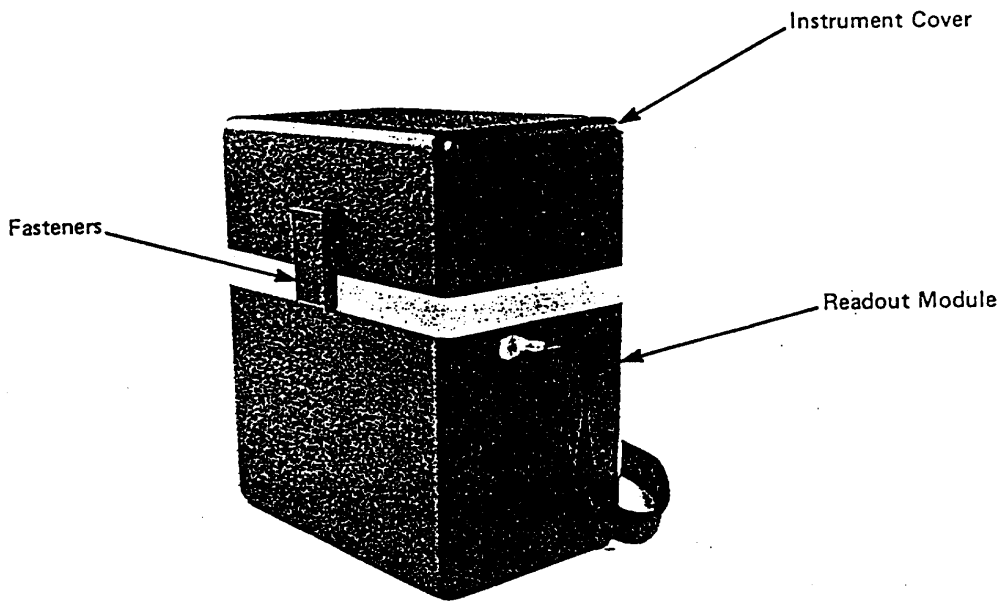


Figure 1. Unpacking the Photoionizer.

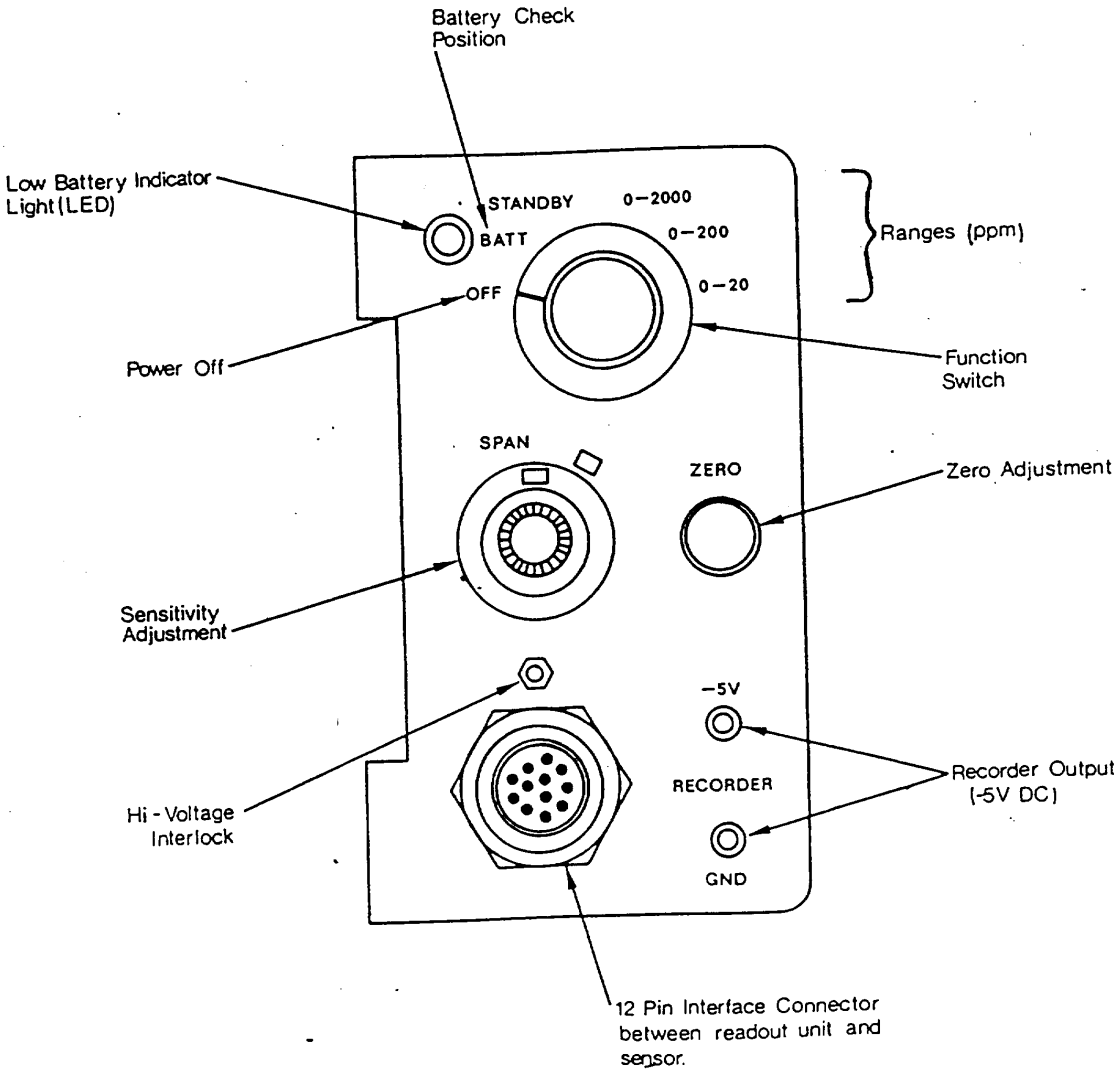
TABLE II

BRIEF DESCRIPTION OF INSTRUMENT CONTROLS AND FUNCTIONS*

Control	Function
Six Position Switch	OFF - Shuts off all power and removes DC voltages. ON - In any other function position or measuring mode, the electronics are on. BATTERY CHECK - Indicates the condition of the battery. If needle position is in lower portion of green battery arc, the instrument should be recharged. STANDBY - UV lamp is off but electronics are on. This position will conserve power and extend the useful operating time between recharges of the battery. This position is also utilized to adjust the electronic zero. RANGES - 0-20, 0-200, 0-2000 direct reading ranges available at minimum gain for benzene. More sensitivity is available by adjusting the span potentiometer.
Zero Potentiometer	A ten turn potentiometer is employed to adjust the zero electronically when the instrument is placed in the standby position with the probe attached. This eliminates the need for a hydrocarbon free gas.
Span Potentiometer	A ten turn counting potentiometer is utilized for upscale setting of the meter on calibration gas. Counter-clockwise rotation increases the sensitivity (~10 times). This pot can increase the sensitivity to make the instrument direct reading for nearly any gas which the instrument responds to.

*For position of layout controls see Figure 2.

Figure 2 Control Panel Functions



To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer 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, and 0-2000 ppm scales become 0-2, 0-20, and 0-200 ppm full scale, respectively. This 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, For a listing of approximate gain setting values see Table IV.

A small DC operated fan is used to pull air through the photoionization sensor at a flow rate of three to seven hundred centimeters per minute (ca. 0.5 lpm). The fan provides nearly instantaneous response times (Figure 3) while consuming little power. The characteristics of a fan are such that it cannot tolerate a significant pressure drop without affecting the flow rate and therefore either the instrument reading or response time. Since photoionization is essentially a nondestructive technique, changes in flow rate do not affect the signal but if a large pressure drop is imposed at the inlet of the probe, the sample may not reach the sensor.

TABLE III

VERIFICATION OF ELECTRONIC ZERO FOR PHOTOIONIZATION ANALYZER*

Sample	Instrument Reading (ppm)	% of F.S.
Room Air	0.7	35
Room Air Passed Through 6" x 3/4" OD Charcoal Scrubber	0.1	5
Zero Air	0.25	12.5
Zero Air Passed Through 6" x 3/4" OD Charcoal Scrubber	0.04	2

*Maximum Gain = 2 ppm full scale.

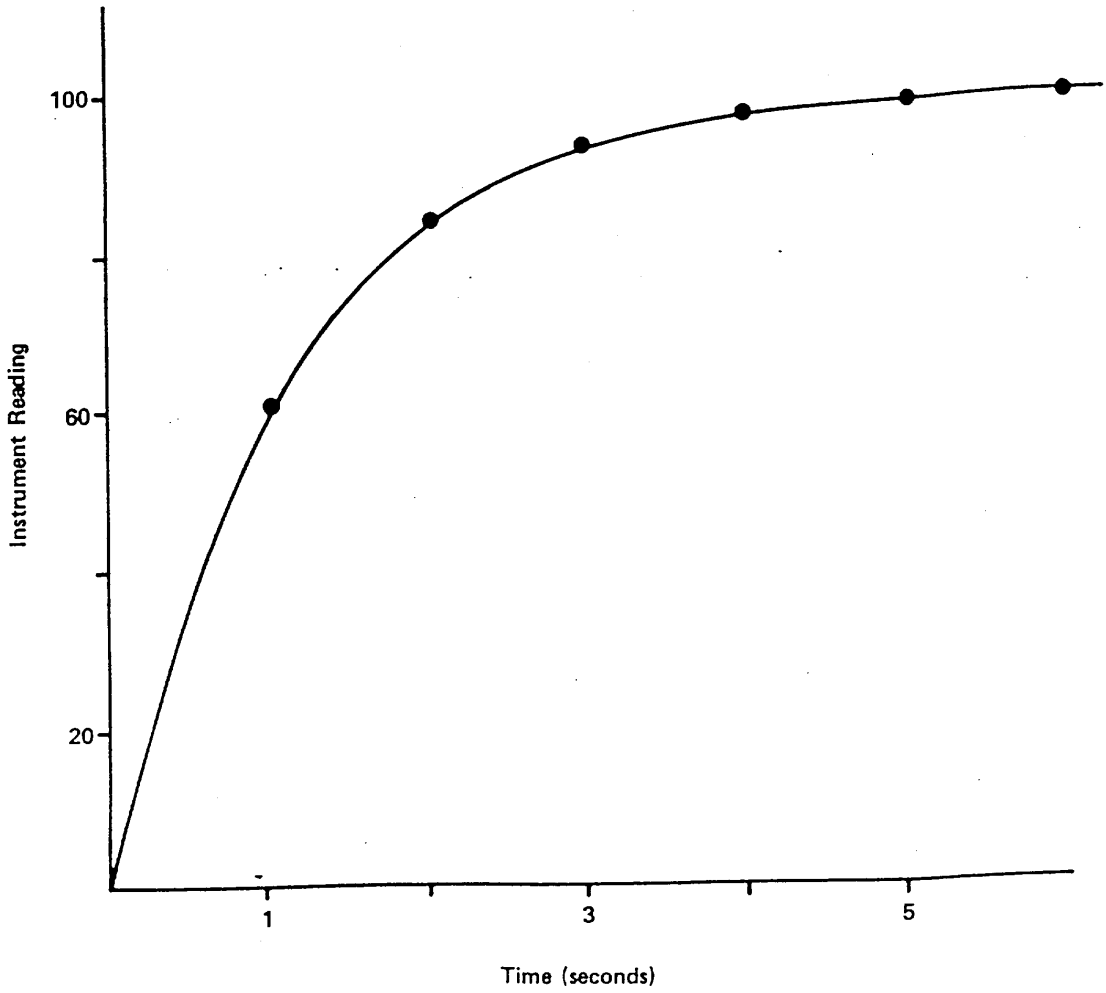
TABLE IV

RELATIVE PHOTOIONIZATION SENSITIVITIES*
FOR VARIOUS GASES

Chemical Grouping	Relative Sensitivity	Examples
Aromatic	10.0	Benzene, Toluene, Styrene
Aliphatic Amine	10.0	Diethylamine
Chlorinated Unsaturated	5-9	Vinyl Chloride, Vinylidene Chloride, Trichloroethylene
Carbonyl	5-7	MEK, MIBK, Acetone, Cyclohexene
Unsaturated	3-5	Acrolein, Propylene, Cyclohexene, Allyl Alcohol
Sulfide	3-5	Hydrogen Sulfide, Methyl Mercaptan
Paraffin (C_5-C_7)	1-3	Pentane, Hexane, Heptane
Ammonia	0.3	—
Paraffin (C_1-C_4)	0	Methane, Ethane . . .

*Sensitivities in ppm (v/v).

Figure 3. Time Response for the Photoionization Analyzer.



The instrument was designed to measure trace gases over a concentration range from less than 1 ppm to 2000 ppm. Higher levels of various gases (to percentage range) can be measured but the recommended procedure is to dilute the sample with clean air to a concentration of less than 500 ppm. This is generally within the linear range of the instrument and if the measured concentration is multiplied by the dilution ratio the correct concentration in the stream can be determined. A typical calibration curve is shown in Figure 4. Note that the calibration curve for benzene (the photoionization standard) is linear (over more than three decades) up to about 600 ppm (v/v).

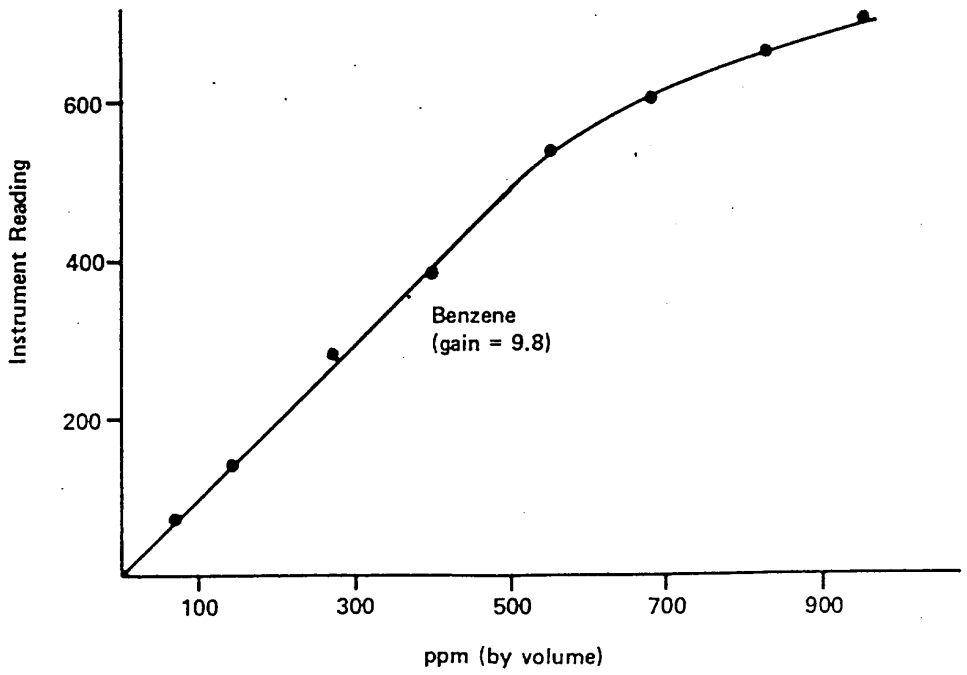
If the probe is held close to AC power lines or power transformers, an error may be observed. For measurements made in close proximity to such items, their effect on measurements can be determined by the following procedure. Zero the instrument in an electrically quiet area, in the standby position, then move the instrument to the questionable area involved. If AC pickup is going to be a problem, the meter (in the standby position) will indicate the magnitude of the error.

The instrument is equipped with an automatic solid state battery protection circuit. When the battery voltage drops below ∿ 11 volts, this circuit will automatically turn off the power to the instrument. This prevents deep discharging of the battery and considerably extends the battery life. If the instrument is unintentionally left on overnight, the battery will be unharmed because of the battery protection circuit. If the instrument battery check reads low and the lamp doesn't fire, plug the charger into the instrument. The power to the analyzer should then be returned.

To charge the battery, place the mini phone plug into the jack on left side of the bezel prior to plugging charger into 120 VAC. When disconnecting charger, remove from 120 VAC before removing mini phone plug. The battery is completely recharged overnight (ca. 14 hours). To ensure that the charger is functioning, turn the function switch to the battery check position, place phone plug into jack and plug charger into AC outlet. The meter should go upscale if charger is working and is correctly inserted into the jack.

The instrument can be operated during the recharge cycle. This will lengthen the time required to completely recharge the instrument battery.

Figure 4. Typical Calibration Curve for Photoionization Analyzer.



SECTION 3

CALIBRATION

Static or dynamic gas generation systems can be utilized for calibration of the instrument. A number of such systems for generating test atmospheres for various gases have been described by G. O. Nelson in "Controlled Test Atmospheres," Ann Arbor Science Publishers, Ann Arbor, Michigan (1971).

The most convenient packages for calibration are the non-toxic analyzed gas mixtures available from HNU Systems in pressurized containers (Catalogue #101-350).

A rapid procedure for calibration involves bringing the probe and readout in close proximity to the calibration gas, cracking the valve on the tank and checking the instrument reading. This provides a useful spot check for the instrument.

The recommended and most accurate procedure for calibration of the instrument from a pressurized container is to connect one side of a "T" to the pressurized container of calibration gas, another side of the "T" to a rotameter and the third side of the "T" directly to the 8" extension to the photoionization probe (see Figure 5). Crack the valve of the pressurized container until a slight flow is indicated on the rotameter. The instrument draws in the volume of sample required for detection, and the flow in the rotameter indicates an excess of sample. Now adjust the span pot so that the instrument is reading the exact value of the calibration gas. (If the instrument span setting is changed, the instrument should be turned back to the standby position and the electronic zero should be readjusted, if necessary.)

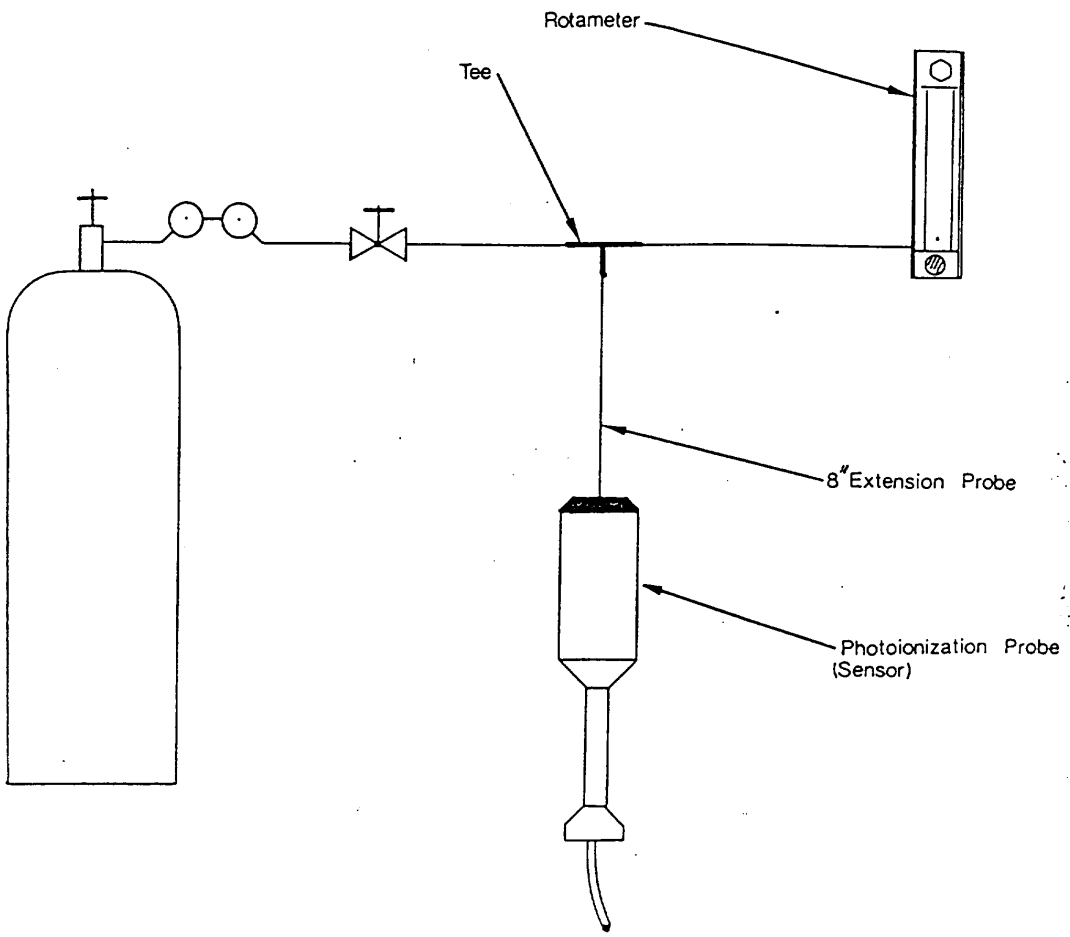


Figure 5. Recommended Calibration Procedure for Photoionization Analyzer

The calibration gas* should be prepared in the same matrix (air, nitrogen, hydrogen, etc.) in which it is to be measured, otherwise an inaccurate reading may be obtained. The increased response which is seen in oxygen free gases can be attributed to a reduction in the quenching of ions by oxygen (actually O_2^-) and is typical of any ionization detector. The quenching effect of oxygen is constant from about ten percent O_2 to very high levels.

If a gas standard prepared in nitrogen is to be used for measurements in air, fill a 0.5 or 1 liter bag with the standard then add 50 or 100 cc of pure oxygen to bring the level to 10-12%.

Any error between this value and 20% oxygen is quite small.

If the sample to be measured is in nitrogen, standards should be prepared in nitrogen. This will result in an increase in sensitivity of approximately 4.

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* Calibration with toxic gases should be performed in a hood since the 101 is a non-destructive analyzer.

Appendix A-A5

Instruction Manual for the Mount Sopris Model 1000-C Borehole Geophysical Logging Device

INSTRUCTION MANUAL

MODEL 1000-C LOGGER

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.

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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.

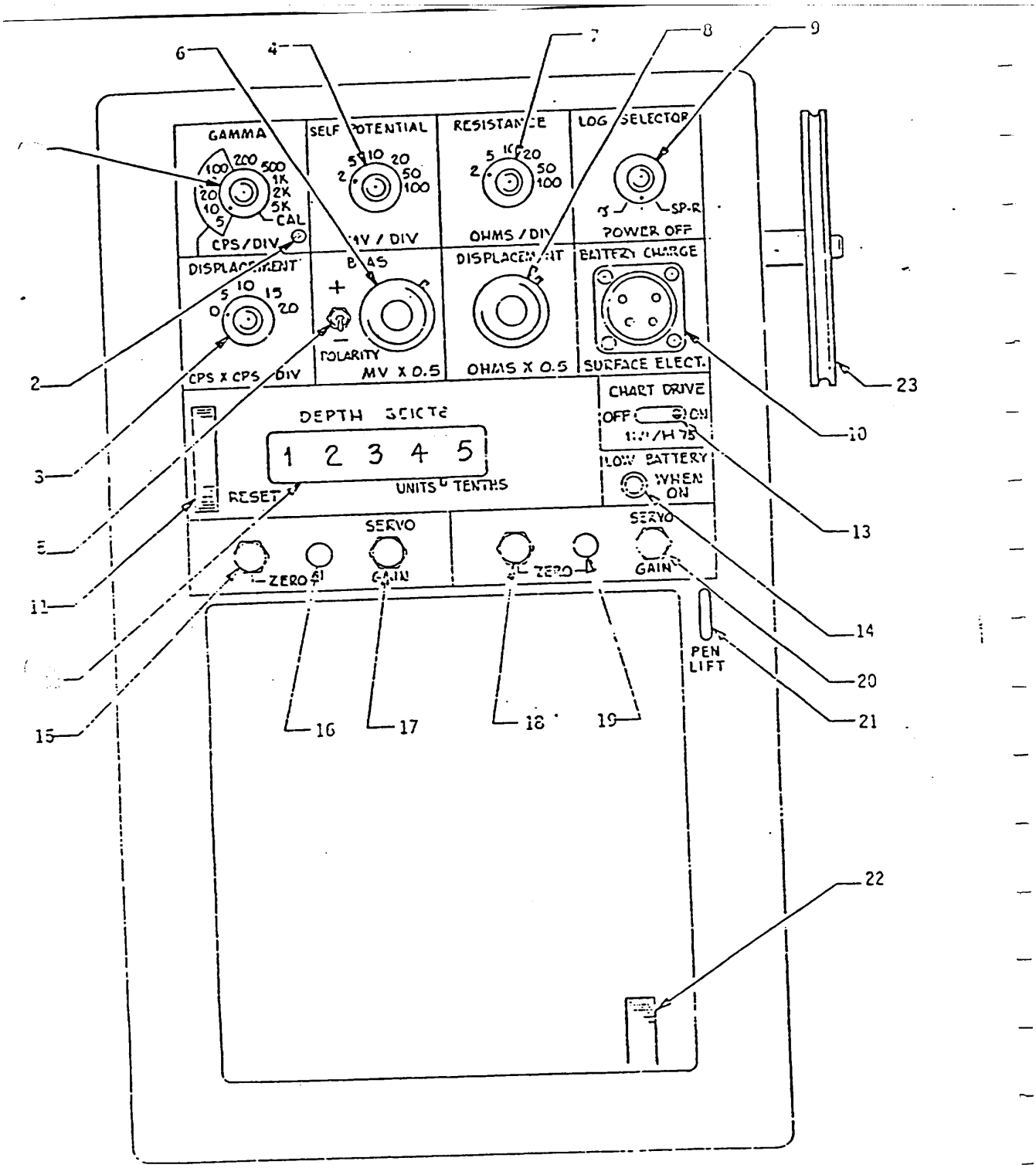


Fig. 1 Recorder Front Panel

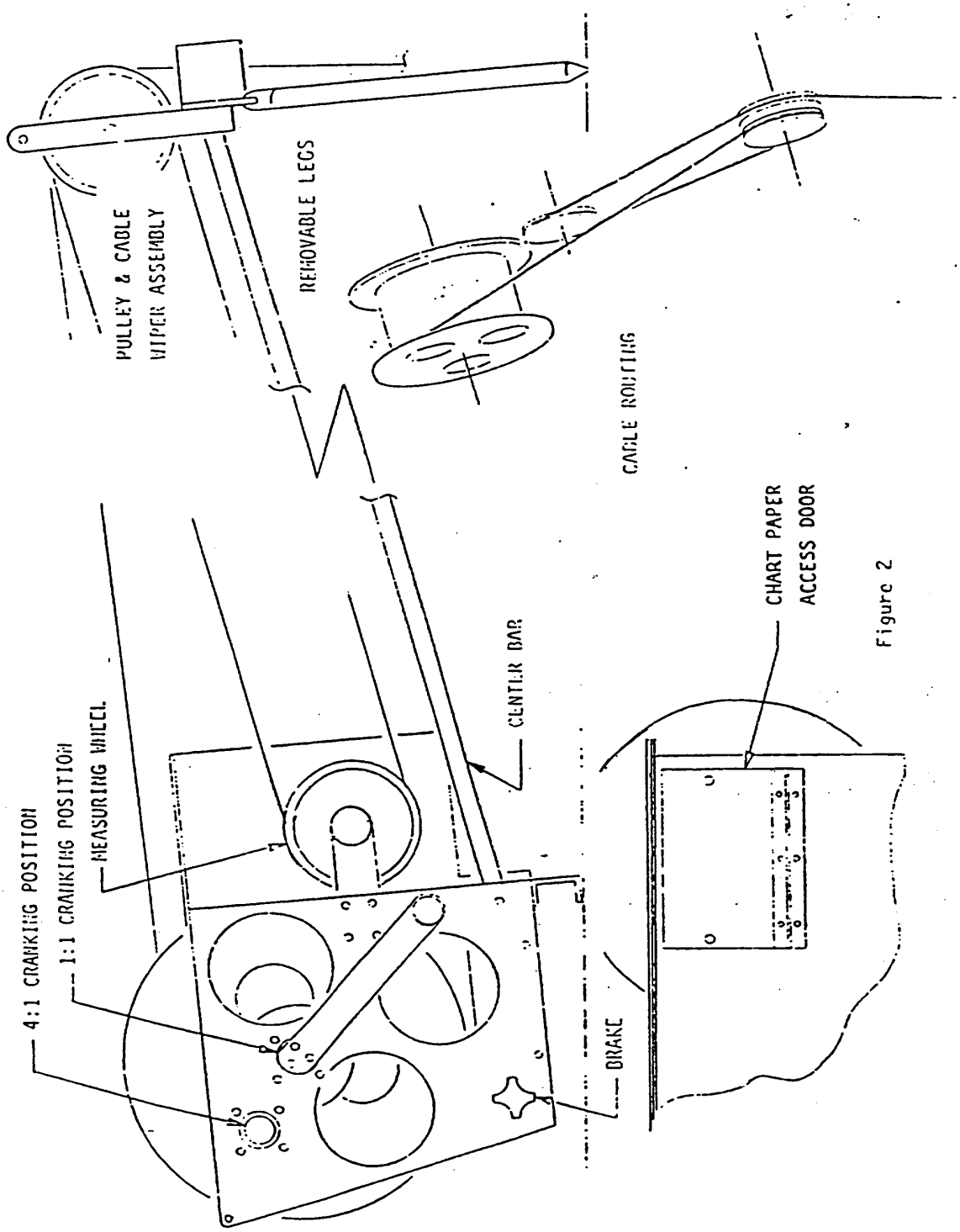

PULLEY & CABLE
WIPER ASSEMBLY
REMOVABLE LEGS
CABLE ROUTING
CHART PAPER
ACCESS DOOR
CENTER BAR
4:1 CRANKING POSITION
1:1 CRANKING POSITION
MEASURING WHEEL
BRAKE

Figure 2

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.

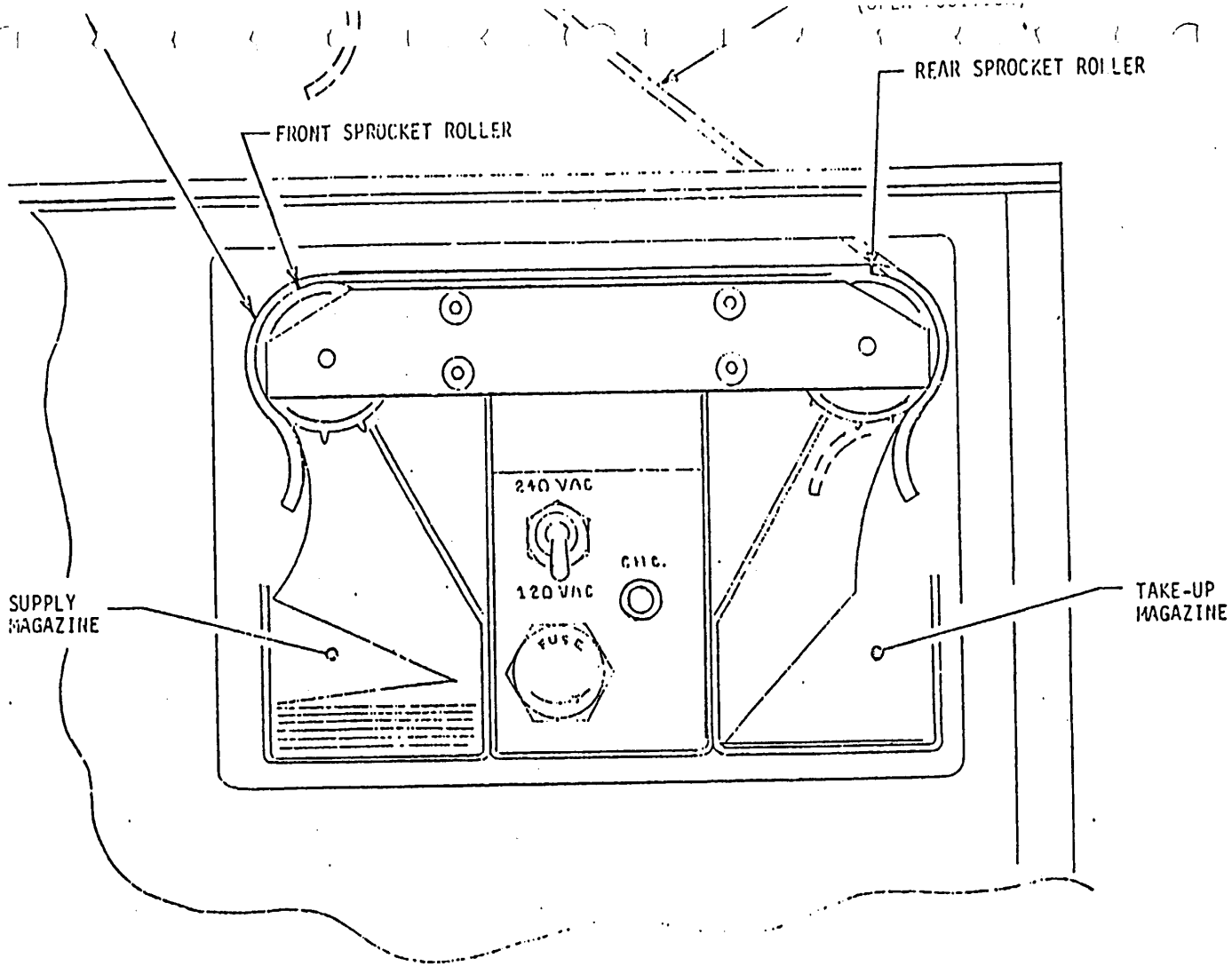


Fig. 3 CHART TRANSPORT ASSEMBLY (A)

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.

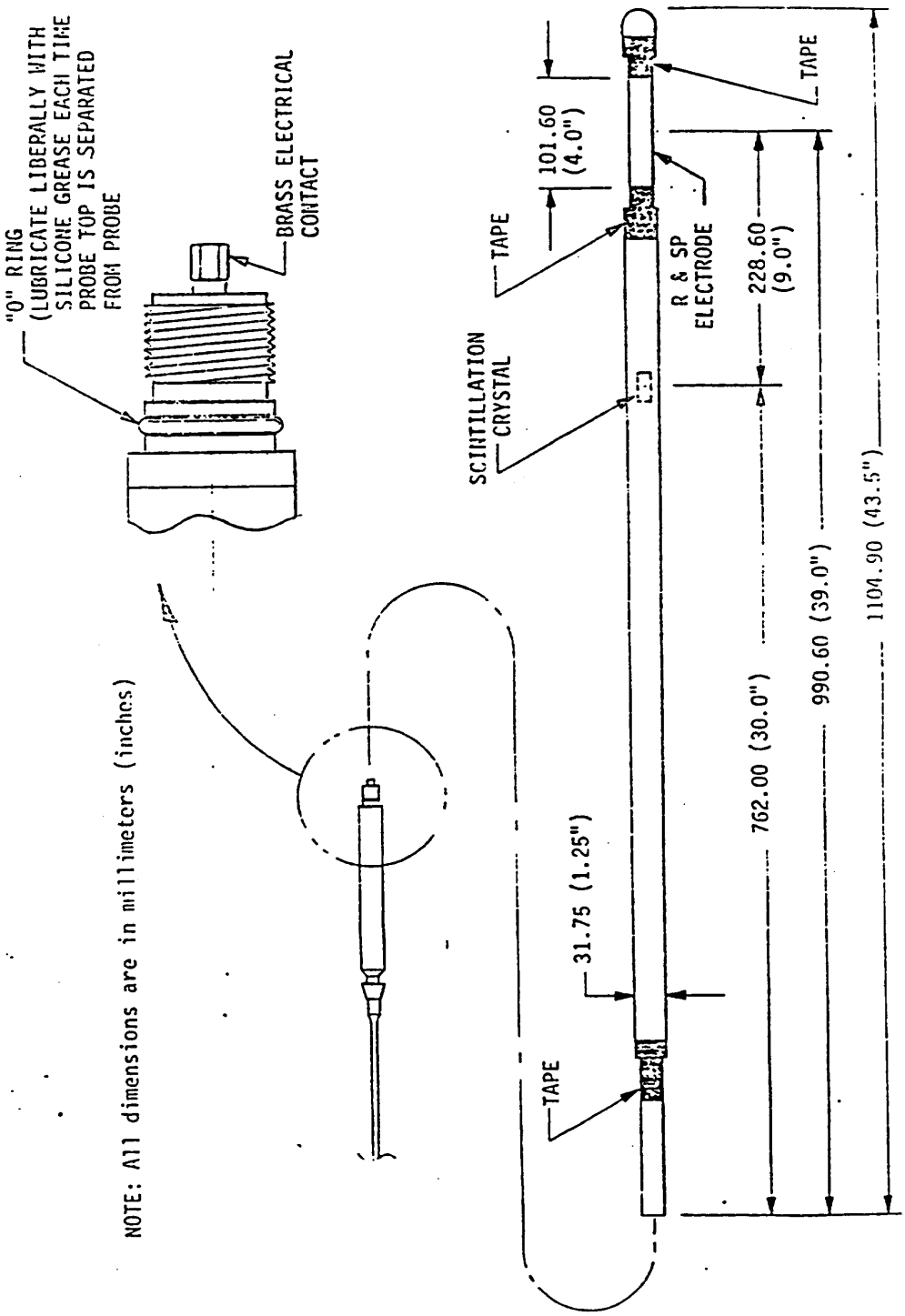
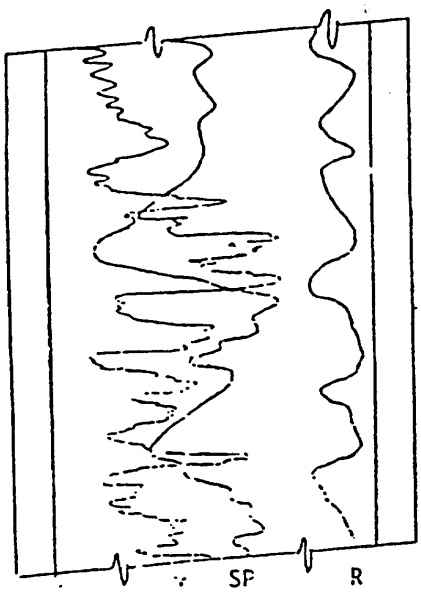
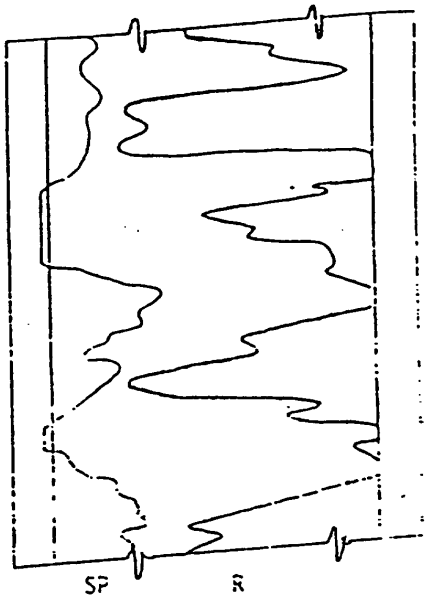


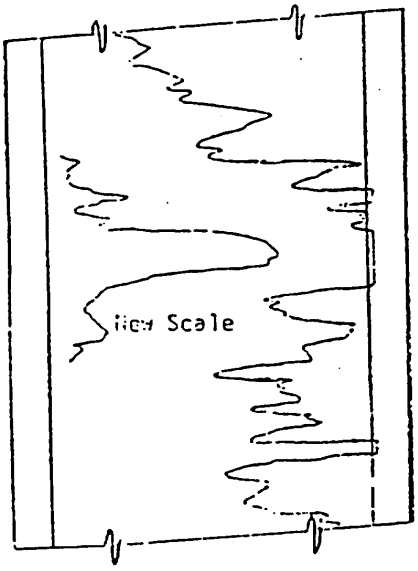
Fig. 4 PROBE DIMENSIONS



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.

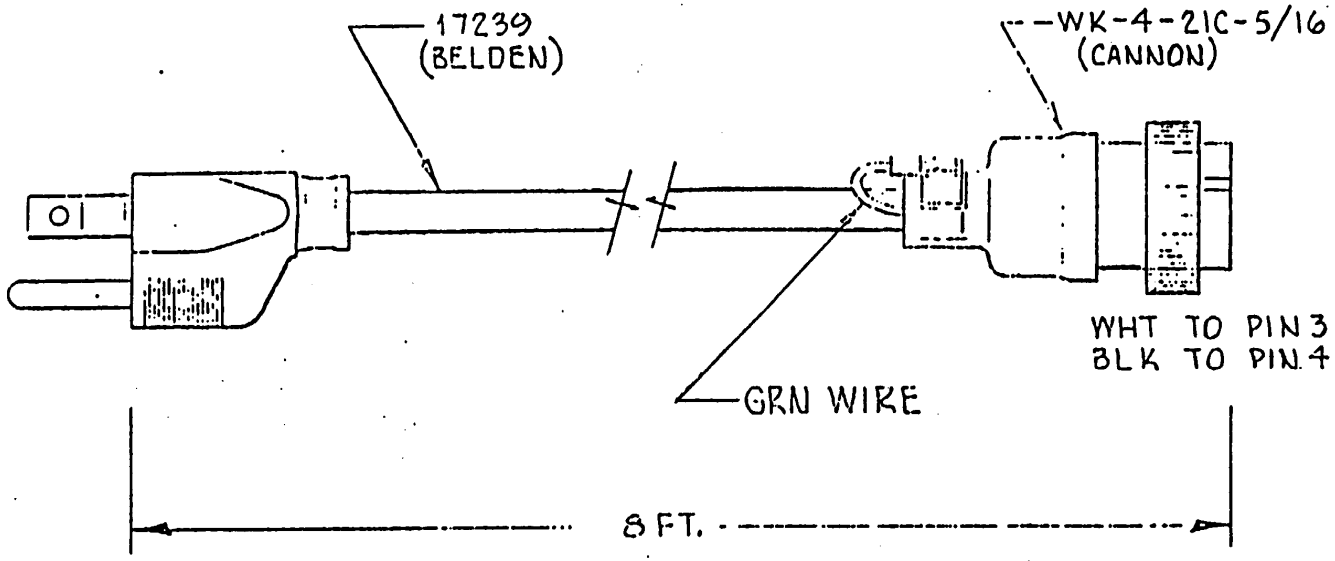
2-5.9 Handle probe with care. Avoid rapid changes in temperature and sharp blows, especially in a lateral direction.

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 FACTORS TO USE WITH

MOUNT SOPRIS MODEL S11, S11E, AND G-375 PROBES

Hole Diameter in Inches	Correction Factor
2.25	1.035
4.5	1.076
6.5	1.115
8.5	1.115

CASING FACTORS TO USE WITH

MOUNT SOPRIS MODEL S11, S11E, AND G-375 PROBES

Casing Wall Thickness in Inches	Correction Factor
1/16"	1.24
1/8"	1.33
3/16"	1.43
1/4"	1.62
3/8"	1.87

Gamma Time Constant (T.C.) Switch

This unit has been fitted with a Gamma time constant response switch. The switch is mounted on the dividing line between the Gamma and SP Range Switches. The switch has two positions, 2.4 and 0.3, corresponding to a full scale response time of 2.4 seconds and 0.3 seconds resulting from a full scale input step.

The switch should normally be set to the 2.3 position for gamma probes and to the faster 0.3 range for such measurements as temperature, caliper, flowmeter and fluid resistivity, etc. If more filtering is desired this last group of probes could be run with the 2.3 position. For the most accurate results the gamma calibrate potentiometer should be adjusted each time the T.C. switch position is changed. The effect of the T.C. switch positions can be seen by operating the 1000C in the Gamma position without a probe attached and switching between the 5K range and the Cal position for each T.C. position.

3 Theory of Operation

Figure 2 is a block diagram which shows the main elements of the HLP-2375 combination probe. Refer to the appropriate schematics located in Appendix 1 as necessary for the discussion of the operation checkout and trouble shooting of this probe.

As Figure 2 indicates, the active circuits of the HLP-2375 probe serve one function (i.e a Gamma Ray Scintillometer).

The other functions of the probe, namely SP and resistance (R) are passive, consisting downhole only of the A electrode. Circuitry for these measurements is located in the HLM-2680 module which has been designed to complement the HLP-2375 probe.

3.1 Gamma Ray Scintillometer

The gamma ray scintillation circuit may be subdivided into two sections consisting of (1) the photomultiplier dynode power supply circuits and (2) the pulse detection shaping and line driving circuitry.

Photomultiplier Dynode Power Supply (See schematics for boards A2 and A3) - Unregulated power of at least 20 Vdc at the cablehead is required to operate the HLP-2375 probe. The photomultiplier dynode voltages are derived by stepping the basic 20 Vdc power supply up to a square wave of 120v (peak amplitude) through the multivibrator formed by Q1 and Q2 and transformer T1. The exact value of the AC voltage is deter-

the descriminator (adjustable through R6) the comparator will trigger causing transistor Q4 to saturate. The negative going pulse at the collector of Q4 triggers both one shots (U2). One of the one shots is used to drive the complementary emitter follower which drives a positive pulse of approximately 1 microsecond and 6.8 volts onto the cable. The other one shot is set at 5 microseconds and is used to electronically inhibit the first one shot from retriggering for that period of time. This introduces a known probe dead-time which is electronically compensated for in all Mt. Sopris ratemeters.

6 Service and Trouble Shooting

The HLP-2375 has one main active function and it should be a simple matter to isolate problems to either the natural gamma function, the uphole electronics, or cable. Before trouble shooting the probe, it is always wise to verify that the proper probe power (20 Vdc) is available between armor (common) and the center conductor (20 Vdc). The steps listed below outline a procedure for trouble shooting the HLP-2375 probe:

I. Preliminary Checks

A. Check cable for shorts or leakage, and for opens (cable resistance).

B. With probe connected and ratemeter module on, check between lines 1 and 2. There should be a DC voltage of 20 to 30 volts upon which some positive gamma pulses are riding.

1. If there is no DC voltage, then the problem is in the ratemeter.

2. If the DC voltage is present, continue.

C. Is the problem in the SP or resistance logs?

1. Yes - Check to see that when electrode A and the mud pit are connected through a power resistor, that a constant current sinewave is passing through the resistor.

   a. If no current is passing in circuit, check for continuity between line 2 (center) and the 4 in. (A) lead electrode on the sonde. Also check for continuity between the mud pit electrode and pin K of the connector in the back of the module bin. If there is continuity but no current, the problem lies in the HLM-2680 module current generator.

   b. If current signal appears to be correct but there is no resistance output, the problem still lies with the RESISTANCE circuit.

2. No - If there is a gamma problem and there is 20 to 30 Vdc between lines 1 and 2, then problem is downhole. Go to II.

   (Note: Downhole power, lines 1 and 2, should be 30 VDC with probe disconnected and ratemeter on.)

II. <u>Probe Checkout - Uphole OK</u>

A. Remove top sub on tool and carefully slide out the electronics section. After the electronics section is outside the housing, turn on the ratemeter.

1. Verify that there is at least 20 Vdc between lines 1 and 2 (red and black wires) at the cablehead. If the voltage is less than this, there is either a bad downhole power supply (presumably, this possibility has been eliminated by now), or there's something loading the power supply.

2. If the power supply is being loaded, check the gamma board.

B. Gamma Board

1. If the problem lies with the gamma circuit and the H.V. is OK, remove all the IC's on the board. Check to see that the 17 Vdc supply is OK. If not, check for a bad regulator transistor Q7, or a diode D7. Also check Q5 and Q6 of the line driver for shorts or bad transistors.

2. Check to see that there are random pulses at point A on the board. (It helps to have a small gamma source, but there should be enough background count to perform this check.)

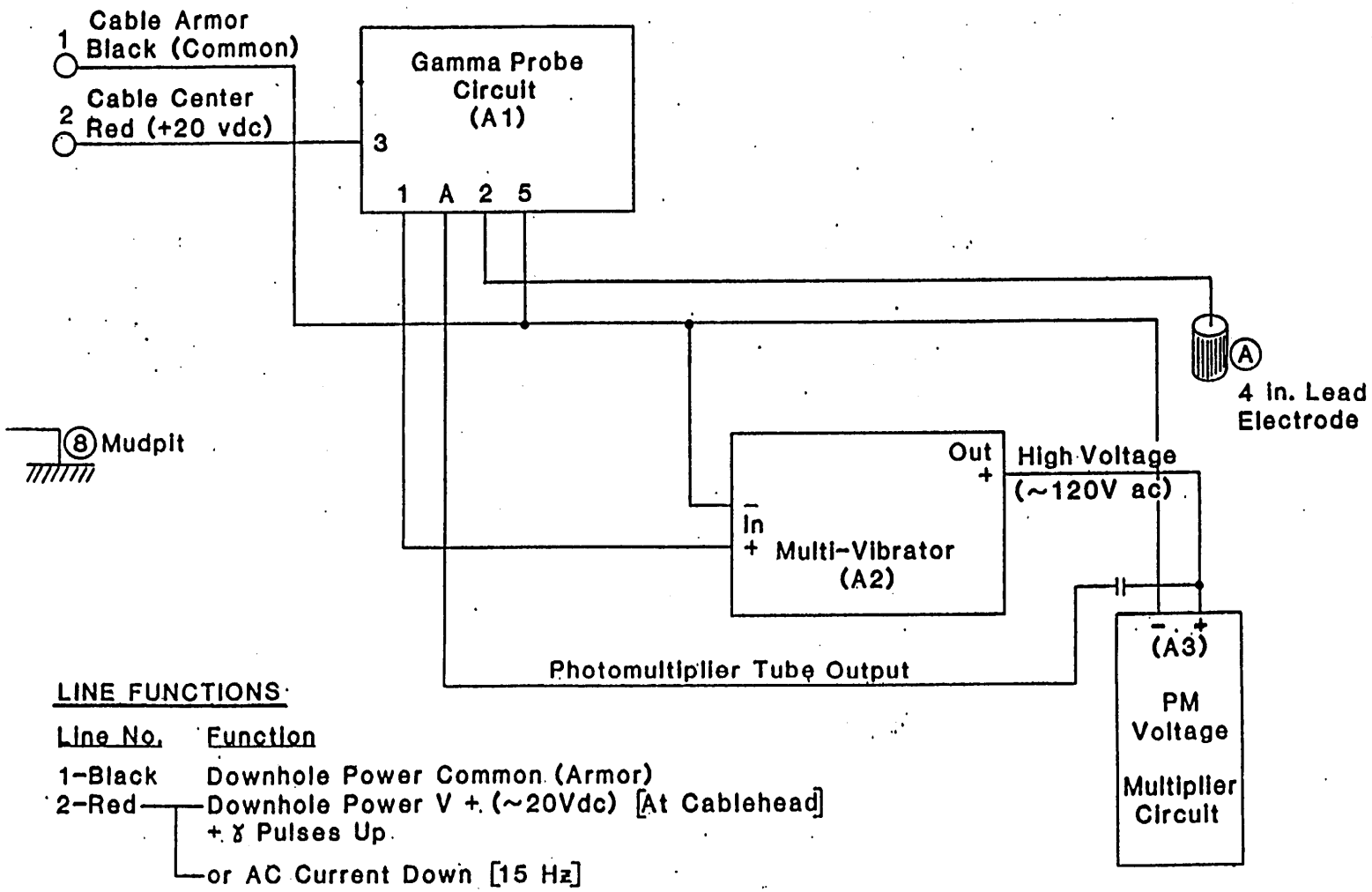
3. Follow the signal through Q1 and Q2, replacing these transistors if necessary.

4. Replace each IC in turn. Note the 17 Vdc power and check to see that the pulses are properly amplified.

5. Check for proper pulse widths on the one shots.

C. Photomultiplier Power Supply Board

Check the output of the high voltage board. Depending on the zener diodes selected, this should be a 500 Hz square wave with an amplitude of approximately 120v. This voltage should be rectified and multiplied at each successive dynode stage, with the lowest voltage being at dynode D10 and the highest (approximately 1400 Vdc) at the photocathode. If no output voltage is present across the secondary of T1, there is probably a faulty transistor Q1 or Q2 in the astable multivibrator circuit used to step up the basic 20 Vdc supply voltage.



LINE FUNCTIONS

Line No.	Function
1-Black	Downhole Power Common (Armor)
2-Red	Downhole Power V +. (~20Vdc) [At Cablehead]
	+ γ Pulses Up
	or AC Current Down [15 Hz]

MUDPIT CURRENT RETURN

FIGURE 2: FUNCTIONAL BLOCK DIAGRAM FOR THE HLP-2375 COMBINATION PROBE

Appendix A-A6

Calibration and Maintenance of the Flame-Ionization Detector (FID)

INSTRUMENT OPERATION PROCEDURE

FOXBORO Model 128 Flame-Ionization Detector
For the Detection of Organic Vapors
Through Flame-Ionization

January, 1991

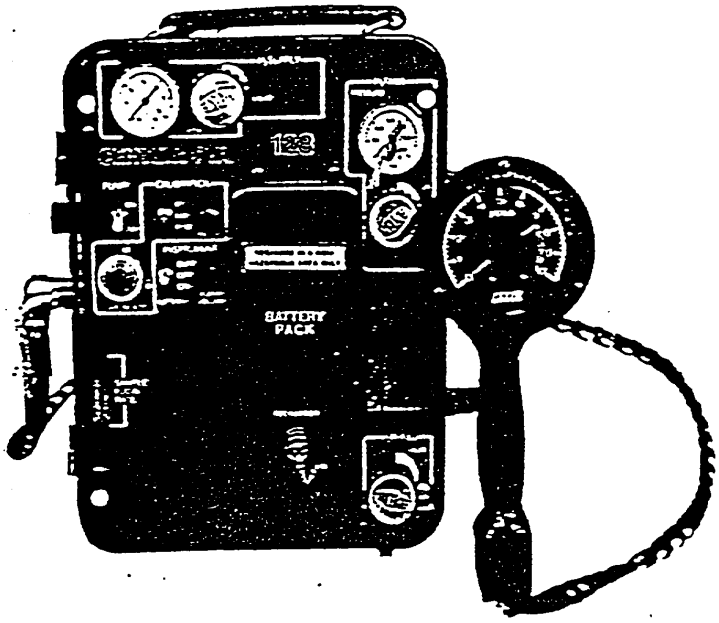
SCOPE AND APPLICATION

The OVA 128 is a sensitive instrument designed to measure trace quantities of organic materials in the air. The instrument uses a hydrogen flame ionization detector with the sensitivity to measure in the parts per million (ppm) range (V/V) in the presence of atmospheric moisture, nitrogen oxides, carbon monoxide, and carbon dioxide. The instrument has a single linearly scaled readout from 0 to 10 ppm with a X1, X10, and X100 range switch.

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.

Reference:
Instruction Manual Model OVA 128, Century Organic Vapor Analyzer.

Apparatus:
OVA Model 128.
Battery Charger.
Side Pack Assembly and the Probe/Readout Assembly (See Figure 1).
Recorder.
Calibration Kit - Kit containing a gas cylinder of Zero air, a gas cylinder of 95 to 100 ppm Span Gas (Methane in air), and gas regulators.
Gas chromatographic columns.



ORGANIC VAPOR ANALYZER

FIGURE 1

1.0 CONTROLS AND INDICATORS

1.1 Side Pack Assembly

1.1.1 INSTR/BATT Test Switch - three position toggle switch controls all instrument electrical power except the pump and alarm power. It also permits the display of the battery charge condition on the readout meter.

1.1.2 PUMP (ON/OFF) Switch - toggle switch controls power to the internal pump and audio alarms.

1.1.3 IGNITER Switch - momentary push button switch connects the power to the igniter coil in the detector chamber and simultaneously disconnects power to pump.

1.1.4 CALIBRATE Switch (range selector) - selects the desired range: X1 (0 to 10 ppm); X10 (0 to 100 ppm); X100 (0 to 1000 ppm).

1.1.5 CALIBRATE ADJUST (zero) knob - potentiometer used to zero the instrument.

1.1.6 GAS SELECT KNOB (span control) - ten-turn dial readout potentiometer sets the gain of the instrument (commonly referred to as span control).

1.1.7 Recorder connector - five-pin connector used to connect the instrument to the recorder.

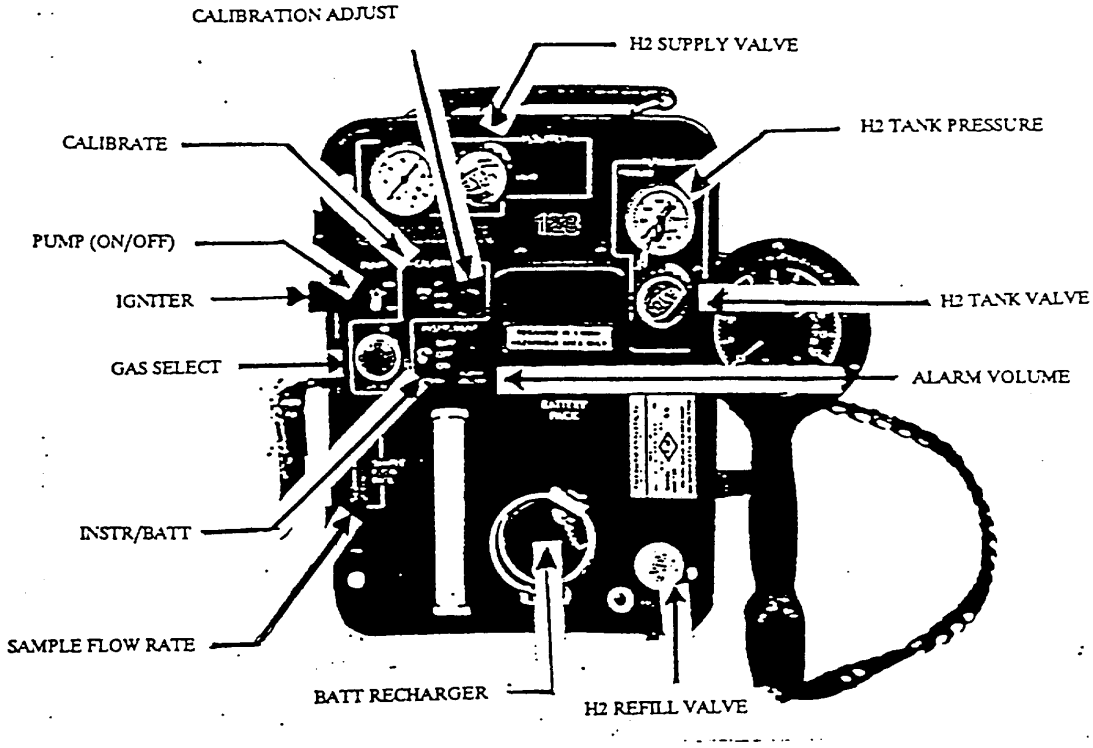
1.1.8 Charger connector - BNC connector used to connect the battery pack to the battery charger.

1.1.9 HYDROGEN TANK VALVE - valve used to supply or close off the fuel supply from the hydrogen tank.

1.1.10 HYDROGEN TANK PRESSURE indicator - high pressure gauge measures pressure in the hydrogen fuel tank which is an indication of fuel supply.

1.1.11 HYDROGEN SUPPLY VALVE - valve used to supply or close off hydrogen fuel to the detector chamber.

1.1.12 HYDROGEN SUPPLY PRESSURE indicator - low pressure gauge used to monitor hydrogen pressure at the capillary restrictor.



CONTROLS AND INDICATORS

FIGURE 2

1.1.14 REFILL CONNECTION - 1/4 in. AN fitting to connect the hydrogen refill hose to the instrument.

1.1.15 REFILL VALVE - valve to open one end of the instrument fuel tank for refilling with hydrogen.

1.1.16 EARPHONE JACK - used to connect the earphone.

1.1.17 VOLUME knob - potentiometer adjusts the volume of the internal speaker and earphone.

1.1.18 Readout and sample connectors - used to connect the sample hose and the cord from the probe/readout to the side pack.

2.0 STARTUP AND OPERATING PROCEDURES (Survey Mode)

2.1 Connect the Probe/Readout assembly to the sidepack assembly by attaching the sample line and electronic jack to the sidepack.

2.2 Select the desired sample probe and connect the probe handle. Before tightening the knurled nut, check that the probe accessory is firmly seated against the flat seals in the probe handle and in the tip of the telescoping probe.

2.3 Move the Instrument/Battery switch to the test position. The meter needle should move to a point beyond the white line, indicating that the battery has more than four hours of operating life before charging is necessary.

2.4 Move the Instrument/Battery switch to the "ON" position and allow a 5 minute warmup.

2.5 Turn the pump switch on.

2.6 Use the calibrate adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the calibrate switch must be set to the appropriate range.

2.7 Turn the volume knob fully clockwise.

2.8 Using the alarm level adjust knob, turn the knob until the audible alarm is activated.

2.9 Move the calibrate switch to X1 and adjust the meter reading to zero using the calibrate adjust (zero) knob.

2.10 Open the hydrogen tank valve 1 or 2 turns and observe the reading on the hydrogen tank pressure indicator. (Approximately 150 psi of pressure is required for each hour of operation).

2.11 Open the hydrogen supply valve 1 or 2 turns and observe the reading on the hydrogen supply pressure indicator. The reading should be between 8 and 12 psi.

2.12 After approximately one minute, depress the igniter button until the hydrogen flame lights. The meter needle will travel upscale and begin to read "total organic vapors". CAUTION: Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.

2.13 The instrument is ready for use.

2.14 NOTE: Use calibrate knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set calibrate switch to X1 and readjust the zero on the meter. To avoid false flame-out alarm indicator, set mete to 1 PPM with calibrate knob and make the differential readings from here. If the ambient background organic vapors are "zeroed out" using the calibrate adjust knob, the needle may move off-scale in the negative direction when the OVA is moved to a location with lower background.

3.0 SHUT DOWN

3.1 Close the hydrogen supply valve

3.2 Close the hydrogen tank valve

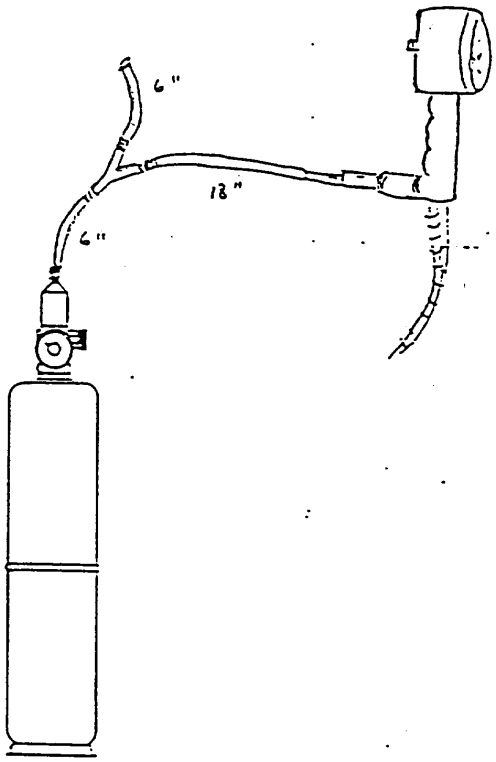
3.3 Move the instrument switch and pump switch to OFF.

4.0 CALIBRATION

Calibration by using a known methane concentration in air in the range of 95-100 ppm.

4.1 Attach one 6" piece of tubing to the regulator. The other 6" piece is the vent.

4.2 Attach the 18" branch length of tubing to the readout assembly. (See Figure 3).



CALIBRATION CYLINDER/PROBE ASSEMBLY

FIGURE 3

4.3 Place the instrument in normal operation and allow a minimum of 15 minutes for warm-up and stabilization.

4.4 Set the Gas Select control to ~~300~~. 30

4.5 Use the zero air cylinder and with the calibrate adjust zero knob adjust the meter reading to zero.

4.6 Place instrument in normal operation with calibrate switch set to X10 and gas select control set to 300.

4.7 Introduce a methane sample of a known concentration (between 90 and 100 ppm, not to exceed 100 ppm) and adjust trim pot R-32 so the meter reading corresponds to the known sample.

4.8 The instrument has been calibrated for methane with the panel mounted gain adjustment (gas select) set at reference 300.

4.9 Turn off hydrogen supply valve to put out the flame.

4.10 Leave calibrate switch on X10 position and use calibrate adjust (zero) knob to adjust meter reading to 4 ppm.

4.11 Place calibrate switch in X1 position and using trim pot R-31 adjust meter reading to 4 ppm.

4.12 Move calibrate switch to X10 position again. use calibrate adjust (zero) knob to adjust meter to a reading of 40 ppm.

4.13 Move calibrate switch to X100 position and use trim pot R-33 to adjust meter reading 40 ppm.

4.14 Move calibrate adjust (zero) knob to adjust meter reading to zero.

4.15 Unit is now balanced from range, calibrated to methane, and ready to use.

Appendix B

Analytical Services Standard Operating Procedures (SOPs)

Appendix B1

Alkalinity

Effective: 5-23-91

ALKALINITY - AUTOANALYZER

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

Reference: EPA 1983, Method 310.2 Lachat Instruments 1988, Method 10-303-31-1-A

Sample Handling: Refrigerate at 4°C and analyze within 14 days of collection.

Detection Limit: 10 mg/L as $CaCO_3$

Optimum Range: 10 - 500 mg/L

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 40 seconds
3. Load period: 25 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 5 seconds
6. Inject to end of peak period: 44 seconds
7. Gain: 150 x 10
8. Zero: 180
9. Interference filter: 410 nm
10. Sample loop: 90 cm
11. Standards for curve set-up: 0, 20, 50, 100, 250, 500 mg/L.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassed Milli-Q water - 2 options:**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20 L.) Store in cubitainer.

2. **Stock alkalinity standard (1000 mg/L as $CaCO_3$):** In a 1 liter volumetric flask, dissolve 1.060 g of anhydrous primary standard grade sodium carbonate (Na_2CO_3- dried at 250°C for 4 hours) in approximately 900 mL of helium purged Milli-Q water, and dilute to mark.

3. **Standards:** (Prepare fresh every month). Dilute to volume using degassed Milli-Q water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of Alk. Stock	Dilute to
0 mg/L	A	0	200 mL
20 mg/L	B	4.0	200 mL
50 mg/L	C	10	200 mL
100 mg/L	D	50	500 mL
250 mg/L	E	125	500 mL
500 mg/L	F	100	200 mL
CRDL 10 mg/L		2.0	200 mL

Note: Final volumes are not the same! Computer refers to standards by letter.

4. **Hydrochloric acid (0.1M):** In a 1 liter flask, dilute 8.3 mL of concentrated HCL in D.I. water and dilute to the mark.

5. **KHP buffer (0.025 M, pH 3.1):** In a 1 liter flask, dissolve 5.10 g of primary standard grade potassium acid phthalate (KHP) ($KHC_8H_4O_4$) in approximately 800 mL of Milli-Q water. Add 70 mL of 0.1M HCl, then carefully, add additional acid to bring pH to between 3.10 and 3.12. **Note:** Use only 0.1M HCL to adjust the pH of buffer. Do **not** back adjust the pH with NaOH. Vacuum filter through a 0.45 micron membrane filter. **Store In Glass and Prepare Daily!**

6. **Methyl orange reagent:** In a 1 liter volumetric flask, dissolve 0.125 g of methyl orange indicator in about 700 mL of Milli-Q water and dilute to the mark. **Mix well and vacuum filter through a 0.45 micron membrane filter before each use.** Store in amber glass! **Note:** Amount of methyl orange may be adjusted to correct for variances in lots of methyl orange.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero settings are guidelines and must be optimized each day.

3. The alkalinity standards can be combined with chloride and sulfate standards for use with the 3 channel method.

4. Turbidity will interfere. Samples must be filtered prior to analysis. (Use Whatman #1 or #4 filter paper.)

5. Color will interfere, dilute the sample and also spike the dilution to confirm the quality of the result.

System Operation:

1. Refer to "Auto Analyzer Operation start-up procedure." (IOP# LAAC-Section A).

2. Analyze an initial calibration check standard, a blank, a CRDL standard and a known reference standard at the beginning of each run. The blank must be below the detection limit and the standards must be within required control limits before any samples are analyzed.

3. Spike samples by mixing sample with an equal volume of 250 mg/L standard (E), for a final spike level of 125 mg/L.

4. The calibration check standard is 100 mg/L (D).

5. Refer to "Auto Analyzer shut-down procedure". (IOP# LAAC-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. The derived concentration of each calibration must be $\pm$ 10% of the true value. The derived value for the blank must be below the method detection limit.

2. The CRDL standard concentration should be at the method detection limit (MDL). Results must be within the acceptable limits of ± the MDL or the instrument must be re-calibrated before samples are analyzed.

3. A quality control calibration check standard of 100 mg/L (D) and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. Standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. See LAAC SOP for further detail.

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

    a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

    b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

    c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

    d. Select "Enter Sample ID's". Press <enter>.

    e. Type in sample information. Check standards will automatically be placed in the tray information portion.

    f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

    After calibration is complete:

    - go into the foreground (Press Alt, Esc keys)
    - select "display calibration graph" (Press <enter>)
    - review the data
    - return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.
- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray <u>before</u> pressing "R"!

B. <u>Instrument Shut-Down</u>

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.
2. If column is used, stop the pump and disconnect from manifold.
3. Pull lines from reagents into a wash beaker of D.I..
4. Pump D.I. through lines for 2-5 minutes.
5. Pump air through lines until manifold is dry.
6. Turn off pump.
7. Release tubing cartridges and lower tension levers. Release tubing.
8. Turn off main switch on rear power strip.
9. Empty and rinse waste containers, if necessary.
10. Perform back-up on current data files, once a week. (See Section C)
11. Turn off the computer and printer.

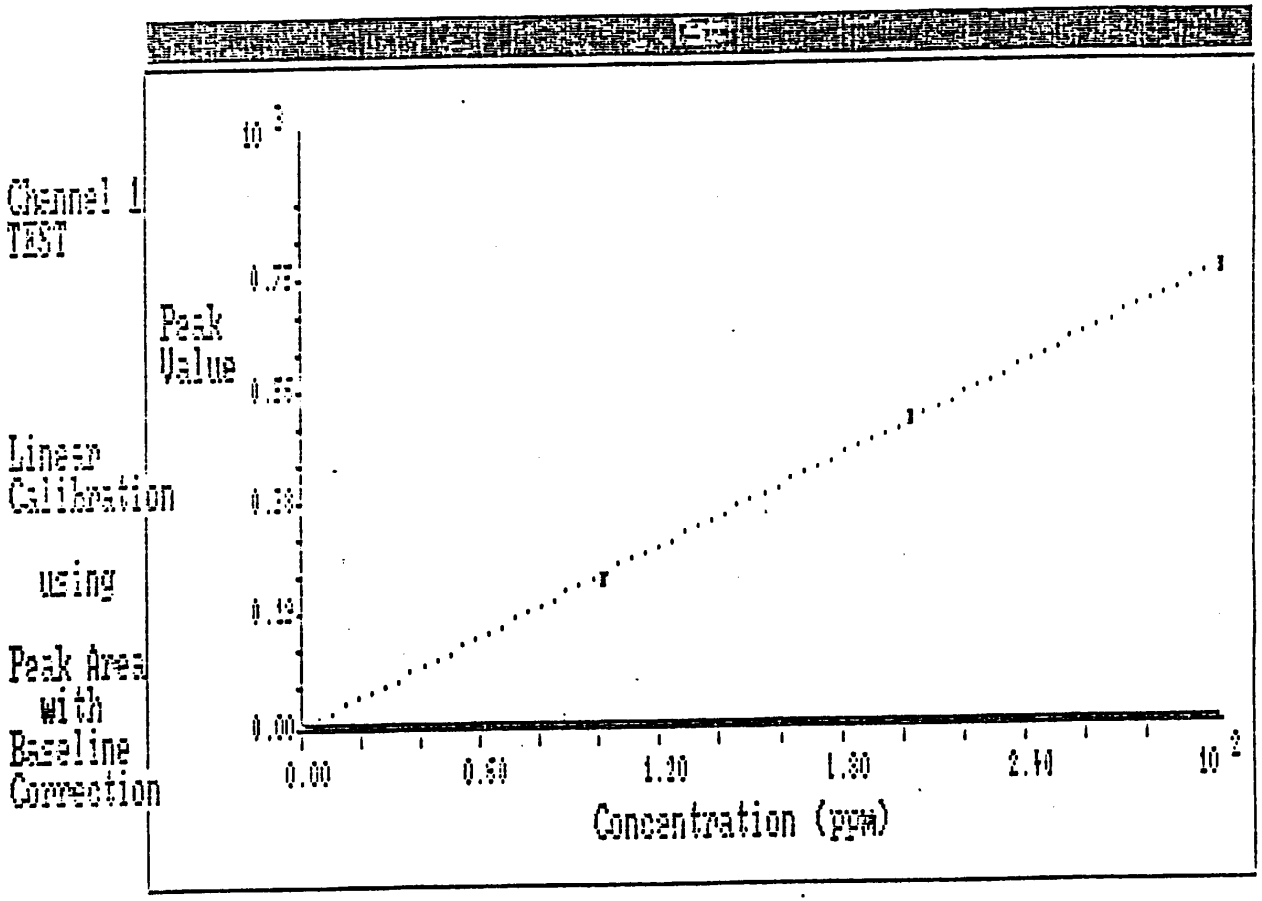
C. <u>Backing-up the Data Files</u>

1. Exit to DOS
2. At C> Type: cd\fialab\data Press <enter>
3. At C> Type: copy *.rpt a: Press <enter>
   After everything is copied - remove disc.
4. At C> Type: del *.* Press <enter>
5. Are you sure (Y/N)? Type: Y Press <enter>
6. At C> Type: cd\ Press <enter>
7. Turn off the red switch on the computer power strip to turn off the computer, printer and screen.

D. Quikchem Calibration

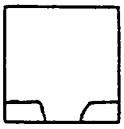
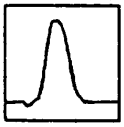
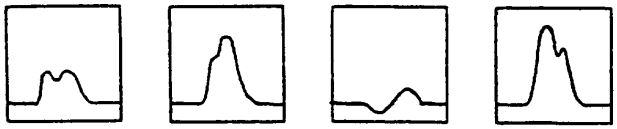
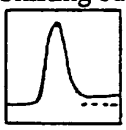
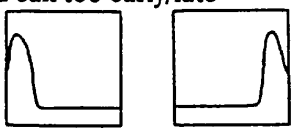
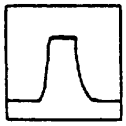
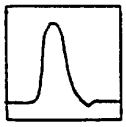
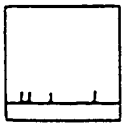
QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

FIGURE 1



Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

LACHAT TROUBLESHOOTING GUIDE

Problem:	Cause:
Negative peak 	1. Contaminated carrier 2. Bad or no SO_4 column or sample extremely high in hardness 3. Samples high in oxidizing agents
Reproduceable dip when valve switches (before peak) 	1. Bad color reagent 2. Could also be valve turning artifact caused by a highly colored reagent
Odd looking peaks 	1. Index of refraction problem, matrix related (usually acid or pH buffering) 2. Also method interferences: high hardness on SO_4 method, oxidizing samples on nitrate method 3. Bad reagents
Shifting baseline 	1. Flow problem 2. Precipitate build-up on SO_4 manifold tubing
Peak too early/late 	1. Usually flow problem 2. Valve not set to an initial load position state before starting run 3. Incorrect pump setting or other timing problem
Peak cut off in window 	1. Reagents exceeded-reagents improperly prepared 2. Standards incorrectly prepared
Reproduceable dip after peak 	1. Bad column
Small intermittent peaks 	1. Milli-Q or reagents not degassed properly or adequately 2. Waste line coil not installed (for the methods which require it)

Three basic areas of troubleshooting:
1. Fluidics (clogs, old pump tubing, crimp in manifold tubing)
2. Chemistry
3. Timing (not usually a problem after initial development)

BLH/rff
[rff-genpol-604]

Appendix B2

Chloride

Effective: 5-14-91

CHLORIDE - AUTOANALYZER

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

Reference: EPA 1983, Method 325.2
Lachat Instruments 1987, Method 10-117-07-1-B

Detection Limit: 2 mg/L.

Optimum Range: 2 - 100 mg/L

Sample Handling: Refrigerate at 4°C and analyze within 28 days of collection.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 30 seconds
3. Load period: 8 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 8 seconds
6. Inject to end of peak period: 31 seconds
7. Gain: 200
8. Zero: 250
9. Interference filter: 480 nm
10. Sample loop: 20 cm
11. Standards for curve set-up: 0, 10, 20, 50, 80, and 100 mg/L

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated.)

1. Degassed Milli-Q water - 2 options:

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20 L.) Store in cubitainer.

2. **Stock chloride standard (1000 mg/L):** Dry 2 g of primary grade sodium chloride (NaCl) at 140°C overnight. In a 1 liter volumetric flask, dissolve 1.648 g of dried primary grade sodium chloride (NaCl) in 500 mL D.I. water. Dilute to the mark and invert to mix. Refrigerate.

3. **Standards:** (Prepare fresh every month). Dilute to volume using D.I. water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of Chloride Stock (ml)	Dilute to
0 mg/L	A	0	200 mL
10 mg/L	B	2.0	200 mL
20 mg/L	C	4.0	200 mL
50 mg/L	D	25	500 mL
80 mg/L	E	40	500 mL
100 mg/L	F	20	200 mL
CRDL 2 mg/L		2.0	1000 mL

Note: Computer refers to standards by letter. Final volumes are not the same!

4. **Stock mercuric thiocyanate reagent:** In a 1 liter volumetric flask, dissolve 4.17 g of mercuric thiocyanate ($Hg(SCN)_2$) in one liter of methanol. Invert to mix. Store in amber glass. Refrigerate.

   Caution: Mercury is a very toxic metal. **Wear gloves!**

5. **Stock ferric nitrate reagent (0.5M):** In a 1 liter volumetric flask, dissolve 202.0 g of ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$ in approximately 800 mL of deionized water. Add 25 mL of concentrated nitric acid and dilute to one liter. Invert to mix. Refrigerate.

6. **Combined color reagent:** Mix 150 mL of stock mercuric thiocyanate solution with 150 mL of stock ferric nitrate reagent and dilute to 1000 mL with deionized water. Vacuum filter through a 0.45 micron membrane filter. Store at room temperature. Do Not Refrigerate.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero settings are guidelines and must be optimized each day.

3. The chloride standards may be combined with alkalinity and sulfate standards for use with the 3 channel method.

4. Any sample with turbidity must be filtered prior to analysis. (Use Whatman #1 or #4 filter paper.) Record on data sheet.

5. Color is an interference, dilute the sample and also spike the dilution to confirm the quality of the result. Record on the data sheet.

System Operation:

1. Refer to "Auto Analyzer Operation start-up procedure." (IOP# LAAC Section A).

2. Analyze an initial calibration check standard, a blank, a CRDL standard and a known reference standard at the beginning of each run. The blank must be below the detection limit and the standards must be within required control limits before any samples are analyzed.

3. To spike samples, mix equal volumes of sample and 80 mg/L Cl standard (E) for a final spike level of 40 mg/L Cl.

4. The calibration check standard is 50 mg/L (D).

5. Refer to "Auto Analyzer shut-down procedure". (IOP# LAA-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. Note that the calibration curve is calculated in a "piece-wise" fashion and is not linear. Be sure that calibration points describe a smooth curve.

2. The CRDL standard concentration should be at the method detection limit (MDL). Results must be within the acceptable limits of ± the MDL or the instrument must be re-calibrated before samples are analyzed.

3. A quality control calibration standard of 50.0 mg/L and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. See LAA SOP for further detail.

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

    a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

    b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

    c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

    d. Select "Enter Sample ID's". Press <enter>.

    e. Type in sample information. Check standards will automatically be placed in the tray information portion.

    f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

    After calibration is complete:

    - go into the foreground (Press Alt, Esc keys)
    - select "display calibration graph" (Press <enter>)
    - review the data
    - return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.
- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray <u>before</u> pressing "R"!

B. <u>Instrument Shut-Down</u>

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.
2. If column is used, stop the pump and disconnect from manifold.
3. Pull lines from reagents into a wash beaker of D.I..
4. Pump D.I. through lines for 2-5 minutes.
5. Pump air through lines until manifold is dry.
6. Turn off pump.
7. Release tubing cartridges and lower tension levers. Release tubing.
8. Turn off main switch on rear power strip.
9. Empty and rinse waste containers, if necessary.
10. Perform back-up on current data files, once a week. (See Section C)
11. Turn off the computer and printer.

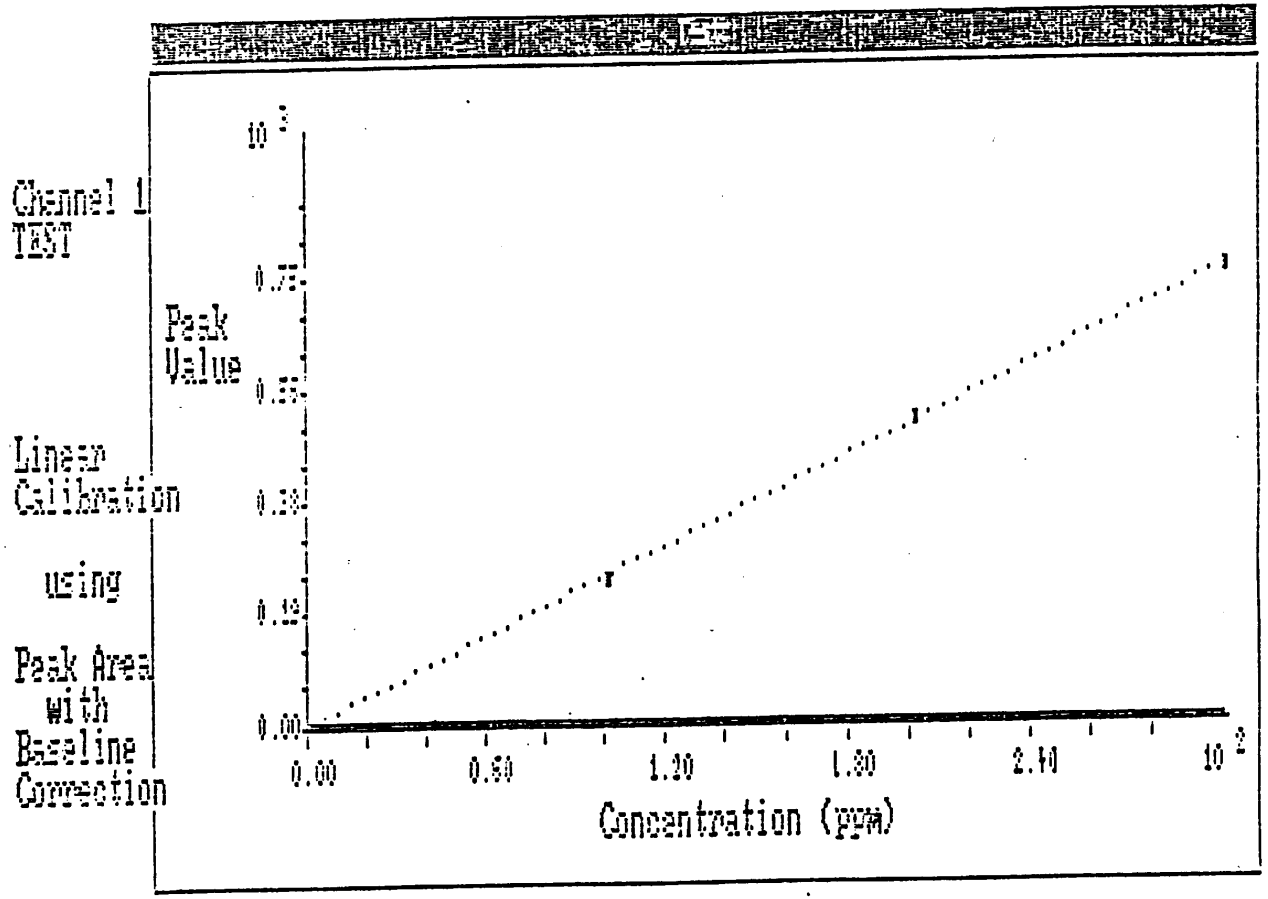
C. <u>Backing-up the Data Files</u>

1. Exit to DOS
2. At C> Type: cd\fialab\data Press <enter>
3. At C> Type: copy *.rpt a: Press <enter>
   After everything is copied - remove disc.
4. At C> Type: del *.* Press <enter>
5. Are you sure (Y/N)? Type: Y Press <enter>
6. At C> Type: cd\ Press <enter>
7. Turn off the red switch on the computer power strip to turn off the computer, printer and screen.

D. Quikchem Calibration

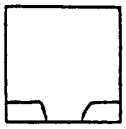
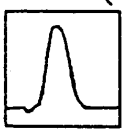
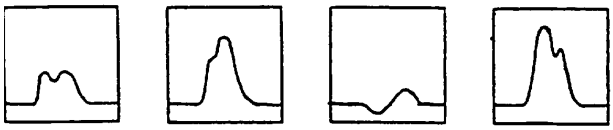
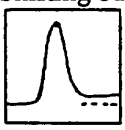
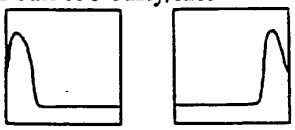
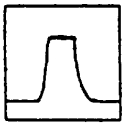
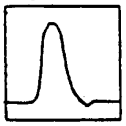
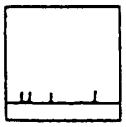
QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

FIGURE 1



Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

LACHAT TROUBLESHOOTING GUIDE

Problem:	Cause:
Negative peak 	1. Contaminated carrier 2. Bad or no SO_4 column or sample extremely high in hardness 3. Samples high in oxidizing agents
Reproduceable dip when valve switches (before peak) 	1. Bad color reagent 2. Could also be valve turning artifact caused by a highly colored reagent
Odd looking peaks 	1. Index of refraction problem, matrix related (usually acid or pH buffering) 2. Also method interferences: high hardness on SO_4 method, oxidizing samples on nitrate method 3. Bad reagents
Shifting baseline 	1. Flow problem 2. Precipitate build-up on SO_4 manifold tubing
Peak too early/late 	1. Usually flow problem 2. Valve not set to an initial load position state before starting run 3. Incorrect pump setting or other timing problem
Peak cut off in window 	1. Reagents exceeded-reagents improperly prepared 2. Standards incorrectly prepared
Reproduceable dip after peak 	1. Bad column
Small intermittent peaks 	1. Milli-Q or reagents not degassed properly or adequately 2. Waste line coil not installed (for the methods which require it)

Three basic areas of troubleshooting:
1. Fluidics (clogs, old pump tubing, crimp in manifold tubing)
2. Chemistry
3. Timing (not usually a problem after initial development)

BLH/rff
[rff-genpol-604]

Appendix B3

Sulfate

Effective: 5-23-91

SULFATE - AUTOANALYZER

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

Reference: EPA 1989, Method 375.2
Lachat Instruments 1989, Method 10-116-10-2-B

Detection Limit: 10 mg/L.

Optimum Range: 10 - 200 mg/L

Sample Handling: Refrigerate at 4°C and analyze within 28 days of collection.

Instrument Conditions:

1. Pump speed: 35 seconds
2. Load period: 10 seconds
3. Inject period: 30 seconds
4. Inject to start of peak period: 10 seconds
5. Inject to end of peak period: 56 seconds
6. Cycle period: 50 seconds
7. Gain: 700
8. Zero: 200
9. Interference filter: 460 nm
10. Sample loop: 10 cm
11. Standards for curve set-up: 0, 25, 50, 100, 150, 200 mg/L.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassing with helium - 2 options:**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20 L.) Store in cubitainer.

2. **Carrier (0.3 ppm SO_4):** In a 1 liter volumetric flask, add 0.3 mL of 1000 ppm stock sulfate solution and dilute to mark with degassed Milli-Q water.

3. **Barium chloride solution (6.24mM):** In a 1 liter volumetric flask, dissolve 1.526 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in 500 mL of Milli-Q water and dilute to 1 liter.

4. **Hydrochloric acid (1.0N):** In a 100 mL volumetric flask, containing approximately 80 mL of Milli-Q water, add 8.3 mL of concentrated hydrochloric acid and dilute to the mark with Milli-Q water.

5. **Barium - MTB color reagent:** (The purity of the methylthymol blue and the alcohol can be critical. **Use the sources stated below**).

In a dry 1000 mL volumetric flask, place 0.2364* g of methylthymol blue (3', 3" bis-N, N-bis carboxymethyl)-amino methylthymolsulfon-ephthalein pentasodium salt (Kodak No. 8068). Add 50 mL of barium chloride solution ("3" above). The solution may be used to aid in the transfer of the dye. Swirl to dissolve. Add 8.0 mL of the 1.0N HCL solution ("4" above) and mix - solution may turn orange. Add 142 mL deionized water and dilute to 1000 mL with ethanol (Aldrich 24.511.9) Mix. The pH of this solution should be 2.5. Prepare this solution the day before use and store it refrigerated in an amber bottle.

* Strength of reagent varies with lot number. The optimum weight of reagent used must be determined specifically for each new lot of MTB to maintenance consistent method sensitivity. This weight is determined by Lachat and supplied with each batch purchased.

6. **Sodium hydroxide (50% stock solution): Cautiously** dissolve 500 g of sodium hydroxide (NaOH) in 600 mL of Milli-Q water. Cool and dilute to 1 liter. Store in plastic bottle. **Caution: The solution will become very hot!**

7. **Sodium hydroxide (0.18 N):** In a 1 liter volumetric flask, add 14.4 mL of 50% sodium hydroxide ("6" above) to degassed Milli-Q water, and dilute to the mark.

8. **Buffered EDTA (for cleaning manifold):** In a 1 liter volumetric flask, dissolve 6.75 g ammonium chloride (NH_4Cl) in 500 mL DI water. Add 57 mL concentrated ammonium hydroxide and 40.0 g tetrasodium EDTA dihydrate. Dissolve by swirling; dilute to the mark with DI water.

9. **Sulfate stock (1000 mg/L):** Dry approximately 2 g of sodium sulfate (Na_2SO_4) at 105°C for 2 hours. Cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.479 g of the dried sodium sulfate in Milli-Q water and dilute to 1 liter. (1.0 mL = 1.0 mg SO_4).

10. **Working standard:** (Prepare fresh every month). Dilute to volume using degassed Milli-Q water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of Sulfate Standard	Dilute to
0 mg/L	A	0	200 mL
25 mg/L	B	5.0	200 mL
50 mg/L	C	10	200 mL
100 mg/L	D	50	500 mL
150 mg/L	E	75	500 mL
200 mg/L	F	40	200 mL
CRDL 10 mg/L		2.0	200 mL

Note: Final volumes are not the same! Computer refers to standards by letter.

Preparation of Ion Exchange Column:

1. Make a slurry of approximately 0.5 g of BioRex 70, 50-100 mesh ion exchange resin in Milli-Q water.

2. Remove one column end from the glass column. Fill the column with water, then aspirate the slurry or allow it to settle by gravity to pack the column. Take care to **avoid trapping air bubbles** in the column and its fittings at this point and all subsequent operations.

3. After the resin has settled, replace the end fitting. To ensure a good seal, remove any resin particles from the threads of the glass, the column end and the end fittings. To store the column, the ends of the Teflon tubing may be joined with a union.

4. To test the effectiveness of the column, make up a standard of pure sodium sulfate and compare its peak height to an identical standard with hardness typical of the samples added. If the column is being depleted, the standard with hardness will read lower because the divalent cations are complexing the free MTB. The concentration of the standard should be mid-range. If depletion has occurred, repack the column with fresh resin.

5. **Regenerating Resin:** Batch regeneration is recommended because the hydrogen form of BioRex 70 can swell considerable more than the sodium form. Collect the used resin in a small beaker or flask. Wash with dilute HCL until the wash tests free of calcium and/or magnesium. This procedure removes the divalent cations by converting the carboxylate exchange group to the protonated form - COOH. Convert the resin back to the sodium form by neutralizing with washes of 0.5M NaOH until the wash has a pH of 9 or greater. Rinse with deionized water for storage or repacking. A column may be used for 3-4 trays (approximately 150 samples) before it needs to be replaced.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. Sulfate standards may be combined with alkalinity and chloride standards for use with the 3-channel method.

3. The gain and zero settings are guidelines and must be optimized each day.

4. All coils (including waste coil) must be changed at least once each quarter to prevent build-up in lines.

4. **Interferences:**

   - The cation exchange column removes multivalent cations. Run a mid-range sulfate standard containing a typical concentration of $CaCO_3$ periodically to check performance. Any decrease in peak height should indicate the need to regenerate or replace the resin. (At 600 ppm $CaCO_3$, the column is good for 80 + injections.)

- Samples with pH <2 should be neutralized. High acid concentrations can displace multivalent cations from the column.
- Color will interfere. Dilute the sample and also spike the dilution to confirm the quality of the result. Record on the data sheet.
- Turbidity - turbid samples may be filtered (use Whatman #1 or #4 filter paper) prior to analysis on Lachat.
- Orthophosphate also forms a precipitate with barium at high pH. Check the response of pure orthophosphate standards, if samples are known to be high in phosphate.

5. **Troubleshooting:** Baseline noise with reagents pumping.

   a. Noise with column in line but good baseline without column.

      - Repack column, **air bubbles** may be causing pulsing.
      - Check flow fit connectors and end fittings on column for blockage or leaks.

   b. Noise with and without column in line.

      - Degas carrier and/or reagents. Fine bubbles cause sharp spikes on baseline.
      - Place a longer piece of manifold tubing on the outlet of the flow cell leading to the waste container. This method requires the use of the screw type flow cell.
      - Replace the pump tubes. The **silicone tube,** used for the color reagent, wears faster than the PVC pump tubes.
      - With water pumping in the lines, check all hydraulic connections for blockages, leaks, etc.

Baseline drift:

1. Clean the manifold with the buffered EDTA.
2. Turn the gain high and use the shortest sample loop possible. This improves the linearity of the calibration curve, prolongs the useful life of the column, and minimizes the build up of $BaSO_4$ on the manifold tubing.

System Operation:

1. Refer to "Auto Analyzer Operation start-up procedure." (SOP# LAAC-Section A).

2. Pump reagents through the lines **before** inserting the column. Use a short piece of manifold tubing in place of the column. When all air has passed and the baseline is steady, turn **off** the pump and insert the column. The column should be placed in a vertical position with flow in the top and out the bottom. In this configuration, the column will operate effectively even if the resin packs down more to leave a gap at the top. Resume pumping.

3. Analyze an initial calibration check standard, a blank, a CRDL standard and a known reference at the beginning of each run. The blank must be less than the detection limit and the standards must be within required control limits before any samples are analyzed.

4. **To spike:** Mix equal volumes of sample and 150 mg/L standard (E) for a final spike level of 75 mg/L.

5. The calibration check standard is 100 mg/L (D).

6. To shut down, turn off pump and remove the column.

   To remove the column:

   a. Turn off the pump.
   b. Remove the column.
   c. Join ends of the column with a union.
   d. Replace the column on the manifold with the short teflon tubing piece.
   e. Rinse manifold with Milli-Q water.
   f. Rinse manifold with EDTA cleaning solution.
   g. Continue with "Auto Analyzer Shut-down procedures" (SOP # LAAC-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. Note that the calibration curve is calculated in a "piece-wise" fashion and is not linear. Be sure that calibration points describe smooth curve. If not, necessary troubleshooting must be performed before continuing (check reagents, pump tubing, valves, etc.).

2. The CRDL standard concentration should be at the method detection limit (MDL). Results must be within the acceptable limits of ± the MDL or the instrument must be recalibrated before samples are analyzed.

3. A quality control calibration check standard of 100 mg/L and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. Standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. See LAAC SOP for further detail.

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

d. Select "Enter Sample ID's". Press <enter>.

e. Type in sample information. Check standards will automatically be placed in the tray information portion.

f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

After calibration is complete:

- go into the foreground (Press Alt, Esc keys)
- select "display calibration graph" (Press <enter>)
- review the data
- return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.
- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray <u>before</u> pressing "R"!

B. <u>Instrument Shut-Down</u>

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.
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3. Pull lines from reagents into a wash beaker of D.I..
4. Pump D.I. through lines for 2-5 minutes.
5. Pump air through lines until manifold is dry.
6. Turn off pump.
7. Release tubing cartridges and lower tension levers. Release tubing.
8. Turn off main switch on rear power strip.
9. Empty and rinse waste containers, if necessary.
10. Perform back-up on current data files, once a week. (See Section C)
11. Turn off the computer and printer.

C. <u>Backing-up the Data Files</u>

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2. At C> Type: cd\fialab\data Press <enter>
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D. Quikchem Calibration

QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

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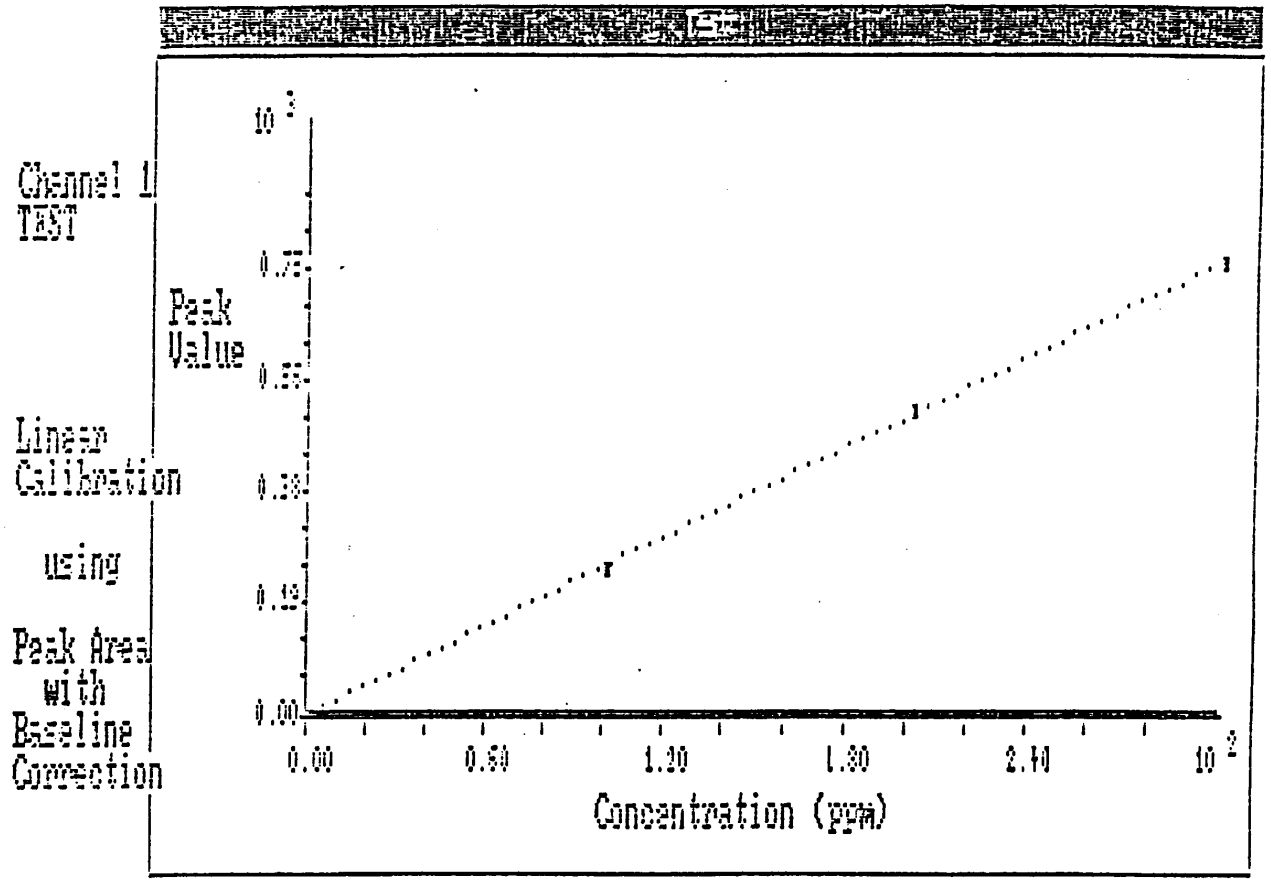
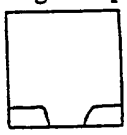
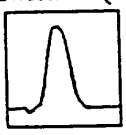
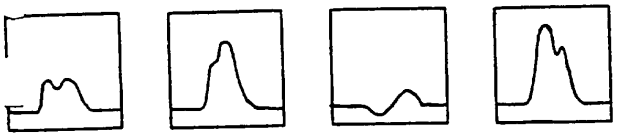
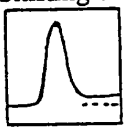
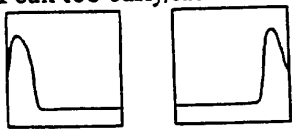
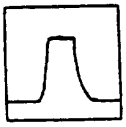
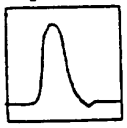
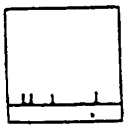


Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

LACHAT TROUBLESHOOTING GUIDE

Problem:	Cause:
Negative peak 	1. Contaminated carrier 2. Bad or no SO_4 column or sample extremely high in hardness 3. Samples high in oxidizing agents
Reproduceable dip when valve switches (before peak) 	1. Bad color reagent 2. Could also be valve turning artifact caused by a highly colored reagent
Odd looking peaks 	1. Index of refraction problem, matrix related (usually acid or pH buffering) 2. Also method interferences: high hardness on SO_4 method, oxidizing samples on nitrate method 3. Bad reagents
Shifting baseline 	1. Flow problem 2. Precipitate build-up on SO_4 manifold tubing
Peak too early/late 	1. Usually flow problem 2. Valve not set to an initial load position state before starting run 3. Incorrect pump setting or other timing problem
Peak cut off in window 	1. Reagents exceeded-reagents improperly prepared 2. Standards incorrectly prepared
Reproduceable dip after peak 	1. Bad column
Small intermittent peaks 	1. Milli-Q or reagents not degassed properly or adequately 2. Waste line coil not installed (for the methods which require it)

Three basic areas of troubleshooting:
1. Fluidics (clogs, old pump tubing, crimp in manifold tubing)
2. Chemistry
3. Timing (not usually a problem after initial development)

BLH/rff
[rff-genpol-604]

Appendix B4

Total Dissolved Solids

Effective Date: 5-3-91

TOTAL DISSOLVED SOLIDS

Scope and Application: This method is applicable to drinking water, surface water, groundwater, and domestic and industrial wastewaters.

Method: Gravimetric, dried at 180°C.

Reference: EPA 1983, Method 160.1

Detection Limit: 10 mg/L (using a 100 mL sample volume)

Sample Handling: Refrigerate at 4°C and analyze sample within 7 days of sampling.

Reagents and Apparatus:

1. Glass fiber filters, Whatman GF/C
2. Gelman filtration funnel and support
3. Suction flask, 1000 mL
4. Porcelain evaporating dishes
5. Volumetric pipet, 50 mL
6. Drying oven at 180°C ± 2°C
7. Desiccator
8. Analytical balance
9. Deionized water

Notes:

1. **Interferences:** Samples with high concentrations of bicarbonate, Ca, Mg, Cl, and SO_4 will require prolonged drying, desiccation, and rapid weighing.

2. Total residue should be < 200 mg. Excessive residue (>200 mg) is difficult to dry thoroughly. Use a smaller volume if TDS is suspected to be high; likewise use a larger volume if TDS is suspected low.

3. Groundwater samples which have already been filtered through a 0.45 micron membrane filter do not need to be carried through the filtration step of the procedure.

Procedure:

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

2. **Evaporating Dish Preparation:** If volatile dissolved solids are also to be analyzed, prepare the evaporating dishes by ashing at 550 ± 50°C for one hour in a muffle furnace.

Otherwise, heat the dishes at 180 ± 2°C for one hour. Cool in desiccator. Weigh. Record the weight. The dishes must be cool before being weighed (about one hour). Repeat this cycle until a constant weight is obtained (± 0.5 mg).

3. **Filter Preparation:** Place the glass fiber filter on the filtration support, place the funnel on top, and wash the filter with three 20 mL portions of deionized water while vacuum is applied. Discard the washings. The filters may be prepared ahead of time. If this is the case, dry them for 1 hour at 103 - 105°C and store in the desiccator until needed.

4. Assemble the filtering apparatus, place a prepared filter on the support and begin suction. Shake the sample and measure out 100 mL in graduated cylinder.

5. Pour sample into filtering apparatus. Apply vacuum until all the sample has been filtered.

6. Pipet 50 mL of filtered sample (less, if the sample is expected to have a high dissolved solids content) into a prepared evaporating dish.

7. Evaporate the sample to dryness in the oven at 180 ± 2°C. Cool in a desiccator for at least one hour and weigh. Repeat the drying cycle until the weight loss is ≤ 0.5 mg.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be within acceptable ranges, or data must be flagged appropriately.

2. A blank must be analyzed with every set of samples. (This is a check on contamination, cleanliness of dishes, oven, pipettes, etc.)

3. An EPA or ERA reference standard must be analyzed at the beginning of each analytical run.

Calculations:

$$\text{TDS, mg/L} = \frac{(A-B)}{C} \times 1000000$$

Where A = weight of dish plus residue (g)
B = weight of dish (g)
C = volume of filtered sample used (mL)

Appendix B5

Ammonia Nitrogen

Effective Date 1-29-90

AMMONIA NITROGEN

Scope and Application: This method is applicable to the determination of ammonia-nitrogen in drinking water, surface water, groundwater, sludges, soils, and industrial wastes.

Method: Micro-distillation, Colorimetric

Reference: EPA, 1983, Method 350.2

Detection Limit: 0.10 mg/L for aqueous samples
5.00 mg/kg for soils and sludges

Optimum Range: 0.10 - 2.00 mg/L for aqueous samples
5.00 - 100 mg/kg for soils and sludges

Sample Handling: Acidify aqueous samples with concentrated sulfuric acid to pH <2 and refrigerate at 4°C. Refrigerate soils and sludges at 4°C. Analyze within 28 days of sampling.

Reagents and Apparatus:

1. Kjeldahl flasks, 100 mL
2. Keeney distillation apparatus
3. Spectrophotometer, set at 425nm with sipper cell
4. Erlenmeyer flasks, 50 mL
5. Sulfuric acid, concentrated
6. Milli-Q water
7. pH meter, 0.1 pH unit sensitivity
8. Volumetric glassware, Class A (pipets and flasks)
9. Top loading balance, 0.01g sensitivity
10. Graduated cylinders, 50 mL
11. Mixing cylinders, 50 mL
12. Ammonium chloride (NH_4Cl)
13. Boric acid (H_3BO_3)
14. Mercuric iodide (HgI_2)
15. Potassium iodide (KI)
16. Sodium hydroxide (NaOH)
17. Sodium tetraborate ($Na_2B_4O_7 \cdot 10H_2O$)
18. Sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$)
19. Analytical balance, 0.0001g sensitivity
20. 150 mL beaker
21. Stir bars and stir plate

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated).

1. Ammonium chloride stock solution(1000 mg/L): In a 1 liter volumetric flask, dissolve 3.819g NH_4Cl in approximately 300 mL Milli-Q water and bring to volume. Preserve with H_2SO_4 to a pH<2. Refrigerate.

2. Ammonium chloride standard solution (10 mg/L): Dilute 10.0 mL of the ammonium chloride stock solution to 1 liter with Milli-Q water in a volumetric flask. Preserve with H_2SO_4 to a pH<2. Prepare monthly. Refrigerate.

3. Boric acid solution: Dissolve 20.0g H_3BO_3 in Milli-Q water and dilute to 1 liter in a volumetric flask.

4. Nessler reagent: Dissolve 100g of mercuric iodide and 70g of potassium iodide in about 200 mL of Mill-Q water. Add this mixture slowly, while stirring to a COOLED solution of 160g NaOH in 500 mL Milli-Q water. Dilute the mixture to 1 liter. Store in a Pyrex bottle and keep out of direct sunlight. Refrigerate. NOTE: Commercially available.

5. Sodium hydroxide (1N): Dissolve 40g of NaOH in Milli-Q water and dilute to 1 liter.

6. Sodium hydroxide (0.1N): Dilute 100 mL of 1N NaOH to 1 liter with Milli-Q water.

7. Sodium tetraborate solution (0.025M): Dissolve 9.5g of $Na_2B_4O_7 \cdot 10H_2O$ or 5.0g anhydrous $Na_2B_4O_7$ in Milli-Q water and dilute to 1 liter.

8. Borate buffer: Add 88 mL of 0.1N NaOH solution to 500 mL of 0.025M sodium tetraborate solution. Dilute to 1 liter with Milli-Q water.

9. Sodium thiosulfate (1/70N): Dissolve 3.5g $Na_2S_2O_3 \cdot 5H_2O$ in Milli-Q water and dilute to 1 liter. (1 mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample).

NOTES:

1. Residual chlorine must be removed prior to distillation by pretreating the sample with sodium thiosulfate solution.

2. Pre-steam the distillation apparatus with 10% NaOH before use.

3. Cyanate and some volatile alkaline compounds may cause an off-color nesslerization. This off-color can be eliminated by boiling the sample at a low pH (pH 2-3) to drive off the compound. This should be done prior to the distillation step.

Procedure: Sample must be homogenized prior to analysis to ensure a representative sample aliquot.

Distillation:

1. All glassware is to be soap and water washed, tap water rinsed, and Milli-Q water rinsed prior to use.

2. The reservoir should be 2/3 full with Milli-Q water. Add a few boiling chips. Add sulfuric acid to reservoir to bring to a pH <2. Turn on the heater. Set heater control to HIGH. Allow the steam reservoir to heat up. This unit will take about 45 minutes to heat-up. Turn the heater control to about a setting of 8 and bring to boiling. Analysis can begin once boiling begins.

3. Prepare the distillation apparatus as follows: Steam out the distillation apparatus with a 10% NaOH solution. Turn on water and continue until 40 mL has been distilled.

4. Aqueous samples:

   Place 50 mL or an aliquot of sample diluted to 50 mL in a 150 mL beaker. Record the volume used. Add 1N NaOH while stirring very slowly until the pH is 9.5 ±0.1 using pH meter.

   To spike: Place 50 mL sample and 5 mL of the 10 mg/L ammonia standard into a beaker, adjust pH to 9.5 ±0.1 and continue with procedure. Final spike level is 1.0 mg/L.

   Non-aqueous samples:

   Place approximately 1.0g in a 150 mL beaker. Record weight used. Add 50 mL Milli-Q water and adjust the pH with 1N NaOH, while stirring slowly, to pH 9.5 ±0.1 using pH meter.

   To spike: Place 1.0g sample, 5 mL of the 10 mg/L ammonia standard in the beaker. Add 50 mL Milli-Q water, adjust pH, and continue with procedure.

5. Transfer the pH-adjusted sample to a 100 mL Kjeldahl flask. Add 2.5 mL of borate buffer.

6. Add 5 mL of boric acid to a 50 mL Erlenmeyer flask and place flask at the condenser outlet with the tip of the condenser immersed in the boric acid.

7. Connect the Kjeldahl flask to the distillation apparatus and secure with springs.

8. Close the stopcock on the condensation chamber. Close the drain stopcock. The steam will now pass through the Kjeldahl flask.

9. Steam distill 30-40 mL at a rate of 4-5 mL/min.

10. Rinse tip of condenser into erlenmeyer flask, remove the erlenmeyer flask.

11. Rinse the tip of the condenser and steam outlet into a waste beaker.

12. Continue distilling remaining samples, blanks and standards. When all samples, blanks and standards are distilled, the colorimetric determination can be performed.

Colorimetric Determination:

1. Prepare the following series of blanks and standards in 50 mL mixing cylinders containing 5 mLs of boric acid solution (These do not need to be taken through the distillation step).

mL of 10 mg/L ammonium chloride solution	Dilute to	Concentration (mg/L)
0	50 mL	BLANK
0.5	50 mL	0.10
1.0	50 mL	0.20
2.0	50 mL	0.40
5.0	50 mL	1.00
10.0	50 mL	2.00

2. Add 2.0 mL of Nessler reagent to the blank and standards. Stopper and mix by inverting several times.

3. After 20 minutes, read the absorbances on the spectrophotometer set at 425nm using the sipper cell. Zero the spectrophotometer to the reagent blank.

4. Transfer distilled samples to 50 mL mixing cylinders and dilute to 50 mL with Milli-Q water. Mix.

5. Determine the ammonia in the distillate as follows:

   - Transfer 25 mL of distillate, or an aliquot diluted to 25 mL, to a mixing cylinder.
   - Add 1 mL of Nessler reagent and mix by inverting several times.
   - After 20 minutes, read the absorbance as described in Step 3.

Calculations:

1. Aqueous Samples:

   a. Calculate using linear regression.

   b. Multiply in any dilution factors performed in the distillation and colorimetric steps to obtain the final result in mg/L.

2. Non-Aqueous Samples:

   a. Calculate using regression to obtain a mg/L value.

   b. Multiply in any dilution factor performed in the colorimetric step (mg/L).

   c. Multiply result obtained from "Step b" by 50 and divide by grams of sample used to obtain the final result in mg/kg.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standards (1.00 mg/L) in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, spectrometer bulb, solutions, etc.).

2. A distilled blank, standard (1.00 mg/L), and known reference standard are to be analyzed at the beginning of the analytical run. The standards must be within acceptable ranges and the blank less then the detection limit, or troubleshooting must be performed.

3. A quality control calibration standard of 1.00 mg/L and a blank are to be analyzed, initially and after every 10 samples. This standard does not need be carried through the distillation procedure. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (± 10% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

Appendix B6

Nitrate & Nitrite Nitrogen

Effective: 5-22-91

NITRATE + NITRITE, NITROGEN - AUTOANALYZER

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

Reference: EPA 1983, Method 353.2
Lachat Instruments, 1989 Method 10-107-04-1-C

Detection Limit: 0.02 mg/L

Optimum Range: 0.02 - 2.00 mg/L $NO_3 + NO_2$ - N

Sample Handling: Preserve with sulfuric acid to pH <2 and refrigerate at 4°C. Analyze within 14 days. Alternatively, unpreserved samples, kept at 4°C can be analyzed within 48 hours of sampling.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 50 seconds
3. Load period: 20 seconds
4. Inject period: 20 seconds
5. Inject to start of peak period: 22 seconds
6. Inject to end of peak period: 68 seconds
7. Gain: 450
8. Zero: 400
9. Interface filter: 520 nm
10. Sample loop: 17 cm
11. Standards for curve set-up: 0, 0.20, 0.50, 1.00, 2.00 mg/L
12. Column: cadmium reduction

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassed Milli-Q water (2 options):**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in a cubitainer, or

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water. Store in cubitainer. (15 min/20 L)

2. **Stock nitrate standard (100 mg/L NO_3):** In a 1 liter volumetric flask, dissolve 0.7218 potassium nitrate (KNO_3) in about 600 mL of Milli-Q water. Add 2 mL of concentrated H_2SO_4 as a preservative. Dilute to the mark. Store in a dark glass bottle. Refrigerate.

3. **Working stock nitrate standard (10 mg/L NO_3):** Add 50 mLs D.I. water to a 100 mL volumetric flask. Add 0.2 mLs concentrated H_2SO_4 and pipet 10.0 mL of the stock nitrate standard. Dilute to the mark with DI water. Prepare fresh every 2 weeks. Refrigerate.

4. **Standards:** (Prepare fresh every 2 weeks.) Preserve with 0.2 mL H_2SO_4. Dilute to volume with D.I. water. Refrigerate.

	Concentration of Standard	Letter Identifier	Volume of NO_3 Standard	Dilute to
	0 mg/L	A	0	100 mL
	0.20 mg/L	B	2.0	100 mL
	0.50 mg/L	C	5.0	100 mL
	1.00 mg/L	D	10	100 mL
	2.00 mg/L	E	20	100 mL
CRDL	0.02 mg/L		2.0	1000 mL

Note: Computer refers to standards by letter.

5. **Sodium hydroxide (15M):** To 250 mL of D.I. water, add 150.0g NaOH. **Slowly!** This solution will get very HOT! Swirl to dissolve. Store in a plastic bottle.

6. **Ammonium chloride buffer solution:** In a 1 liter volumetric flask, dissolve 85.0g of ammonium chloride (NH_4Cl)* and 1.0g of disodium ethylenediamine tetracetate dihydrate (EDTA) in approximately 800 mL D.I. water. Adjust the pH to 8.5 with 15M NaOH (approximately 8 mL). Dilute to the mark and filter through a 0.45 um filter.

   *** See Notes #5.**

7. **Sulfanilamide color reagent:** In a 1 liter volumetric flask, add approximately 800 mL of Milli-Q water. Then add 100 mL concentrated phosphoric acid (H_3PO_4). Add 40.0g sulfanilamide and dissolve completely. Dissolve 1.0g N-1-naphthlethylenediamine dihydrochloride (NED) and dilute to one liter. Store in **dark bottle** at 4°C. Stable for **2 months** when refrigerated.

8. **Column Preparation:**

   a. **Cadmium preparation:** Place 10-20g of coarse cadmium powder (granules) in a 250 mL beaker and wash with 50 mL of acetone, then distilled water, then two 50 mL portions of 1 M hydrochloric acid (8 mL concentrated hydrochloric acid plus 92 mL deionized water). Then rinse thoroughly with deionized water. If using cadmium for second time, rinse with 1 M hydrochloric acid before beginning process. **CAUTION: Collect and store all waste cadmium. Wear gloves!**

   b. **Copperization:** Prepare a 2% copper sulfate solution (20g of $CuSO_4 \cdot 5H_2O$) per liter of deionized water) and add a 100 mL portion to the cadmium prepared in "a" above. Swirl gently for about 5 minutes, then decant the liquid and repeat with a fresh 100 mL portion of 2% copper sulfate. Continue this process until colloidal copper is visible in the supernatant (a red-brown precipitate) and solution remains blue in color. Rinse with D.I. water until all colloidal copper is removed from the supernatant. Wash once with ammonium chloride buffer. The cadmium should be black or dark gray. The cadmium granules may be stored in a stoppered bottle in ammonium chloride buffer.

c. **Packing the column (wear gloves!):** Place a small piece of polyurethane foam (or glass wool) loosely in the end of the glass tube. Insert the plugged end of the glass tube into the column end fitting. Cut a length of 0.032" id teflon tubing 3 to 4 inches longer than the column.

Insert the teflon tube in the end fitting and fill the whole tube with water, holding the flexible tube in a U-shape so that the ends are level. Place the second end fitting on the other end of the teflon tubing. (Placing a small funnel onto the end fitting may aid filling.) Taking care that no air bubbles are introduced, place the copperized cadmium granules in the column. Tap the column gently, every 1-2 cm, to pack the granules. When the column is packed to within about 5 mm of the end of the glass column, insert another foam plug, then the column end fitting. Store the column with the ends connected with a length of teflon tubing, as air pockets or having the column dry out will necessitate repacking. If air remains in the column, connect the column to the manifold and turn the pump on maximum. Tap column firmly until all air is removed.

d. **Column activation:** The column must be activated before use or it will not reduce nitrate. This may be accomplished by pumping the 10 mg/L nitrate standard through the sample line. When the solution is injected, a brilliant pink color will be visible in the coil. The cadmium column efficiency should be above 80%, if less, the column must be repacked.

To check column efficiency, standardize with nitrite standards and then analyze all nitrate standards. The recovery of the nitrate standards should be consistent at all standard concentrations.

Notes:

1. **Interferences:**

   - Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample must be pre-filtered.
   - Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate these interferences.
   - Samples that contain large concentrations of oil and grease will coat the surface of the column. This interference is eliminated by pre-extracting the sample with an organic solvent.

2. Samples must be diluted to obtain concentrations within the optimum working range.

3. The gain and zero settings are guidelines and must be optimized each day.

4. Color will interfere: dilute the sample and also spike the dilution to confirm the quality of the result. Record on data sheet.

5. ACS grade ammonium chloride has been found occasionally to contain significant nitrate contamination, so an alternative preparation for the ammonium chloride buffer (Reagent #6) is as follows:

   In the hood, add 126 mL concentrated HCl to a 1 liter volumetric flask containing 500 mL degassed Milli-Q water. Mix. Add 95 mL ammonium hydroxide and 1.0 gm disodium EDTA. Dissolve and dilute to the mark. The pH should be 8.5 ± 0.1, adjust pH if necessary.

System Operation:

1. Refer to Auto Analyzer Operation - Start-up Procedure (IOP# LAAC-Section A).
2. After pumping reagents through the lines, turn off the pump and insert column, making sure that air bubbles are not introduced into the column.
3. Activate column if necessary. (See #8d. above.)
4. Analyze an initial calibration check standard, a blank, a CRDL standard and a known reference standard at the beginning of each run. The blank must be below the detection limit and standards must be within required control limits before any samples are analyzed.
5. To spike samples, mix equal volumes of sample and 1.00 mg/L standard (D) for a final spike level of 0.50 mg/L.
6. The calibration check standard is 1.00 mg/L NO_3 (D).
7. If only nitrate is requested, nitrites must be analyzed and subtracted from the nitrate + nitrite value.
8. After use, turn off the pump and remove the column from the manifold.
9. Refer to Auto-Analyzer Shut-down Procedure. (IOP# LAAC-Section B.)

Quality Control:

1. Establish a standard curve with the standards listed above. The derived concentrations for each calibration standard must be within 10% of the true value. The derived value for the blank must be below the method detection limit.
2. The CRDL standard concentration should be at the method detection limit (MDL). Results must be within the acceptable limits of ± the MDL of the instrument must be recalibrated before samples are analyzed.
3. A quality control calibration check standard of 1.00 mg/L (D) and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. Standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately.

Calculation:

1. Calculate with Lachat Quikchem software, in the concentration mode, using the IBM XT computer.

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

    a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

    b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

    c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

    d. Select "Enter Sample ID's". Press <enter>.

    e. Type in sample information. Check standards will automatically be placed in the tray information portion.

    f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

    After calibration is complete:

    - go into the foreground (Press Alt, Esc keys)
    - select "display calibration graph" (Press <enter>)
    - review the data
    - return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.
- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray <u>before</u> pressing "R"!

B. <u>Instrument Shut-Down</u>

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.
2. If column is used, stop the pump and disconnect from manifold.
3. Pull lines from reagents into a wash beaker of D.I..
4. Pump D.I. through lines for 2-5 minutes.
5. Pump air through lines until manifold is dry.
6. Turn off pump.
7. Release tubing cartridges and lower tension levers. Release tubing.
8. Turn off main switch on rear power strip.
9. Empty and rinse waste containers, if necessary.
10. Perform back-up on current data files, once a week. (See Section C)
11. Turn off the computer and printer.

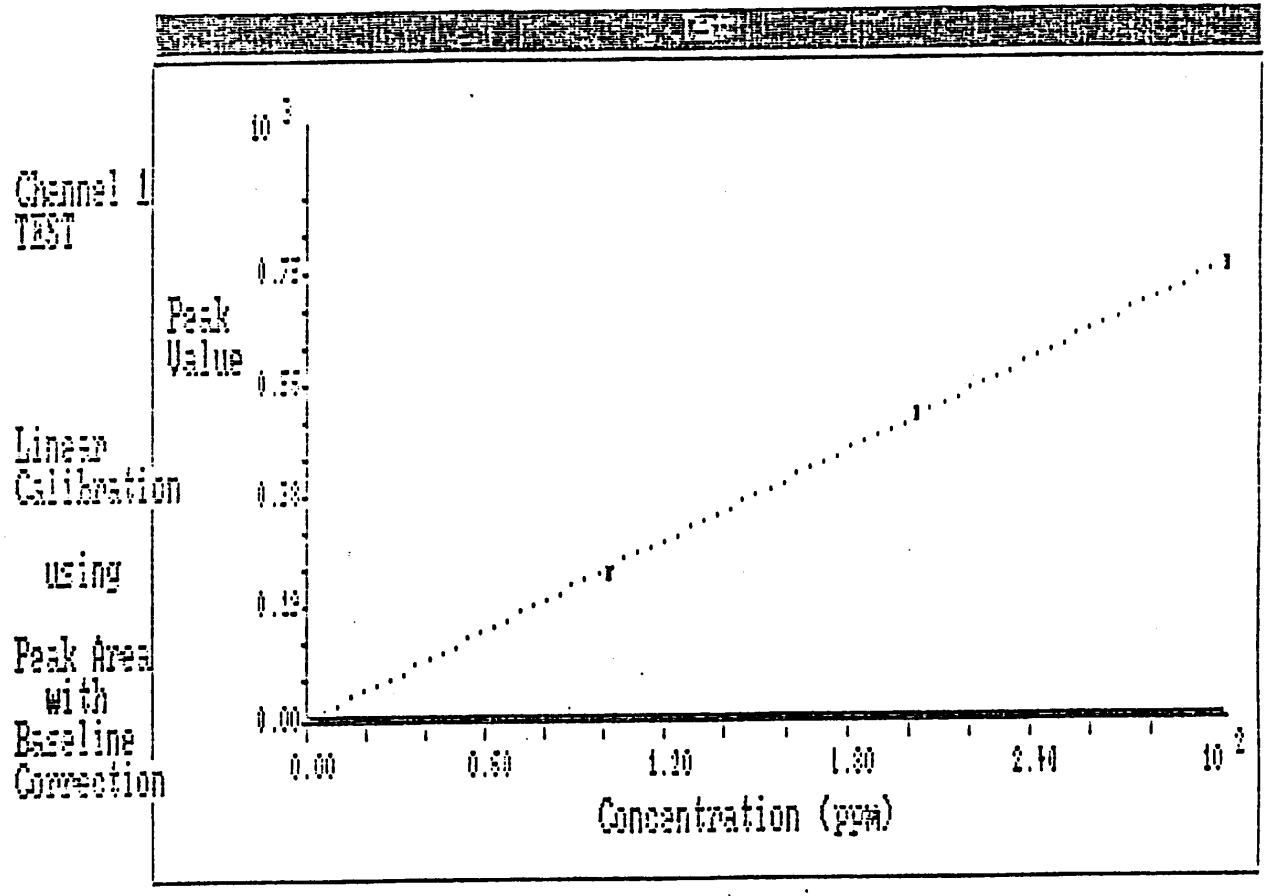
C. <u>Backing-up the Data Files</u>

1. Exit to DOS
2. At C> Type: cd\fialab\data Press <enter>
3. At C> Type: copy *.rpt a: Press <enter>
   After everything is copied - remove disc.
4. At C> Type: del *.* Press <enter>
5. Are you sure (Y/N)? Type: Y Press <enter>
6. At C> Type: cd\ Press <enter>
7. Turn off the red switch on the computer power strip to turn off the computer, printer and screen.

D. Quikchem Calibration

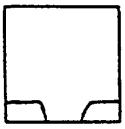
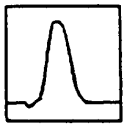
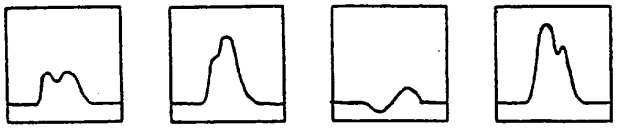
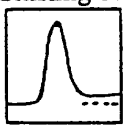
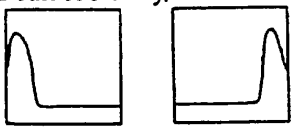
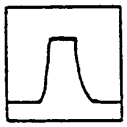
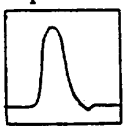
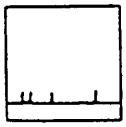
QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

FIGURE 1



Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

LACHAT TROUBLESHOOTING GUIDE

Problem:	Cause:
Negative peak 	1. Contaminated carrier 2. Bad or no SO_4 column or sample extremely high in hardness 3. Samples high in oxidizing agents
Reproduceable dip when valve switches (before peak) 	1. Bad color reagent 2. Could also be valve turning artifact caused by a highly colored reagent
Odd looking peaks 	1. Index of refraction problem, matrix related (usually acid or pH buffering) 2. Also method interferences: high hardness on SO_4 method, oxidizing samples on nitrate method 3. Bad reagents
Shifting baseline 	1. Flow problem 2. Precipitate build-up on SO_4 manifold tubing
Peak too early/late 	1. Usually flow problem 2. Valve not set to an initial load position state before starting run 3. Incorrect pump setting or other timing problem
Peak cut off **in** window 	1. Reagents exceeded-reagents improperly prepared 2. Standards incorrectly prepared
Reproduceable dip after peak 	1. Bad column
Small intermittent peaks 	1. Milli-Q or reagents not degassed properly or adequately 2. Waste line coil not installed (for the methods which require it)

Three basic areas of troubleshooting:
1. Fluidics (clogs, old pump tubing, crimp in manifold tubing)
2. Chemistry
3. Timing (not usually a problem after initial development)

BLH/rff
[rff-genpol-604]

Appendix B7

Total Phenolics

Effective Date: 6-26-91

TOTAL PHENOLICS - DISTILLATION

Scope and Application: This method is applicable to the analysis of drinking water, surface water, domestic and industrial wastewater and solids.

Method: Automated 4AAP with distillation

Reference: EPA 1983, Method 420.1

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.5 mg/L

Sample Handling: Aqueous samples are to be collected in a one liter glass container with a teflon-lined cap. Preserve to pH < 2 with sulfuric acid and refrigerate at 4°C. Non-aqueous samples are to be collected in a glass container and refrigerated at 4°C. Samples should be analyzed within 28 days of collection.

Reagents and Apparatus:

1. Phenol
2. Concentrated sulfuric acid (H_2SO_4)
3. Copper sulfate ($CuSO_4$)
4. Sodium hydroxide (NaOH)
5. Glass distillation appartus
6. One liter distillation flasks
7. 250 mL volumetric flasks
8. D.I. water
9. NoChromix or similar cleaning solution
10. 400 mL beakers
11. pH meter (glass-body electrode), 0.1 pH sensitivity
12. 0.45 um filters
13. Top loading balance, 0.01 g sensitivity
14. Analytical balance, 0.0001 g sensitivity

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Stock phenol solution (1000 mg/L):** Dissolve 1.000 g phenol in freshly boiled and cooled D.I. water in a volumetric flask and dilute to 1 liter. Preserve with 2 mL of concentrated H_2SO_4 per liter.

2. **Working phenol solution A (10 mg/L):** Dilute 10 mL of stock phenol solution to 1 liter with D.I. water in a volumetric flask. **(Prepare fresh daily.)** Preserve with 2 mL concentrated H_2SO_4 per liter.

3. **Copper sulfate solution:** Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in D.I. water and dilute to 1 liter in a volumetric flask.

4. **NaOH (1N):** Dissolve 40 g NaOH in D.I. water in a 1 liter volumetric flask, dilute to the mark.

5. **NaOH (6N):** Dissolve 240 g NaOH in D.I. water in a 1 liter volumetric flask, dilute to the mark.

6. **H_2SO_4 (1N):** Dilute 28 mL of concentrated H_2SO_4 with 500 mL D.I. water in a 1 liter volumetric flask, and dilute to the mark.

Notes:

1. **Interferences:**

   a. Oxidizing agents such as chlorine interfere. Add excess ferrous ammonium sulfate immediately after sampling to remove interference. If chlorine is not removed, the phenols may be partially oxidized and results will be low.

   b. Sulfur compounds interfere. Interferences are eliminated by acidifying the sample to a pH of <4 with H_3PO_4 or H_2SO_4 and aerating briefly by stirring and adding $CuSO_4$.

2. If the solutions do not remain a blue to green color after the addition of the $CuSO_4$, or lose the color upon heating, additional $CuSO_4$ is required. Record the additional amount of $CuSO_4$ required on the data sheet.

3. Rinse condensers with D.I. water between distillations to prevent contamination.

Procedure:

1. All glassware is to be soap and water washed, soaked in NoChromix followed by tap rinse and D.I. water rinse prior to analysis. After washing, but before tap and D.I. rinse, distillation flasks should be soaked in NoChromix or or similar cleaning solution for a minimum of 15 minutes.

2. **For aqueous samples:** Using a 250 mL graduated cylinder, place 250 mL of preserved sample into a beaker.

   For solid samples: Weigh out 1.0 g using the analytical balance and place in a beaker. Record weight. Add 250 mL D.I. water.

   To Spike samples: Pipet 2.5 mL of 10 mg/L working phenol solution A into 250 mL of sample. This corresponds to a 0.100 mg/L spike.

3. Using a glass bodied pH electrode, adjust the sample to pH of 4.0 $\pm$ 0.1 using 1N H_2SO_4 and 1N or 6N NaOH. Add 5 mL $CuSO_4$ solution. Transfer to distillation flask and add several boiling chips.

4. Set flask on mantle, connect to condenser, and place a labeled 250 mL volumetric flask under tip of condenser. Turn the mantle to medium-high heat.

5. Distill at least 200 mL of sample. Stop the distillation, and when boiling ceases, add about 50 mL of **warm** D.I. water to the flask (**warm water is required to prevent the flask from cracking**). Resume distillation until 250 mL have been collected.

6. If the distillate is turbid, filter through a 0.45 micron membrane filter.

7. Distillates are ready for analysis. Proceed with Lachat SOP# PhenAAC for automated color development.

Quality Control:

1. The calibration standards do not need to be carried through the distillation procedure.

2. An EPA or ERA reference standard is to be distilled with each batch of samples.

3. A reagent blank and standard of 0.100 mg/L are to be distilled with each batch of samples as a check for contamination and efficiency of distillation.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data is to be flagged appropriately.

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

2. To calculate the spike:

$$\% \text{ Recovery} = \frac{\text{Spike conc.} - \text{Sample conc.}}{0.100} \times \frac{\text{mL of Sample}}{250} \times 100$$

Effective: 11-22-91

TOTAL PHENOLICS - AUTOANALYZER

Scope and Application: This method is applicable to **distilled** groundwater, drinking water, wastewater, sediments and soils. All samples must be distilled prior to analysis with the autoanalyzer.

Reference: EPA, 1983, Method 420.2.
Lachat Instruments, 1989, Method 10-210-00-1-A
Standard Methods, 16th Edition, pages 556-561

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.500 mg/L

Sample Handling: Samples should be capped and refrigerated at 4°C after distillation. Distillates should be analyzed within 7 days of distillation and within 28 days of sample collection.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 40 seconds
3. Load period: 20 seconds
4. Inject period: 20 seconds
5. Inject to start of peak period: 10 seconds
6. Inject to end of peak period: 50 seconds
7. Gain: 200x10 (use 10x gain)
8. Zero: 350
9. Interference filter: 500 nm
10. Sample loop: 120 cm
11. Standards for standard curve: 0, 0.050, 0.100, 0.200, 0.500 mg/L

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Degassed Milli-Q water - 2 options:**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water. (15 min/20 L.) Store in cubitainer.

2. **4-aminoantipyrine color reagent:** In a 250 mL volumetric flask, dissolve 0.16 g of 4-aminoantipyrine ($CH_3NN(C_6H_5)COC(HN_2)\cdot CCH_3$), in approximately 150 mL of degassed Milli-Q water and dilute to 250 mL. Prepare fresh daily.

3. **1M NaOH:** In a 1L volumetric flask, dissolve 40.0 g NaOH in 900 mL MIlli-Q water. Dilute to mark and invert several times. Store in a plastic bottle.

4. **Buffered potassium ferricyanide (pH 10.3):** In a 250 Liter volumetric flask, dissolve 0.5000 g potassium ferricyanide ($K_3Fe(CN)_6$), 0.7750 g boric acid (H_3BO_3) and 0.9375 g potassium chloride (KCl) in 100 mL of D.I. water. Add 12 mL of 1 M sodium hydroxide and dilute to the mark. Filter through a 0.45 micron membrane filter before each use. Prepare fresh weekly. Refrigerate.

5. **Stock phenol (1000 mg/L):** In a 1 liter volumetric flask, dissolve 1.00 g of phenol in 500 mL of Milli-Q water. Add 2 mL concentrated H2SO4 as a preservative. Dilute to 1000 mL. (1.0 mL = 1.0 mg phenol). Refrigerate.

6. **Working stock standard (10.0 mg/L):** In a 500 mL volumetric flask, pipet 5.0 mL of the stock phenol and 1 mL H_2SO_4, dilute to the mark with Milli Q water. (Prepare fresh daily!).

7. **Standards:** (Prepare fresh daily!)

Concentration of Standard	Letter Identifier	Volume of 10 mg/L working standard	Dilute to
0.000 mg/L	A	0.0	100 mL
0.050 mg/L	B	0.5	100 mL
0.100 mg/L	C	1.0	100 mL
0.200 mg/L	D	2.0	100 mL
0.500 mg/L	E	5.0	100 mL

Note: Computer refers to standard by letter.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero are guidelines, they must be optimized each day.

3. **Interferences:**

   - Interferences are eliminated or reduced to a minimum by the distillation procedure.

   - Color will interfere, dilute the sample and spike the dilution to confirm the quality of the result.

   - Turbidity will interfere. If present, filter through a 0.45 micron membrane filter prior to analysis on Lachat System. Record on data sheet.

   - A peak with a shoulder indicates an interference. If this occurs, dilute the sample. If the problem still exists, redistill the sample and its spike at a dilution. Record on data sheet.

4. If a sample has a value of 0.015 or less, it must be re-read to confirm the positive hit.

System Operation:

1. Refer to "Auto Analyzer Operation Start-up Procedure". (IOP# LAAC-Section A)

2. Analyze an initial calibration check standard, a blank, a distilled known reference standard, a distilled standard and a distilled blank at the beginning of each run. The blanks must be below the detection limit and the standards must be within required control limits before any samples are analyzed.

3. Spikes are distilled at a level of 0.100 mg/L.

4. The calibration check standard is 0.100 mg/L (C).

5. The distilled standard is 0.100 mg/L.

6. If a sample and spike are **overrange:**

   a. Dilute the sample and spike if dilution ≤ 1:5. The distilled spike should be detectable.
   b. Dilute the sample, spike and analyze a manual spike if dilution > 1:5.

7. Refer to Auto Analyzer shut-down procedure. (IOP# LAAC-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. The derived concentration for each calibration standard must be within 10% of the true value. The derived value for the blank must be below the method detection limit.

2. A distilled standard of 0.100 mg/L and a distilled blank are analyzed with each set of samples. The distilled standard must be within acceptable ranges and blank must be below method detection limit before any samples are analyzed.

3. A quality control calibration check standard of 0.100 mg/L (C) and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. Standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data is to be flagged appropriately.

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

d. Select "Enter Sample ID's". Press <enter>.

e. Type in sample information. Check standards will automatically be placed in the tray information portion.

f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

After calibration is complete:

- go into the foreground (Press Alt, Esc keys)
- select "display calibration graph" (Press <enter>)
- review the data
- return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.
- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray <u>before</u> pressing "R"!

B. <u>Instrument Shut-Down</u>

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.
2. If column is used, stop the pump and disconnect from manifold.
3. Pull lines from reagents into a wash beaker of D.I..
4. Pump D.I. through lines for 2-5 minutes.
5. Pump air through lines until manifold is dry.
6. Turn off pump.
7. Release tubing cartridges and lower tension levers. Release tubing.
8. Turn off main switch on rear power strip.
9. Empty and rinse waste containers, if necessary.
10. Perform back-up on current data files, once a week. (See Section C)
11. Turn off the computer and printer.

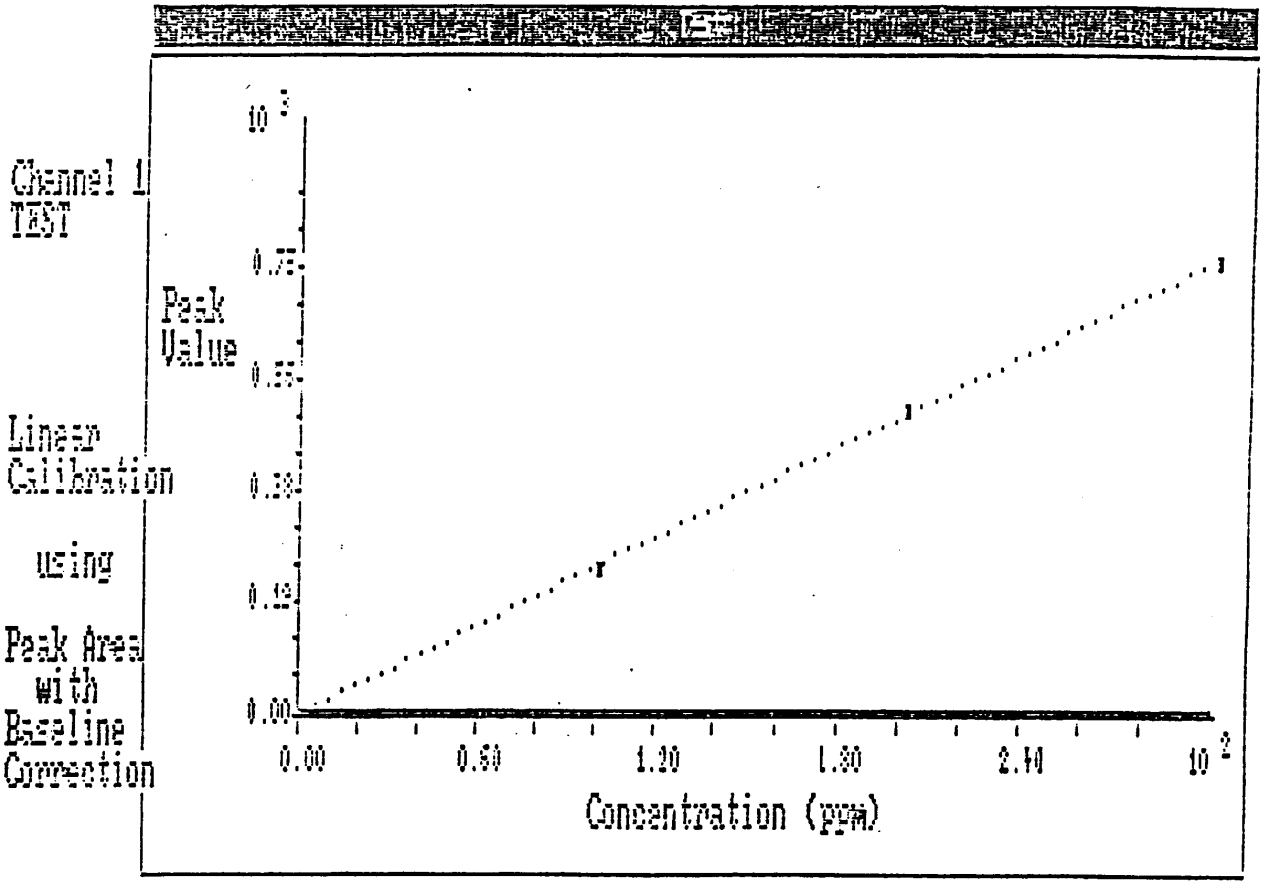
C. <u>Backing-up the Data Files</u>

1. Exit to DOS
2. At C> Type: cd\fialab\data Press <enter>
3. At C> Type: copy *.rpt a: Press <enter>
   After everything is copied - remove disc.
4. At C> Type: del *.* Press <enter>
5. Are you sure (Y/N)? Type: Y Press <enter>
6. At C> Type: cd\ Press <enter>
7. Turn off the red switch on the computer power strip to turn off the computer, printer and screen.

D. Quikchem Calibration

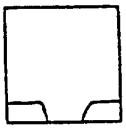
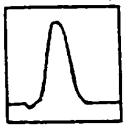
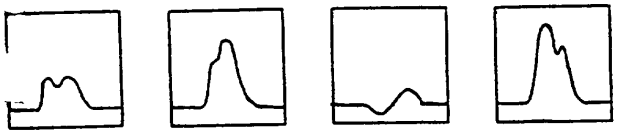
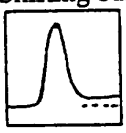
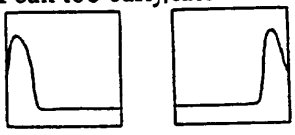
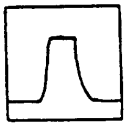
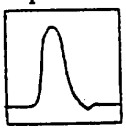
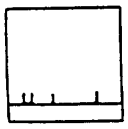
QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

FIGURE 1



Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

LACHAT TROUBLESHOOTING GUIDE

Problem:	Cause:
Negative peak 	1. Contaminated carrier 2. Bad or no SO_4 column or sample extremely high in hardness 3. Samples high in oxidizing agents
Reproduceable dip when valve switches (before peak) 	1. Bad color reagent 2. Could also be valve turning artifact caused by a highly colored reagent
Odd looking peaks 	1. Index of refraction problem, matrix related (usually acid or pH buffering) 2. Also method interferences: high hardness on SO_4 method, oxidizing samples on nitrate method 3. Bad reagents
Shifting baseline 	1. Flow problem 2. Precipitate build-up on SO_4 manifold tubing
Peak too early/late 	1. Usually flow problem 2. Valve not set to an initial load position state before starting run 3. Incorrect pump setting or other timing problem
Peak cut off in window 	1. Reagents exceeded-reagents improperly prepared 2. Standards incorrectly prepared
Reproduceable dip after peak 	1. Bad column
Small intermittent peaks 	1. Milli-Q or reagents not degassed properly or adequately 2. Waste line coil not installed (for the methods which require it)

Three basic areas of troubleshooting:
1. Fluidics (clogs, old pump tubing, crimp in manifold tubing)
2. Chemistry
3. Timing (not usually a problem after initial development)

BLH/rff
[rff-genpol-604]

Appendix B8

Grain Size Distribution

Designation: D 422 – 63 (Reapproved 1990)$^{\epsilon 1}$

Standard Test 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.

$^{\epsilon 1}$ NOTE—Section 19 was added editorially in September 1990.

1. Scope

1.1 This test 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. Referenced 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 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

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

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

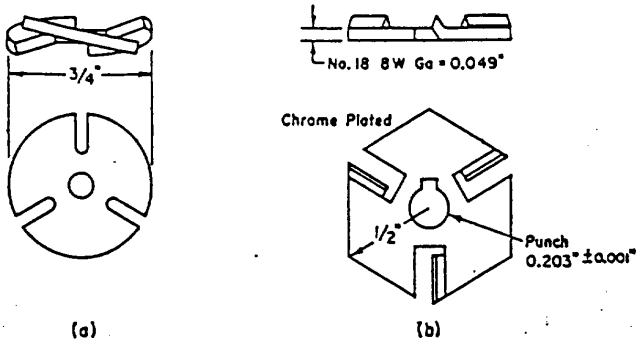
Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

[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 14.03.

[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.



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

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)	

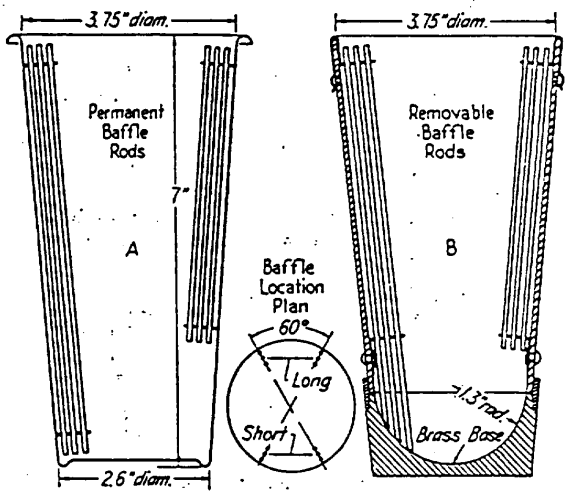
NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

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.



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

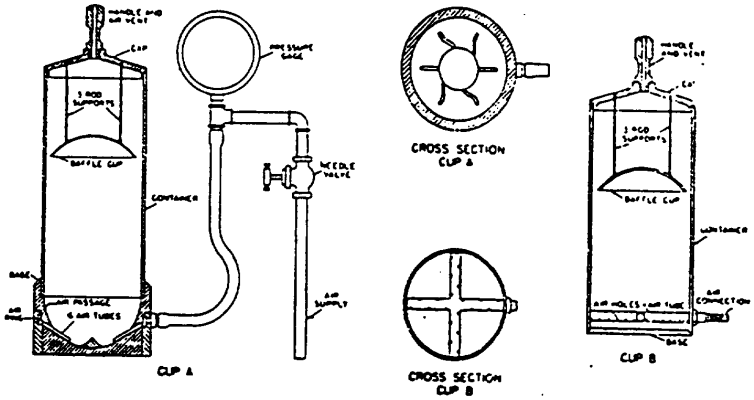


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

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 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.

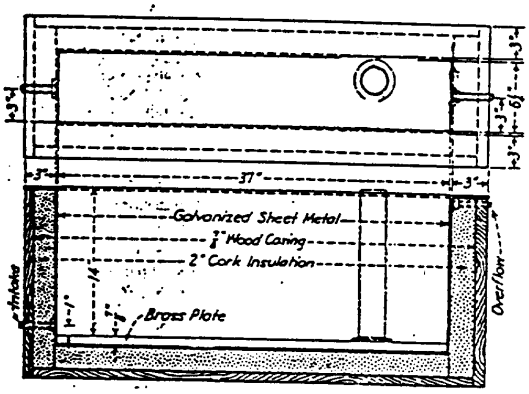


FIG. 4 Insulated Water Bath

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 hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns.

Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, 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.

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.

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. According to Stokes' law:

$$D = \sqrt{[30\eta/980(G - G_1)] \times L/T}$$

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_l = 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.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm^3, and

A = cross-sectional area of sedimentation cylinder, cm^2.

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm^3

A = 27.8 cm^2

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

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

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

arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(3) Silt size, 0.074 to 0.005 mm ..... %
(4) Clay size, smaller than 0.005 mm ..... %
  Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-μm)	.....
No. 200 (75-μm)	.....
HYDROMETER ANALYSIS	
0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

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Designation: D 1140 - 54 (Reapproved 1990)[ε1]

An American National Standard

Standard Test Method for Amount of Material in Soils Finer Than the No. 200 (75-μm) Sieve[1]

This standard is issued under the fixed designation D 1140; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

[ε1] NOTE—Editorial changes were made throughout in September 1990.

1. Scope

1.1 This test method covers determination of the total amount of material in soils finer than the No. 200 (75-μm) sieve.

2. Referenced Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]

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

3. Apparatus

3.1 *Sieves*—A nest of two sieves, the lower being a No. 200 (75-μm) sieve and the upper a No. 40 (425-μm) sieve, both conforming to ASTM Specification E 11.

3.2 *Containers*—A pan or vessel of sufficient size to contain the test sample covered with water and to permit vigorous agitation without advertent loss of any part of the sample, and a second pan or container for use in drying the test sample after washing.

4. Test Sample

4.1 The test sample shall be selected from material that has been thoroughly mixed. A representative sample, sufficient to yield not less than the approximate weight of dried material shown in the following table, shall be selected using a sample splitter or by the method of quartering:

Nominal Diameter of Largest Particle, in.	Approximate Minimum Weight of Sample, g
0.0787 (No. 10 sieve) (2.0 mm)	200
0.187 (No. 4 sieve) (4.75 mm)	500
¾ (19.0 mm)	1500
1 (25.0 mm)	2000
1½ or over (37.5 mm)	2500

5. Procedure

5.1 Dry the test sample to a constant weight at a temperature not exceeding 230 ± 9 F (110 ± 5 C) and weigh to the nearest 0.05 percent, or alternatively, weigh the test sample moist and use an auxiliary moisture content sample to determine the moisture content of the sample. The weight of the moisture content sample shall be between 20 and 30 percent of the weight of the test sample. Calculate the oven-dry weight of the test sample from the moist weight and the moisture content.

5.2 Place the test sample in the container, add sufficient clean water to cover it, and allow to soak a minimum of 2 h (preferably overnight).

5.3 Agitate the contents of the container vigorously and pour the wash water immediately over the nested sieves, arranged with the coarser sieve on top. Repeat the process of adding clear water to the container to cover the sample, agitating the contents of the container, and pouring the wash water over the nested sieves until the wash water is clear. When the total sample is small, the entire contents of the soaking container may be transferred to the nested sieves after the first washing and the washing operation completed in accordance with 5.4. The wash water need not be saved.

NOTE 1—The percentage value secured at the end of the test may not be correct (being too low) for soils containing relatively high percentages of the minus 200 fraction. This appears to be due chiefly to inadequate agitation. When it is desired to secure the exact percentage for the minus 200 fraction for such a soil, the portion of the sample passing the No. 40 sieve and retained on the No. 200 sieve secured in the washing operation, shall be transferred to the dispersion cup of the stirring apparatus used in Method D 422. The cup filled half full with water and the contents agitated for 1 min. After this agitation the contents of the cup shall be transferred to the nested sieves and washing continued.

If the stirring apparatus has not been used prior to the drying of the portion of the sample larger than the No. 200 (75-μm) sieve, and it is desired to do so after drying, the dried material shall be separated on the No. 40 (425-μm) sieve; the portion retained shall be saved; and the portion passing shall be placed in the dispersion cup with water and agitated for 1 min with the stirring apparatus as previously described. The contents of the cup shall be transferred to the No. 200 sieve, washed, and dried. The revised total weight retained on the No. 200 sieve shall be secured by combining and weighing the two fractions.

5.4 Transfer the sample to the nested sieves and wash with running water (Note 2). When the sample is larger than can be handled at one time on the nested sieves, wash a portion of the sample and transfer to the container in which it is to be dried.

NOTE 2—Tapping of sieves has been found to expedite the washing operations.

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

Current edition approved Sept. 15, 1954. Originally issued 1950. Replaces D 1140 - 50 T.

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

[3] *Annual Book of ASTM Standards*, Vols 04.01, 04.02, 04.06, 05.05, and 14.02.

5.5 Dry the washed material retained on the nested sieves in a container to a constant weight at a temperature not exceeding 230 ± 9 F (110 ± 5 C) and dry-sieve it on the nested sieves (Note 3). Weigh the dry material retained on the nested sieves to the nearest 0.05 percent.

NOTE 3—Some material passes the No. 200 (75-μm) sieve on dry sieving that did not pass during the washing operation. When desired, a sieve analysis may be made on the portion of the sample retained on the No. 200 sieve, in accordance with Method D 422.

6. Calculation

6.1 Calculate the results as follows:

$$P = [(W_o - W_1)/W_o] \times 100$$

where:

P = percentage of material finer than No. 200 (75-μm) sieve,
W_o = weight of original sample on an oven-dry basis, g and
W_1 = oven-dry weight of sample after washing and dry-sieving, g.

7. Keywords

7.1 grain-size; No. 200 sieve; particle-size; sieve analysis

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WARZYN

SIEVE ANALYSIS ASTM C147-80

COMPUTER CALCULATION SHEET

JOB:

Job No. _____
Date _____
Sheet _____ of _____
Techs _____
Checked By _____

BORING/SAMPLE									
PAN NO.									
START WT. or HYD (No Tare)									
END WT./REMARKS (No Tare)									
1 1/2									
1									
3/4									
1/2									
3/8									
#4									
#8									
#10									
#16									
#30									
#40									
#50									
#80									
#100									
#200									

note: all technicians performing work are required to initial sheet.

WARZYN

SIEVE ANALYSIS

JOB:

SAMPLE NO.

PAN NO.

Report No. _____

Job No. _____

Date _____

Sheet _____ of _____

Start Sample Weight =

End Sample Weight = _____

Weight of P200 Loss =

P200 Loss Weight / Start Sample Weight = % P200 Loss

End Sample Weight =

R #4 Sieve Weight = _____

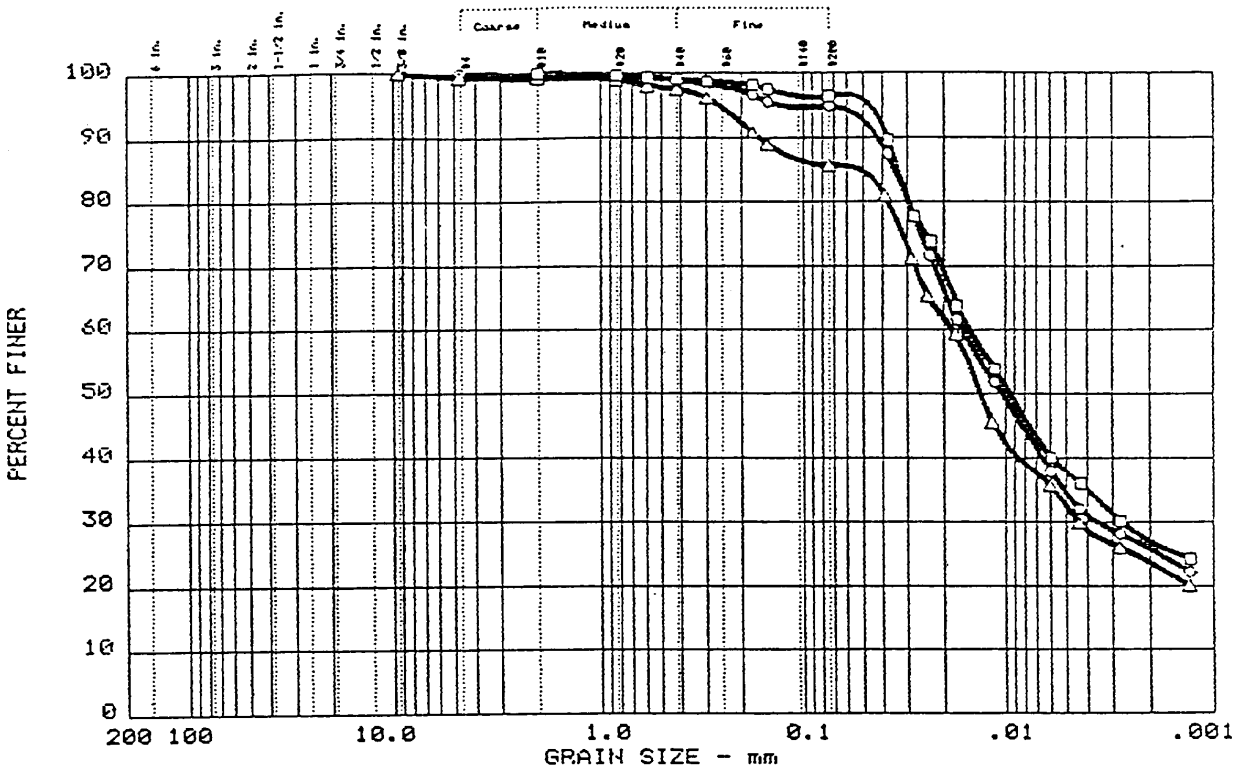
Difference in Weight =

Weight Difference / Start Sample Weight = Mult

Sieve Size	Comm. Wt. Retained	Percent Passing	Multiplier	% P200 Loss in Washing	Percent Passing	_____ Specifications
2 1/2"		X	X	X		
2"		X	X	X		
1 1/2"		X	X	X		
1"		X	X	X		
3/4"		X	X	X		
1/2"		X	X	X		
3/8"		X	X	X		
#4		X	X	X		
Quartered P#4 Wt =						
#4						
#8						
#10						
#16						
#30						
#40						
#50						
#80						
#100						
#200						

DA/mlf
[WEI-15-35]

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	%+3"	% GRAVEL	% SAND	% SILT	% CLAY
○	0.0	0.0	5.2	60.4	34.4
△	0.0	0.6	13.8	53.3	32.4
□	0.0	0.0	3.7	58.5	37.8

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
○	39	19			0.01	0.004				
△	36	16			0.01	0.004				
□	44	21			0.01	0.003				

MATERIAL DESCRIPTION	USCS
○ Brown Lean CLAY, Little Sand	CL
△ Brown Lean CLAY, Some Sand, Trace Gravel	CL
□ Brown Lean CLAY, Trace Sand	CL

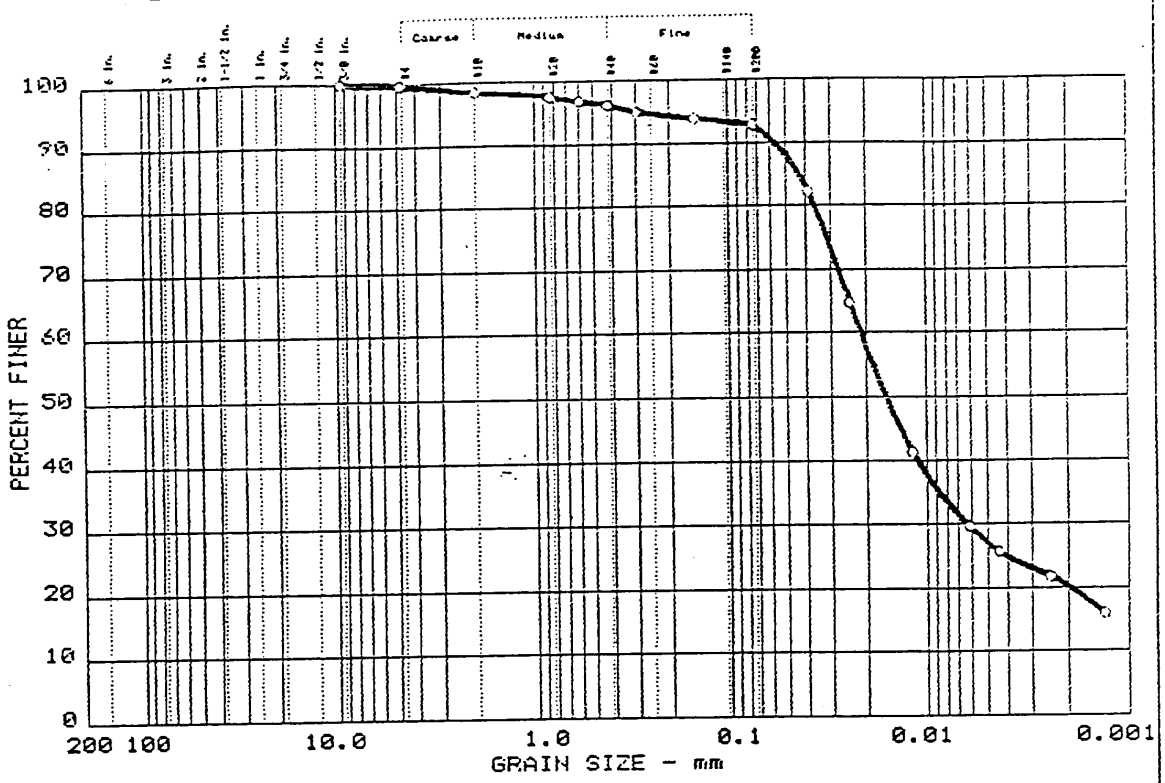
Project No.: 5822.41
Project: Metropolitan Refuse District
○ Sample: TP-1 SAMPLE # 3 @ 1.5-3.0 FT
△ Sample: TP-2 SAMPLE # 3 @ 1.0-2.5 FT
□ Sample: TP-3 SAMPLE # 3 @ 1.0-2.5 FT
Date: 5/14/91

Remarks:
TESTED BY DWA/RP
CHECKED BY DWA
APPROVED BY VJR

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN ENGINEERING INC.

Sheet No.

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	% +3"	% GRAVEL	% SAND	% SILT	% CLAY
○	0.0	0.3	6.7	66.0	27.0

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
○	37	15			0.02	0.006				

MATERIAL DESCRIPTION	USCS
○ Brown Lean CLAY, Little Sand	CL

Project No.: 12153.01
Project: LAND & GAS RECLAMATION, INC LANDFILL
○ Sample: Gramlow Property - Sample # 11

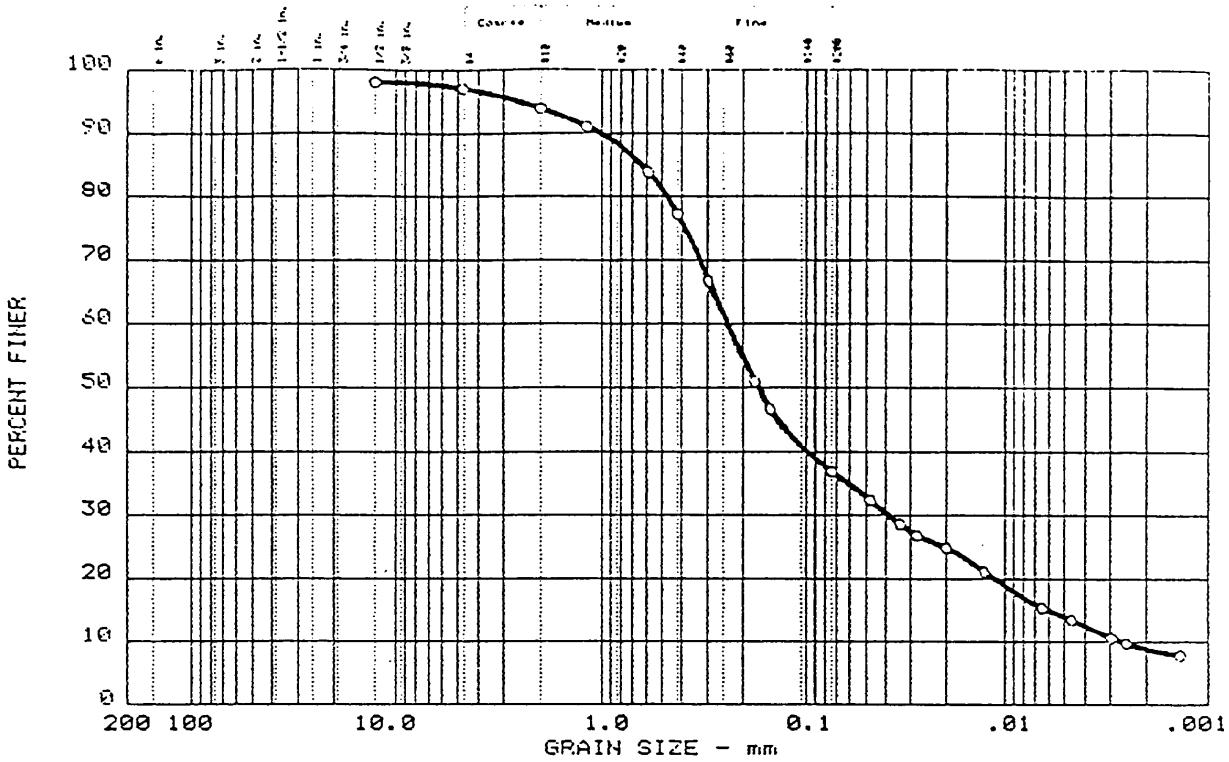
Date: 7/23/91

Remarks:
TESTED BY DWA/RWP
CHECKED BY DWA
APPROVED BY VJR

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN, INC.

Sheet No.

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	%+3"	% GRAVEL	% SAND	% SILT	% CLAY
o	0.0	2.9	60.1	23.0	13.9

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
o	20	9	0.63	0.24	0.17	0.038	0.0060	0.0026	2.37	92.3

MATERIAL DESCRIPTION	USCS
o Brown Fine-Coarse SAND, Some Silt & Clay, Trace Gravel	SC

Project No.: 50422.00
Project: CHAMPION, SARTELL & FERC
o Sample: BORING: 3 SAMPLE: 6C @ 38.5-40.0 FT

Date: 07/14/89

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN ENGINEERING INC.

Remarks:
TESTED BY: MML/DWA
ENTERED BY: MML
CHECKED BY: DWA
APPROVED BY:

Sheet No.

WARZYN

May 6, 1988
99194

PARTICLE-SIZE ANALYSIS OF SOIL

ASTM D 422-63

Procedure to make dispersing agent and calibration:

1. Add 40 grams Sodium Hexametaphosphate to 1000 cc of De-aired Demineralized Water at 20°C. (160gm/4000cc)
2. Add 11.7 grams Sodium Bicarbonate to each 1000cc of agent (brings PH to 8.3) (35 gm/4000cc)
3. Allow to Stand for 24 Hours Before Using. Mix Thoroughly.
4. Mark Stock Solution with Date and Discard at 30 days.
5. Each Soil Sample Receives 125 ml of Stock Solution.
6. Add 125 ml of Stock Solution to a 1000 ml Sedimentation Cylinder and Fill to the 1000 ml mark with Demineralized Water at 20° C.
7. Record Correction Factor with a 152H Hydrometer. Record Temp. (should be 20°).

HYDROMETER CORRECTION

FLUID	SOLIDS ADDED	HYDROMETER READING GM/LITER (152 H Hydrometer)
Cold Tap Water	None	+1
Hot Tap Water	None	0
Hot Tap Water	Deflock Agent	+6
Cold Tap Water	Deflock Agent	+7
Distilled Water	Deflock Agent	+6

99194.00
DWA/mml/DWA
[mml-401-92]

Appendix C

Field Measurement SOPs

Appendix C1

pH

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, otherwise within 24 hours.

Reagents and Apparatus:

1. pH meter
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 10.0.
6. Deionized water

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 7.00 buffer, or electrode storage solution.

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 7.00 buffer.

Calibration:

1. Place combination electrode in fresh pH 7.00 buffer solution.
2. After allowing 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 buffer solution.
4. Wait for reading to stabilize and then turn slope adjustment dial until reading of 4.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00 + 0.05, follow Steps 2-5 again.
6. Rinse electrode with deionized water and place in pH 10.00 buffer. Reading must be in the range of 9.90-10.10 or calibration must be repeated.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.
2. Calibrate meter using calibration procedure.
3. Pour the sample into clean beaker or plastic cup.
4. 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.
5. Check temperature of sample. It should be $\pm$ 2°C of the buffer solutions.
6. Rinse electrode with deionized water.
7. 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.
8. Check calibration with pH 7.00 buffer solution initially and after every 10 samples and at the end of the analytical run and record on data sheet. Buffer solution should read $\pm$0.05 of true value.

Quality Control:

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

Revision Dates

7-22-86

6-28-90

1-8-92

Manager, Operations

Supervisor, Quality Assurance

Kim Johnson

Director, Analytical Services

Appendix C2

Conductivity

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, Method 120.1

Detection Limit: 10 umhos/cm @ 25°C

Sample Handling: Determine on-site or within 28 days

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. conductivity standard, 1000 umhos/cm @ 25°C. Commercially Available

Notes:

1. All conductivity readings must be corrected to 25°C.

Procedure:

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

2. With mode switch of the meter in the OFF position. Check the zero setting. If not at zero, use meter adjusting screw to zero (on front of the meter).

3. Plug probe into jack located on side of meter.

4. 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.

5. Analyze the conductivity standard. If the result is within 90-110% of the standard value, analyze samples. A standard should be analyzed after every 10 samples and at the end of the analytical run. Table 1 lists non-temperature compensated values for both of the standards. Use this table as a reference when a calculator is not available to perform temperature correction on the standards.

6. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.

7. Turn mode switch to the appropriate conductivity scale (X100, X10, or X1). Use the scale that produces a mid-range output on the meter.

8. Wait for needle to stabilize (about 15 seconds) and record conductivity. Multiply the conductivity reading by the scale setting.

9. While gently agitating the probe, take sample temperature (°C) to nearest 0.5°C and record.

10. Rinse probe with deionized water.

11. Record specific conductivity and temperature.

12. Store probe in D.I. water between uses whenever possible.

13. Turn mode switch to the OFF position when finished.

Quality Control:

1. A quality control calibration standard of 1000 umhos/cm is to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within 90-110% of the standard value or the samples run after the last acceptable check standard are to be reanalyzed.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

Calculation:

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

Table 1
NON-TEMPERATURE COMPENSATED CONDUCTIVITY STANDARD READINGS FOR 1,000 AND 10,000 umhos/cm STANDARDS

Conductivity Standard 1,000 umhos/cm			Conductivity Standard 10,000 umhos/cm		
Conductivity Standard Should Read	at	Degrees C°	Conductivity Standard Should Read	at	Degrees C°
500		0	5000		0
520		1	5200		1
540		2	5400		2
560		3	5600		3
580		4	5800		4
600		5	6000		5
620		6	6200		6
640		7	6400		7
660		8	6600		8
680		9	6800		9
700		10	7000		10
720		11	7200		11
740		12	7400		12
760		13	7600		13
780		14	7800		14
800		15	8000		15
820		16	8200		16
840		17	8400		17
860		18	8600		18
880		19	8800		19
900		20	9000		20
920		21	9200		21
940		22	9400		22
960		23	9600		23
980		24	9800		24
1000		25	10,000		25
1020		26	10,200		26
1040		27	10,400		27
1060		28	10,600		28
1080		29	10,800		29
1100		30	11,000		30
1120		31	11,200		31
1140		32	11,400		32
1160		33	11,600		33
1180		34	11,800		34
1200		35	12,000		35

	Revision Date
	9-06-86
	4-27-87
	2-17-88
	2-09-90
Manager, Operations	1-4-92
Supervisor, Quality Assurance	
Director, Analytical Services	

Appendix C3

Field GC VOC Screen

Effective Date: 1-13-92

FIELD ANALYSIS OF VOLATILE ORGANICS

Scope and Application: This method covers the determination of the following 17 organic compounds in water and soil gas.

Target Compounds:

Benzene	trans-1,2-Dichloroethene	Trichloroethene
1,1-Dichloroethane	Ethyl benzene	m-Xylene
1,2-Dichloroethane	Tetrachloroethane	o-Xylene
1,1-Dichloroethene	Toluene	p-Xylene
cis-1,2-Dichloroethene	1,1,1-Trichloroethane	

Note: m-xylene and p-xylene are not separated by this method. Therefore the sum of the unresolved peaks are reported.

Method: Headspace - Gas Chromatographic/Photoionization and Hall Electrolytic Conductivity Detection.

Reference: "EPA Test Methods for Evaluating Solid Waste", SW-846 Methods 3810, 8010 and 8020 with modifications.

Reporting Limits: See table 1

Optimum Range: Headspace 5.0-50 ug/L of water, soil gas 5.0-50 ug/L of soil gas.

Sample 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 in a manner that provides 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).
2. Septum - Teflon-faced silicone (Pierce #12722 or equivalent).
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 mL gas tight syringes.

8. Balance - $\pm$ 0.0001 g.

9. Balance - $\pm$ 0.01 g.

10. Reagent water - organic free water or cold tap water which has been shown to be organic free at the method detection limit.

11. 25 mL TC graduated cylinders.

12. Constant temperature water bath - 55°C.

13. Volumetric flasks - assorted.

14. Pipettes - assorted.

15. Standard reference materials → Chem Service

16. Screw top vials - 10 mL

17. Mininert valves caps lined with teflon.

Standard Preparation:

1. **Stock standard solutions:** Prepare a VOC standard containing the target analytes at 5000 ug/mL in methanol.

   - 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. Replace the stopper.

   - Tare the flask on the analytical balance.

- Remove the stopper and, using a 100 uL syringe, add 0.125 g (correct for % purity) of the reference material to the flask. Make sure the drops fall directly into the methanol without contacting the neck of the flask. Replace the stopper.

- Determine the amount of reference material added. Rinse the syringe with methanol, tare the flask, and add the next standard.

- After all the reference materials are added, fill to volume with methanol, cap and invert to mix.

- Transfer the final stock standard into a screwtop vial and cap the using a mininert valve teflon cap.

2. **Secondary Standard Solutions**

Prepare secondary target standards according to the following scheme:

Standard	mLs	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	2 mL	10 mL	10 ug/mL

Dilute to the volume with methanol and transfer into a screwtop vial with mini inert valve teflon cap.

Note: Stock standards and secondary standards should be prepared before going out into the field. This will reduce in the necessary equipment needed on-site.

3. **Working Headspace Calibration Standards:** Prepare working calibration standards according to the following scheme:

Secondary Standard	Amount	Final Volume	Concentration
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

Fill a 200 mL volumetric flask with reagent water to the mark. Directly inject the secondary standard into the water with an appropriate microliter syringe.

Invert each 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 cap.

Calibration: Target Headspace Standards

1. Remove and discard approximately 10 mL from a freshly prepared standard and place the vial (capped) now having about 10 mL of headspace in a 55°C water bath with the water level sufficient to equal the water level in the vial.

2. Allow time for equilibration of temperature (10 minutes).

3. Through the septum of the vial, using a 5 mL gas tight syringe with needle remove 5 mL of headspace gas for injection into the gas chromatograph.

4. Construct an external standard curve of peak area response versus concentration for each of the compounds of interest.

5. A 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 target compounds varies from the calculated response by more than ± 30%, a new calibration curve should be prepared.

Soil Gas (Total Nanograms):

1. Inject 5.0 uL of the 5 ug/mL standard into the gas chromatograph.

2. Use a 1-point standard curve of peak area response versus total nanograms injected for each of the compounds of interest.

3. A 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%, the average response should be used.

Sample Analysis:

Water Samples:

1. Water samples are received in 40 mL VOC vials. Uncap and decant 10 mL of the sample from the vial. Recap the vial containing 30 mL of sample.

2. Place vials in a 55°C water bath and allow to equilibrate for 10 minutes.

3. Through the septum of the vial, using a 5 mL gas tight syringe with needle remove 5 mL of headspace gas for injection into the gas chromatograph.

4. If any compound of interest is outside the calibration curve **and an accurate concentration is required,** a dilution of the sample is made using organic free water and a fresh vial of sample. The headspace analysis is then repeated.

Soil Gas Samples:

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. Remove 5 mL of soil gas through the sampling septum and inject into the gas chromatograph.

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 bulb.

Chromatographic Conditions:

Column: 8-ft x 1/8-in 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 - 200°C

Oven*: Initial - 60° - 0 min
Rate 20°C/min
Final - 200°C - 7 min

* Conditions listed can be varied as needed for changing applications.

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.

Soil Gas Bulb 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 mass per liter of each parameter found in the samples using the following equation.

$$ng/L = \frac{R(samp) \times ng(std)}{R(std) \times VL}$$

where: R(samp) = Response of parameter in sample
R(std) = Response of parameter in standard
ng(std) = ng of standard injected
VL = Volume of aliquot taken from bulb (in L)

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 concentration being estimated.

3. All raw field data will be forwarded to Warzyn Inc., Analytical Laboratory for final review and archiving.

Quality Control:

1. Each analytical run should begin with a headspace standard curve consisting of 50, 10, 5 ppb and a blank. Every eleventh analysis thereafter and the last sample analyzed should be a 10 ppb standard. Continuing calibration standards should be within $\pm$ 30% of the original standards or a new standard curve should be prepared and samples analyzed since the last check standard reanalyzed.

2. Direct inject 5 uL of a 5 ug/mL target standard (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 analyzed, a duplicate sample should still be analyzed. Duplicates should be within $\pm$ 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 $4^{o}C$ (iced).

TABLE 1

Target VOC Reporting Limits for Water Headspace

Compound	Reporting Limits (ug/L of water)
Toluene	5.0
1,1-Dichloroethene	5.0
Trans-1,2-Dichloroethene	5.0
Trichloroethene	5.0
Tetrachloroethene	5.0
Benzene	5.0
Ethyl Benzene	5.0
1,1,1-Trichloroethene	5.0
1,1-Dichloroethane	5.0
1,2-Dichloroethane	5.0
cis-1,2-Dichloroethene	5.0
m+p-Xylene	5.0
o-Xylene	5.0

Soil Gas Reporting Limits

Compound	Reporting Limits (ug/L of soil gas)
Toluene	5.0
1,1-Dichloroethene	5.0
cis-1,2-Dichloroethene	5.0
Trans-1,2-Dichloroethene	5.0
Trichloroethene	5.0
Tetrachloroethene	5.0
Benzene	5.0
Ethyl Benzene	5.0
1,1,1-Trichloroethene	5.0
1,1-Dichloroethane	5.0
1,2-Dichloroethane	5.0
m+p-Xylene	5.0
o-Xylene	5.0

Appendix D

Laboratory Custody SOPs

Appendix D1

CompuChem

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories (if for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples).

- Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat, safety glasses and gloves). These items need to be worn at all times when in the marked areas (colored line).

- Each sample container is inspected before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, these items are inspected to make sure that they are intact and this observation is recorded on the chain-of-custody form (Attachment 1). If the custody seals, tapes, or padlocks are broken, one must contact Customer Service (for commercial samples) or the Sample Management Office (for EPA samples) for permission to continue processing the sample.

- Each container is opened under the hood and checked for breakage. The condition of the refrigerant is checked (whether any ice remains or whether the cooling packs are solid) and the temperature of a representative sample (liquid samples only) is obtained by immersing a clean thermometer in the sample. The temperature is recorded on the Sample Record (Attachment 2).

- The temperature and pH are recorded on the Log-In Sheet. If a temperature or pH variance occurs, a QA Notice is written and associated with the sample (Attachments 3-4).

- Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

- The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

- When a CompuChem SampleSaver is received, the SampleSaver number is recorded on the Log-In Sheet and is entered into the LMS system by number.

- Samples are removed from the shipping container and the sample identification information on the sample bottles is compared to the sample information on the traffic sheets, packing lists, and Chain-of-Custody Form included in the container (Attachments 5-11).* For EPA samples, Form DC-1 is filled out as well, per USEPA SOW. If discrepancies exist, the problem is noted on the Chain-of-Custody Form; the Receiving Clerk notifies Customer Service (for commercial samples) and the Supervisor notifies SMO (for EPA samples).

- Each water VOA is checked for air bubbles and headspace, and noted on the Chain-of-Custody Form as well as the Log-In-Sheet.

- On each complete and correct <u>EPA Chain-of-Custody and Traffic Report</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

- On each complete and correct <u>Commercial Chain-of-Custody</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

* NOTE: For samples associated with NJDEP X-408, an NJDEP059 "Sample Analysis Request Form" will accompany the samples upon receipt (Attachment 12).

Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. All sample tags (if received) regardless of condition are retained and stored in grey folders which are produced along with sample records, for CLP deliverables samples. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain-of-custody seals), the exceptions are noted on the appropriate documents, initialed and dated.

- The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

- Incoming samples are checked against SMO scheduled receipts (for EPA samples).

- The sample is logged in on the Log-In Sheet Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

- For EPA samples, the samples' account data is entered into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, customer service is contacted to check for the existence of the order and to receive requisitions for analyses. The order is then

completed in the CLMS, and the EPA Scheduling Log (Attachment 13) is also completed.).

- The sample is entered into the sample receipt portion of the CLMS in order to generate a CompuChem number for each sample. The CompuChem number is filled in on the accessioning log sheet (this completes the log sheet).

- The CompuChem number is a unique, six digit identity which is generated by the LMS system in numerical sequence. It can be cross-referenced to the Client ID.

- A CompuChem label is generated in numerical sequence, with the CompuChem number.

- The samples are labelled with the CompuChem number by wrapping each sample bottle with its computer-generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.

- Each Log-In Sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.

- The labelled samples are transferred to the secured, locked walk-in cooler facility.

- The CompuChem number is listed on the original Chain-of-Custody Form next to the associated client ID when possible.

- The Quiz portion of the CLMS is accessed to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, the appropriate worksheets are pulled from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with Customer Service. All laboratory worksheets are distributed to the appropriate Production Planning and Control Planner.

- To produce EPA quality control worksheets for the QC samples associated with a batch of samples, EPA Water or EPA Solid programs of Quiz in the CLMS is accessed and the samples' CompuChem Numbers are entered; these worksheets are copied on green paper. To assemble commercial QC worksheets, the appropriate green fraction worksheets are pulled from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary, a copy of the Chain-of-Custody Record.

- Commercial file folders are assembled for Report Integration; included in the production sample's folder are the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; the QC Sample Record is included in the green Quality Control folder which also goes to Report Integration.

- EPA file folders are assembled for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-of-Custody, original air-bill, a copy of the Log-In Sheet, a copy of the EPA Scheduling Log,

  Custody Tags (if received) and a gray envelope contents sheet (Attachment 9). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (Attachment 10).

- If problems arise concerning received samples, Customer Service is contacted (for commercial samples) or the Technical Management Staff is contacted (for EPA samples see below).

- Samples are sometimes received from the EPA that should not have been sent. Therefore, they require a transfer. The following steps should be taken:

  1. Fill out a new Chain-of-Custody Form using the information on the sample tags.

2. Sign the Chain-of-Custody in the first section labeled "Relinquished By".

3. A Copy of Chain-of-Custody is kept for our records; the original is sent with samples. A copy of the paperwork received with the samples should also be sent.

4. Notations are made on the Traffic Reports stating samples are being sent to another laboratory.

Samples hand-delivered after business hours should be recorded as follows:

1. The actual date of sample receipt shall be recorded on the Chain-of-Custody (see Note).

2. The date and time of sample receipt are recorded on the TRs as follows:

   a. Organic Traffic Report

   Date and time of receipt in Column F, sample condition upon receipt.

   b. Inorganic Traffic Report

   Same as above

The notation "H.D." (Hand-Delivered) or "Received by Common Carrier" should be recorded next to the actual sample receipt time and date. The following calendar day shall be recorded in the block designated for Receipt Date on the Organic/Inorganic Traffic Report.

Samples received after business hours are logged in like samples received during regular business hours (page 1 of this SOP details the procedure for logging in samples).

Note: For New Jersey DEP Chain-of-Custody procedure, see Production Planning and Control SOP 3.2 and the note on page 2 of 19 of this SOP.

№ 00933

COMPUCHEM LABORATORIES

CHAIN-OF-CUSTODY RECORD

PROJECT NAME:

PROJECT NUMBER:

SAMPLERS (SIGNATURE)

PRINTED NAME

CLIENT ID (9 CHARACTERS)

1	2	3	4	5	6	7	8	9

#	GC/MS					GC						INORGANICS				OTHER											SAMPLING INFO		REMARKS
No. of Bottles/Vials	624-8240	625-8270	TCL-VOA	TCL-SVOA	Other:	601-8010	602-8020	608-8080	8140	TCL PEST/PCB's	Herbicides	Other:	Metals	Cyanide	TAL Metals	Other:	TOC	TOX	Oil & Grease	Pet Hydro.	Phenols						MATRIX: Water/Soil	DATE	TIME	

RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME:		COMPANY NAME:		COMPANY NAME:	
RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time
COMPANY NAME:		COMPANY NAME:		COMPANY NAME:	
RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME:		COMPANY NAME:		COMPANY NAME:	

SHIPPING INFO

Number of Shipping Containers-

Method of Shipment

Special Handling Requirements

ATTACHMENT 1

Section No. 1.1
Revision No. 9
Date: September 10, 1990
Page 7 of 20

ATTACHMENT 2

Sample Record

Requisition Number:
Case:
Turnaround: Priority:
Analysis Codes:

CompuChem Number:
Account Number:
Due:

Lab Instructions

Receiving Data

Sample Identification:
SS Number:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

SS Code:
Receiving Codes:

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_____	____	( )
_____	____	( )
_____	____	( )
_____	____	( )

Company Name:

Sample Record

Requisition Number:
Case:

CompuChem Number:
Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

QUALITY ASSURANCE NOTICE

CompuChem# _____
Sample ID _____
Case# _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was ____, the required pH level is ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Preserve In-House _____
Analyze – Qualify with Notice _____
Dispose – Client will Resample _____
Subcontract Lab to Preserve _____

Supervisor Signature _____
Date _____

QANR2
900130

QUALITY ASSURANCE NOTICE

CompuChem# _____
Sample ID _____
Case# _____
Type of Analysis _____
Receipt Date _____

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C (+/-2C). The temperature on the sample above was ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Analyze – Qualify with Notice _____
Dispose – Client will Resample _____

Supervisor Signature _____
Date _____

QANR3
900130

COMPUCHEM LABORATORIES

RECEIVED ON:
CASE # ORG/NORG:
DELIVERED BY:
ACCOUNT #: CONTRACT #

ORDER#
FREIGHT #'s
REGION: $ AMT:

OF PAGES OF
CASE I'D
TAT C-OF-C Y or N
TAGS Y or N

#	REMARKS	REQ	SAMPLE ID	CC#	SD	TEMP	Sul	Chl	pH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: RECEIVED BY: REVIEWED:

COMPUCHEM LABORATORIES

RECEIVED ON:
CLIENT:
DELIVERED BY:
SUBCONTRACT#

ORDER#
FREIGHT #'s
SS#

OF PAGES OF
CASE I'D
TAT C-OF-C Y or N

#	REMARKS	REQ	SAMPLE ID	CC#	SD	TEMP	Sul	Chl	pH	ANAL. CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: RECEIVED BY: REVIEWED:

COMPUCHEM LABORATORIES

date shipped to consignee: _____ date received from consignee: _____

number of sample: _____

consignee name: _____

address: _____

_____

_____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

COMPUCHEM

CONTENTS OF FOLDER

DATE: _____

EPA: () ORGANIC () INORG

COMMERCIAL: ()

CASE #: _____

CLIENT: _____

NO. OF TRAFFIC RPTS: _____

ORDER#: _____

#	ITEM	Y	N	NA	COMMENTS
1	CHAIN-OF-CUSTODY	()	()	()	
2	AIRBILL	()	()	()	
3	DAILY LOG SHEET	()	()	()	
4	TRAFFIC REPORT	()	()	()	
5	TAGS	()	()	()	
6	SMO COVER LETTER	()	()	()	
7	SCHEDULING LOG	()	()	()	

PROBLEMS/COMMENTS:

SAMPLE RECEIVING:

DOCUMENTATION CONTROL:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SD
TRAFFIC REPORT (TR) COVER S

Lab Name: _____ Contract No.: 68-01-_____

Lab Code: _____ Case No.: _____ SAS No.: _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Lowest EPA Sample Number in first shipment of samples received under SDG)

Last Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Highest EPA Sample Number in last shipment of samples received under SDG)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____
Sample Custodian Date

ATTACHMENT 11

SAMPLE LOG-IN SHEET

Lab Name: ______________________ Page ____ of ____

Received By (Print Name): ______________________ Log-in Date: ______________

Received By (Signature): ______________________

Case Number: ______________

Sample Delivery Group No.: ______________

SAS Number: ______________

REMARKS:

1. Custody Seal(s) — Present/Absent* Intact/Broken
2. Custody Seal Nos.: ________ ________
3. Chain-of-Custody Records — Present/Absent*
4. Traffic Reports or Packing List — Present/Absent*
5. Airbill — Airbill/Sticker Present/Absent*
6. Airbill No.: ________ ________
7. Sample Tags — Present/Absent*
   Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ________
11. Time Received: ________

Sample Transfer

Fraction: ________ ________

Area #: ________ ________

By: ________ ________

On: ________ ________

EPA SAMPLE #	CORRESPONDING SAMPLE TAG #	CORRESPONDING ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Reviewed By: ______________________ Logbook No.: ______________________

Date: ______________________ Logbook Page No: ______________________

FORM DC-1

3/90

Form DEP-050
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTE

SAMPLE ANALYSIS REQUEST FORM

SAMPLE LOCATION

NAME OF FACILITY/ COMPANY/PERSON	CODE	MAILING ADDRESS
SAMPLE LOCATION		

SAMPLE COLLECTOR

SAMPLER(S) Name	DIVISION		
Name	CONTRACT #	Task #	Report Format

SAMPLE IDENTIFICATION

NJDEP SAMPLE NO.	SAMPLING DATE	ABSCISSA (Longitude)
SAMPLE POINT ID	TIME (MIL) SAMPLING BEGAN	ORDINATE (Latitude)
SAMPLE SOURCE CODE	TIME (MIL) SAMPLING FINISHED	ELEVATION
REFERENCE POINT		

SAMPLING PROCESS

COLLECTION METHOD	Code	Other	SAMPLE TYPE	Code	Other
TYPE OF SAMPLER	Code	Other	SAMPLE MATRIX	Code	Other
DESCRIPTION					

FIELD MEASUREMENTS

SAMPLE TEMP.	°C	PARAMETER	PARAMETER	PARAMETER	
AIR TEMP.	°C	VALUE	VALUE	VALUE	
WEATHER					

CHAIN-OF-CUSTODY IMPLEMENTED ☐ YES ☐ NO | PRESERVATION CODE(S) ___ ___ ___ ___ ___

REQUESTED ANALYSIS(ES)

PARAMETERS (by Compound or Group of Compounds)	CONTAINER NUMBER	DETECTION LIMIT (digits/units)	EMERG. (Circle One)	NON-EMERG.

ANALYSIS(ES) AUTHORIZED BY	TITLE

ANALYZING LABORATORY

CONTRACT LAB NAME	PERSON ACCEPTING SAMPLE	TITLE	
LAB ID #	LAB CONTROL #	DATE	TIME (MIL)

COMPUCHEM LABORATORIES

EPA SCHEDULING LOG

WEEK ENDING SATURDAY:

CASE#: () NEW CASE () OLD CASE () COMPLETED CASE

REGION: TURNAROUND TIME:

ACCOUNT #: CONTRACT #: CONTRACT $

****

QUANITY RECEIVED # OF SAMPLES

WATERS	
SEDIMENTS/SOILS	
OTHER	

CONCENTRATION: () LOW () MEDIUM () HIGH

ORDER#:	
AIRBILL#:	

****

PROBLEMS/COMMENTS:

SMO CONTACTED AT (TIME) SPOKE TO:

RESOLUTION FROM SMO:

Appendix D2

Warzyn

Effective Date: 1-8-92

CHAIN-OF-CUSTODY
Superfund Level

Scope and Application: Chain-of-custody procedures are used to maintain and document sample possession. This particular procedure is applicable when full chain-of-custody procedures are required for enforcement driven investigations.

Reference: **NEIC Policies and Procedures, May, 1978 (Revised March, 1986).**
EPA-330/9-78-001-R.
U.S. EPA Central Regional Laboratory (10/15/82).

Notes: Due to the legal nature of enforcement driven investigations, possession of samples must be traceable from the time the samples are collected until introduced as evidence in legal proceedings or destroyed. To maintain and document sample possession, strict chain-of-custody procedures are followed.

A sample is under your custody if:

1. It is in your possession, or
2. It is in your view, after being in your possession, or
3. It was in your possession and you locked it up, or
4. It is in a designated secure area.

Field Custody:

1. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched to the appropriate laboratory.

3. Sample tags and labels shall be completed for each sample, using water proof ink (unless prohibited by weather conditions). **For example:** a log book notation would explain that a pencil was used to fill out the sample tag/label because a ball point pen would not function in freezing weather.

4. The field project coordinator determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment:

1. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information is entered in the "Remarks" (#14) section of the custody record (see Figure 3). Shipping containers are "sealed" with numbered chain-of-custody seals. The seal numbers are also noted in the "Remarks" section of the custody record.

2. Samples are accompanied by a chain-of-custody record (see Figure 3). When transferring the possession of samples, field personnel will complete the information required for #1 through #11 on the chain-of-custody. Any remarks pertaining to shipping are included in #14. This information is required prior to the field personnel relinquishing custody of the samples. This record documents sample custody transfer from the sampler, to the mobile laboratory or a distant laboratory.

3. Each shipping container will be accompanied by a chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the field project coordinator.

Sample Receipt and Sample Log-In Procedures:

Safety Precautions: All samples received should be considered hazardous and appropriate precautions should be taken when handling these samples. Under no circumstances should any personnel other than the sample custodian or project leader open coolers. If damage or leakage is noted, stay clear of the coolers and notify the sample custodian or project leader immediately. All samples will be opened in a hooded area!

Sample Receipt:

1. The project leader will notify the sample custodian in writing of incoming samples.

2. The custodian will receive the samples and deliver them to a hood located in the analytical laboratory. The procedures described below will be followed by the sample custodian:

   a. Examine the shipping container and record the following information on the project log form (Refer to Figure 1). A separate project log form is required for each shipping container.

      - The presence/absence of custody seal on the shipping container.
      - The condition of the custody seal (i.e., intact, not intact).

   b. Open the shipping container in a hooded area, remove the enclosed sample documents, and record the following information on the project log form.

      - The presence/absence of the chain-of-custody record(s).
      - The presence/absence of airbills and/or bills of lading documenting shipment of the samples.

   c. Remove the samples from the container and record the following information in the project log form.

      - Condition of samples (intact, broken, leaking, cold, etc).
      - The presence/absence of sample tags.

- Sample tag numbers. Compare these numbers with the chain-of-custody record(s) (Figure 3). If sample tag numbers do not match. Record this fact on both the chain-of-custody record and project log form and contact the operations manager so that the discrepancy can be resolved.

d. Compare the following documents to verify agreement of the information contained on them.

- Chain-of-custody records.
- Sample tags.
- Airbills or bills of lading.

e. Sign the Chain-of-custody (Figure 3 - #12 and #13), project log form and airbills.

Log-In and Tracking:

Note: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

1. **Log-In Procedure:** Samples are logged in as follows (Refer to Appendix 1):

a. **Sample Information:** Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold. The project manager will be informed about discrepancies and will determine action to be taken.

b. **Numbering Samples:** Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book.

Note:
- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
- If metals are requested, metal digestion is required for all matrices.
- Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

c. **Subcontracted Work Required:** Refer to the subcontracting SOP for further instructions on subcontracting samples.

d. **Special Accounting Notes:** If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

e. **Labeling Bottles:**

- Record the lab number on the sample bottle label, tags and caps using a water-proof marker.
- Check that the appropriate preservation and preparation has been circled.
- If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

f. **Preserving Samples:** Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. The project manager will be notified about any incorrectly preserved samples. Check preserved samples as follows:

- Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. **It is critical that the correct type of acid be added.**
- Base-preserved NaOH samples must be preserved to a pH > 12. Using a pH stick, check the pH. If the pH is not > 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 12 is obtained.
- Base-preserved NaOH with ZnAc samples must be preserved to a pH > 9. Using a pH stick, check the pH. If the pH is not > 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 9 is obtained.

1. Samples which have not been preserved in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. Refer to Figure 4 for preservation requirements and preserve accordingly. It is important that samples arrive in appropriate containers (see Figure 4). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.
2. It is important that samples be received and maintained cold for most analyses. If a sample was received warm, make note of this on the field sheet or custody record.

Computer Log-In:

1. Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

Computer Log-on

- Turn on the computer terminal.
- Enter user name > last name [return].
- Local prompt > c_chico [return].
- User name: ACSORA [return].
- Password: [return].
- Please enter your last (15 characters max.): Last name: [return].
- $Labmenu [return].
- Username: Last name [return].
- Password: [return].
- Select Log-in/Order entry and press [commit].

2. The log in screen will appear as shown in figure 2. Using information on the chain-of-custody, proceeds as follows:

   a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the project name on the chain-of-custody, press return.

   b. Sample date automatically defaults to yesterday's date. If this is correct, press return or override with correct date.

   c. Enter sample identification from **station location** on the chain-of-custody and press return.

   d. Enter proper matrix number and press return.

   e. **Desc:** This is used if sample identification is longer than the number of characters in the sample identification field. (Return to override or enter information and return.)

   f. Date received automatically defaults to today's date. If correct, press return or override with correct date.

   g. Days to process automatically defaults to 14 days. If this is correct press return or for standard turnaround time (usually 4 weeks) or if rush work, (indicated on request form) override with the number of days until due.

   h. **Duedate:** First date listed is report date, second date is the lab due date which can be changed if necessary by changing days to process.

   i. **Comments:** Enter "! CLP Protocol", special tests or other information needed to analyze samples correctly and press return.

j. **Manager:** Filled in automatically; press return if correct or override.

k. **Markup/Discount:** Return if correct or override.

- % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.

l. To enter parameters, press **Next Screen** (see Figure 2A).

Note: To move from block to block press Next/Previous screen. To move from field to field, press Return/Tab or F12 (previous field) key.

m. Matrix automatically defaults to matrix entered above (return or override).

n. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.) To delete use remove key.

o. To enter address for the report, press **Next Screen** (see Figure 2B).

p. **Type:** Report (return).

q. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

r. If no address appears, press return until cursor is in the City field. Type in the city and press return.

s. Type in state (press **Commit** and return). After city and state have been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.

3. To duplicate the entry for another sample on the same chain-of-custody:

   a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).

   b. Enter sample identification and press **Commit** key, return.

4. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):

   a. Press **Create Record** key (Insert Here) and enter sample identification.

   b. Press **Previous Screen** (F12) if sample date needs to be changed.

   c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.

d. Press **Previous Screen** to enter new sample or to quit.

e. Press **Clear Form** (F17) to enter a new chain-of-custody (new log in number).

f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.

- Press **Exit/Cancel** to exit from the LIMS system.
- At the VMS $ prompt, enter LO (return) to log off the VAX computer.
- Enter N (return).

5. To print daily log-in reports:

   a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:

   b. Select "Seedpak Reporting" from the main menu, and press **Commit**.

   c. Select FS02 and press **Commit**.

   d. Enter today's date, press **Commit** and return.

   e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.

   f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

Sample Storage:

1. Samples and extracts will be stored in a secure area designated for strict chain-of-custody samples.

2. Damaged samples will be disposed of in an appropriate manner and the method of disposal documented.

3. The laboratory and sample storage areas are secure with strict limited access by only laboratory personnel.

4. Whenever samples are removed from storage, the removal will be documented. All transfers of samples will be documented on the internal chain-of-custody records. (Refer to Figure 5).

5. Samples and extracts will be stored after completion of analysis in accordance with the contract or until sample custodian is instructed otherwise by the enforcement Project Officer.

6. The location of stored organic extracts will be recorded.

7. VOA samples will be stored separately from organic extracts.

8. Standards are stored separate from samples.

9. Samples requiring refrigeration are stored in the walk-in coolers refrigerator. Metal samples are stored in the black cabinets in log-in. Metal digestates are stored on shelves in the metals lab.

Sample Security:

1. Samples will be stored in a secure area.

2. Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked.

3. Visitors will sign a visitors log (located at the reception area and will be escorted while in the laboratory area.

4. Refrigerators, freezers, and other sample storage areas are secure as they are located within the laboratory.

5. Only laboratory personnel have access to the laboratory.

6. Samples will remain in secure sample storage until removed for sample preparation or analysis. All transfers of samples into and out of storage will be documented on an internal chain-of-custody record. An example of an internal chain-of-custody record used for this procedure is attached. (See Figure 5).

7. These internal custody records will be maintained in the project file.

8. After a sample has been requested from storage by the analyst, the analyst is responsible for the custody of the samples. Each analyst must return the samples to the storage area before the end of the working day.

Internal Chain-of-Custody: The following procedure for documentation of internal chain-of-custody for samples requiring continuous custody.

1. All transfer of samples into or out of the storage areas will be documented on an internal chain-of-custody record (see Figure 5). These records are maintained by the sample custodian.

2. Once a sample is removed from a walk-in by the sample custodian, he/she will then give it to the analyst, who is then responsible for the custody of the sample. Each analyst **must return** samples to the walk-in **before the end of the working day**. Samples are not allowed to sit on the bench overnight.

3. The next morning, the sample custodian will put samples back in their proper place on the shelves. Sample custodian is responsible for signing sample request sheet that samples were returned properly. Any remarks will also be noted by the sample custodian.

4. When sample analyses and necessary quality assurance checks have been completed by the laboratory the unused portion of the sample should be disposed of properly. (See sample disposal section.) All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation of the project. **Tags and forms are never disgarded!**

Sample Disposal:

1. Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

   a. Two months after the report issue date, aqueous inorganic samples which are in cold storage are to be transferred to warm storage.

   b. Two months after the report issue date, aqueous organic samples which are in cold storage are to be transferred to warm storage.

   c. Three months after report issue date, soil samples which are in cold storage are to be transferred to warm storage.

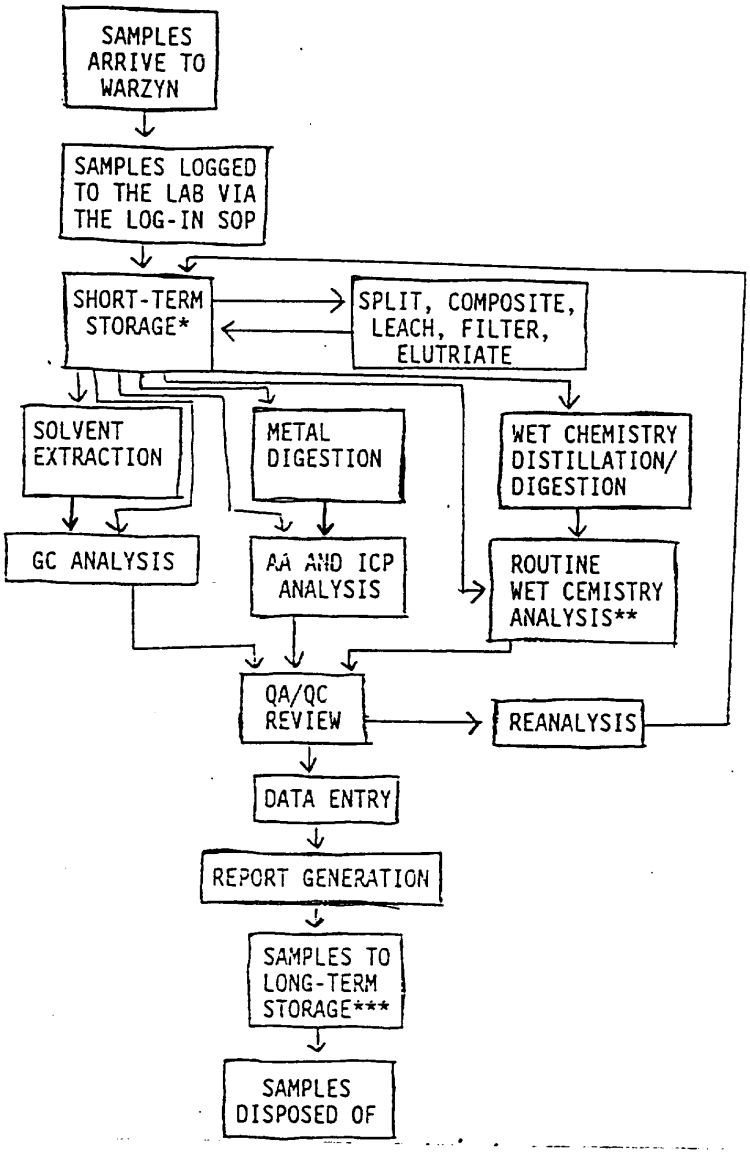
   d. Samples in warm storage should be disposed of as follows:

      - Aqueous Samples: Two months after report issue date, a memo is sent to the project manager stating that the samples will be disposed of 1 month from the date of the memo. The samples are then disposed of 3 months after report issue date unless project manager requests alternate arrangements.

      - Non-Aqueous Samples: Six months after report issue date, a memo is sent to the project manager requesting permission to dispose of the samples. This memo is repeated on a quarterly basis until project manager approves sample disposal.

APPENDIX 1

WARZYN ENGINEERING INC.
ANALYTICAL SERVICES
SAMPLE FLOW DIAGRAM



* SHORT TERM STORAGE: WALK-IN REFRIGERATOR FOR SAMPLES REQUIRING REFRIGERATION, BLACK CABINETS IN SAMPLE LOG-IN AREA FOR SAMPLES NOT REQUIRING REFRIGERATION, WALK--INS FOR SAMPLES REQUIRING "SUPERFUND LEVEL CHAIN-OF-CUSTODY".

** ROUTINE WET CHEMISTRY ANALYSES INCLUDE BOTH AUTOMATED AND MANUAL TESTS.

*** LONG TERM STORAGE: NON-REFRIGERATED STORAGE IS LOCATED IN THE FIELD STORAGE ROOM.

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FIGURE 1

Sample Custodian Signature: _____
Date: _____

(Circle Appropriate Response)

Custody Seal — present/absent; intact/not intact

Chain-of-Custody — present/absent

Sample Tags — present/absent

Sample Tag Numbers — listed/not listed on chain-of-custody

Project Number: _____

Airbill Number: _____

Date Rec'd	Time Rec'd	Chain-of Custody Record No.	Corresponding Sample Tag Nos.	Corresponding Assigned Nos.	pH <2	pH >9	pH >12	Does info. on Custody Records, Traffic and Sample Tags Agree	Remarks Condition of Samples

FIGURE 2

```
+------------------------------ Login/Order Entry ------------------------------+
|Project #: STANDARD___________ Acct:_________ ___________________________________|
+-------------------------------------------------------------------------------+
| Sample #: __________ Sample Date: _________ Sample ID: ____________________    |
|   Matrix: ___________________ Desc: ____________________________              |
| Received: 14-FEB-90  Days to Process:  14__  Due Date: _________ _________    |
| Comments: ____________________________________ Manager: ____________________  |
|    Order: __________ on 14-FEB-90          Price/Cost: 0_________/0_________  |
|    Login: __________                  % Markup/Discount: 0_____               |
+-------------------------------------------------------------------------------+
|  Matrix        Product              Type Price      Cost       Hold date PList |
|  __ __________ ____________________ __ __________ __________ _________ __     |
|  __ __________ ____________________ __ __________ __________ _________ __     |
|  __ __________ ____________________ __ __________ __________ _________ __     |
|  __ __________ ____________________ __ __________ __________ _________ __     |
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|  __ __________ ____________________ __ __________ __________ _________ __     |
|  __ __________ ____________________ __ __________ __________ _________ __     |
|  __ __________ ____________________ __ __________ __________ _________ __     |
+-------------------------------------------------------------------------------+
Char Mode: Replace  Page 1                                   Count: *0
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FIGURE 2A

```
+------------------------------ Login/Order Entry ------------------------------+
|Project #: 60818.00_____________ Acct:9999______ GPC-576_____________________  |
+-----------------------------------------------------------------------------+
| Sample #: 496-003___ Sample Date: 28-JAN-90 Sample ID: MW-3_________________  |
|   Matrix: 1  GroundH2O_____ Desc: ___________________________________________  |
| Received: 31-JAN-90  Days to Process:  14__  Due Date: 14-FEB-90 12-FEB-90   |
| Comments: ___________________________________ Manager: CLARK,_J_____________  |
|    Order: 619_______ on 31-JAN-90          Price/Cost: 312_______/0_________  |
|    Login: 496_______                  % Markup/Discount: 0_____              |
+-----------------------------------------------------------------------------+
|  Matrix        Product            Type Price      Cost       Hold date PList  |
|  1_ GroundH2O_ AG                 S 12_______ 0_________ 12-FEB-90 S          |
|  1_ GroundH2O_ AS                 S 25_______ 0_________ 12-FEB-90 S          |
|  1_ GroundH2O_ BA                 S 12_______ 0_________ _________ S          |
|  1_ GroundH2O_ CD                 S 12_______ 0_________ 12-FEB-90 S          |
|  1_ GroundH2O_ CR                 S 12_______ 0_________ 12-FEB-90 S          |
|  1_ GroundH2O_ DIGASSE            S 0________ 0_________ _________ S          |
|  1_ GroundH2O_ HG                 S 32_______ 0_________ 25-FEB-90 S          |
|  1_ GroundH2O_ PB                 S 22_______ 0_________ 12-FEB-90 S          |
|  1_ GroundH2O_ SDF                S 10_______ 0_________ _________ S          |
|  1_ GroundH2O_ SE                 S 25_______ 0_________ 12-FEB-90 S          |
+-----------------------------------------------------------------------------+

      v Char Mode: Replace  Page 1                    Count:  1
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FIGURE 2B

```
                                  Sample Addresses for 496-003___
Type        #     +-------------------------------------------------+
REPORT____  1_    |        Name: _____________ - ___________________|
                  |       Addr 1: ______________________________________|
                  |       Addr 2: ______________________________________|
                  |        City: ROSEVILLE_____________________________|
                  |  State/ZIP: MN__ ____________                      |
                  |       Phone:  __-___-____                          |
Type              |                                                    |
_________ __      |        Name: _____________ - ___________________|
                  |       Addr 1: ______________________________________|
                  |       Addr 2: ______________________________________|
                  |        City: ______________________________________|
                  |  State/ZIP: ____ ____________                      |
                  |       Phone: ___ ___ ____                          |
Type              |                                                    |
                  |        Name:                                       |
                  |       Addr 1:                                      |
                  |       Addr 2:                                      |
                  |        City:                                       |
                  |  State/ZIP:                                        |
                  |       Phone:                                       |
                  +----------------------------------------------------+
Char Mode: Replace  Page 2                                  Count: *1
```

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One Science Court
University Research Park
P.O. Box 5385
Madison, Wisconsin 53705
(608) 273-0140

FIGURE 3

CHAIN OF CUSTODY RECORD

PROJ. NO. #1	PROJECT NAME # 2A / LOCATION: # 2B	NO. OF CONTAINERS #8	REMARKS #9
SAMPLERS: (Signature) #3			

LAB NO. #15	DATE #4	TIME #5	COMP.	#6 GRAB	#7 STATION LOCATION	NO. OF CONTAINERS #8							REMARKS #9

Relinquished by: (Signature) #10	Date / Time #11A #11B	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature) #12		Date / Time #13A #13B	

Remarks #14

PROJECT MANAGER: #15

FIGURE 4

(Page 1 of 3)

SAMPLE COLLECTION AND PRESERVATIVE REQUIREMENTS

Parameter	Container, (size in mLs)		Preservation	Maximum Holding Time
Wet Chemistry				
Acidity	P,G	(A)	Cool, 4°C	14 days
Alkalinity	P,G	(A)	Cool, 4°C	14 days
BOD_5	P,G	(1000)	Cool, 4°C	48 hours
Boron	P	(A)	Cool, 4°C	
Bromide	P,G	(A)	None Required	28 days
Carbon, Total Organic	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chemical Oxygen Demand	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride	P,G	(A)	None Required	28 days
Chlorine, Residual	P,G	(A)	None Required	Analyze Immediately
Cyanide, Amenable	P,G	(1000)	Cool, 4°C, NaOH to pH>12 (I)	14 days (I)
Cyanide, Total	P,G	(1000)	Cool, 4°C, NaOH to pH>12 (I)	14 days (I)
Density/Specific Weight	P,G		Cool, 4°C	
Fluoride	P,G	(A)	None Required	28 days
Hardness	P,G	(C)	HNO_3 to pH<2	6 months
Nitrogen, Ammonia	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Nitrate	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Nitrite	P,G	(A)	Cool, 4°C	48 hours
Nitrogen, Total Kjeldahl	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Total Organic	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Oil and Grease	G	(1000)	Cool, 4°C, H_2SO_4 to pH<2	28 days
pH	P,G	(A)	None Required	Analyze Immediately
Phenolics, Total	G	(500)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorous, Ortho	P,G	(A)	Filter Immediately, Cool 4°C	48 hours
Phosphorous, Total	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Silica, Dissolved	P	(A)	Cool, 4°C	28 days
Solids, Dissolved	P,G	(D)	Cool, 4°C	7 days
Solids, Settleable	P,G	(1000)	Cool, 4°C	48 hours
Solids, Suspended	P,G	(D)	Cool, 4°C	7 days
Solids, Total	P,G	(D)	Cool, 4°C	7 days
Solids, Volatile	P,G	(D)	Cool, 4°C	7 days
Specific Conductance	P,G	(A)	Cool, 4°C	28 days
Sulfate	P,G	(A)	Cool, 4°C	28 days
Sulfide	P,G	(500)	Cool, 4°C, add 2 mL 2N Zinc acetate/1000 mL plus NaOH to pH>9	7 days
Surfactant (MBAS)	P,G	(1000)	Cool, 4°C	48 hours
Thiocyanate	P,G	(500)	Cool, 4°C	
Total Organic Halides (TOX)	Amber Glass	(E)	Cool, 4°C	28 days
Turbidity	P,G	(A)	Cool, 4°C	48 hours
Metals				
Hexavalent Chromium	P,G	(A)	Cool, 4°C	24 hours
Mercury	P,G	(C)	HNO_3 to pH<2	14 days in plastic, 28 days in glass
Metals (all, except above)	P,G	(C)	HNO_3 to pH<2	6 months
Organics				
Gas Chromatography				
Volatile Organics	VOC vial	(2 ea, 40)	Cool, 4°C (J)	14 days (J)
Phenols	Amber glass	(H)	Cool, 4°C (K)	7 days (O)
Pesticides and PCBs	Amber glass	(H)	Cool, 4°C (L)	7 days (O)
Pesticides	Amber glass	(H)	Cool, 4°C (L)	7 days (O)
PCBs	Amber glass	(H)	Cool, 4°C	7 days (O)
Polynuclear Aromatics	Amber glass	(H)	Cool, 4°C (K), Store in Dark	7 days (O)
Organophosphorus Pesticides	Amber glass	(H)	Cool, 4°C, Store in Dark	7 days (O)
Chlorinated Herbicides	Amber glass	(H)	Cool, 4°C	7 days (O)
Total Hydrocarbon Screen	Amber glass	(H)	Cool, 4°C	7 days (O)
GC/MS				
Priority Pollutants/HSL				
Volatiles	VOC vial	(2 ea, 40)	Cool, 4°C	
Semi-volatiles	Amber glass	(H)	Cool, 4°C	
Pesticides/PCBs	Amber glass	(H)	Cool, 4°C	
CLP Protocol				
Volatiles	VOC vial	(2 ea, 40)	Cool, 4°C	
Semi-volatiles	Amber glass	(H)	Cool, 4°C	
Pesticides/PCBs	Amber glass	(H)	Cool, 4°C	
Radiological Tests				
Alpha, Beta	P,G	(250)	HNO_3 to pH<2	6 months
Radium	P,G	(1 gal)	HNO_3 to pH<2	6 months
Bacterial Tests				
Coliform, Fecal and Total	P,G	(F)	Cool, 4°C (F)	6 hours
Fecal Streptococci	P,G	(F)	Cool, 4°C (F)	6 hours
Solids				
RCRA Classification	P,G	(1000) (G)	Cool, 4°C	--
Volatile Organics	VOC vial	(140) (H)	Cool, 4°C	--
Semi-volatile Organics	G	(8 oz)	Cool, 4°C	--
Metals	P,G	(8 oz)	Cool, 4°C	--
Inorganics	P,G	(8 oz)	Cool, 4°C	--

FIGURE 4

Notes

(A) 1-3 Analyses; 250 mL required
4-7 Analyses; 500 mL required
>8 Analyses; 1000 mL required

(B) 1-3 Analyses; 250 mL required
4-7 Analyses; 500 mL required

(C) The metals selenium, arsenic, silver, mercury and hardness require additional volume. Use the following bottle scenario;
- One 250 mL bottle for all metals excluding above
- One 250 mL bottle for all metals plus two of the above analytes (except mercury). If mercury is required, an additional 250 mL is required. Label bottle for mercury analyses.
- One 500 mL bottle for all metals, including all five of the above analytes. Two 250 mL bottles may be used instead.

(D) Dissolved solids require 100 mL of sample. This volume can be included in the (A) unpreserved bottle.
The volume required for suspended, total and volatile solids depends on the nature of the sample. Dirty samples require 100-250 mL, clean require 1000 mL. Sample accordingly.

(E) Collect sample in either a 250 mL amber bottle with inverted cap or 250 or 500 mL amber bottle with teflon septa. Collect with no headspace.

(F) Collect 250 mL using either sterilized Whirl-Pak bag or nalgene bottle. Collect in Whirl-Pak bags with sodium thiosulfate ($Na_2S_2O_3$) additive if the sample is chlorinated.

(G) Collect the sample in a 1-liter wide-mouth jar. Collect at least 500 grams for analysis.

(H) Volatile organics may also be collected using two 40-mL VOC vials.

(I) Add 0.6 g ascorbic acid if residual chlorine is present.
Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, remove by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is then filtered and NaOH is added to pH >12.

(J) Preserve chlorinated samples with 1 crystal of sodium thiosulfate ($Na_2S_2O_3$) per vial.
Samples can be either preserved with 1:1 HCl or left unpreserved.
Samples receiving no pH adjustment must be analyzed within 7 days of sampling.
Samples that contain hydrocarbons (samples from gasoline or fuel oil contaminated sources) must be preserved with 1 drop of 1:1 hydrochloric acid (HCl).
If either acrolein and acrylonitrile are to be analyzed, the pH should be adjusted to 4-5 with 1:1 hydrochloric acid (HCl). Samples receiving no pH adjustment must be analyzed within 3 days of sampling.

FIGURE 4
(Page 3 of 3)

(K) Add 0.008% sodium thiosulfate ($Na_2S_2O_3$) if residual chlorine is present.

(L) Adjust pH to 5-9 with sulfuric acid (H_2SO_4). The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of Aldrin, add 0.008% sodium thiosulfate ($Na_2S_2O_3$) to the sample.

(M) Collect two (2) 1000 mL amber glass bottles with teflon-lined caps per analysis.

(N) Collect two (2) 80 oz amber glass bottles with teflon-lined caps for one or both analyses listed. Alternatively, four (4) 1-liter amber bottles with teflon-lined caps may be used.

(O) The holding time is 7 days until extraction, 40 days after extraction.

BJH/cac
[cac-700-93A]

FIGURE 5

WARZYN SAMPLE REQUEST SHEET

Requested By: _____

Date/Time Submitted: _____

Need Samples By:

Date: _____

Time: _____

I am requesting:

Project No.	Sample No(s)	Parameter	Matrix	Remarks

I am returning: Date/Time:

Project No.	Sample No(s)	Remarks

Appendix E

Data Validation Procedure for Evaluating Inorganic Data

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

Scope and Application:

This standard operating procedure (SOP) describes Warzyn's procedure for evaluating inorganic data according to procedures specified in a Quality Assurance Project Plan (QAPP). Each analytical procedure is specific to a project and is found or referenced in the QAPP. The reviewer must evaluate whether the procedure was adhered to and that the required Quality Control (QC) requirements were met as described in the QAPP and evaluate the usability of the data.

References:

1. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, Hazardous Site Evaluation Division, U.S. EPA, July, 1988 (or most current).

2. Statement of Work for Inorganics Analysis, U.S. EPA Contract Laboratory Program, SOW No. 788, July, 1988 (or most current).

3. Quality Assurance Project Plan (QAPP) containing the analytical procedures required for evaluation. Each QAPP is specific to a project.

Requirements:

1. It is recommended that the reviewer be familiar with the RAS Inorganics validation process (refer to the Validation Guidelines and Statement of Work above) prior to evaluating this data. The evaluation and action criteria described in this SOP are very similar to the RAS Inorganics validation process.

2. All completed validated data packages must include the following:

   a. Data Validation Narrative
   b. Validated Results; with data qualifiers added and pages stamped "VALIDATED".

NOTE: It is not necessary to include the raw data with the completed validated data package, since a copy of the raw data package already exists in the project file. Only include raw data if there are major concerns or deficiencies with the data set.

Procedure:

1. Screen the data package to see that the samples and analytes undergoing evaluation concur with what was requested on the "Request for Data Validation" form (refer to Figure 1). If discrepancies occur, contact the data management coordinator for clarification prior to continuing.

2. Review the QAPP and the specific analytical procedure for the analyte undergoing evaluation. They will contain:

   a. A summary of the method required and method reference,
   b. The applicable matrices and recommended hold times,
   c. An approximate number of samples and matrix,
   d. Special technical instructions,
   e. Required deliverables,
   f. Data Requirements (Detection Limits, Precision, etc.), and
   g. Quality Control Requirements.

3. The reviewer must evaluate the data according to the quality control criteria specified in the QAPP. The evaluation of each analyte, in most cases, will include a review of the following criteria:

   a. Methodology,
   b. Raw Data/Quality Control Deliverables,
   c. Detection Limits,
   d. Holding Times,
   e. Calibration (Initial and Continuing),
   f. Blanks (Calibration and Preparation),
   g. Laboratory Control Samples (EPA Reference Samples),
   h. Laboratory Duplicates,
   i. Matrix Spikes,
   j. Sample Result Verification,
   k. Field Duplicates,
   l. Other (as specified in the procedure),
   m. Overall Assessment for a Case.

4. Evaluate the data for the criteria above as follows:

A. METHODOLOGY

<u>Evaluation</u>

1. Examine the data to determine if the acceptable analytical methodology was used as indicated in the QAPP.

2. Determine if the analytical procedure was followed.

<u>Action</u>

1. If an acceptable analytical method was not used, contact the Project Manager. It is the Project Manager's responsibility to inform the reviewer to continue the validation process of the data as received, or to reject the data. In some cases reanalysis or

resampling may be required, however the decision is left up to the Project Manager. The Project Manager may require that the reviewer determine the "usability" of the data package as submitted prior to deciding what formal action should be taken.

2. If the analytical procedure was not followed as stated, use professional judgement in determining whether the data should be qualified as estimated or unusable.

B. RAW DATA/QUALITY CONTROL DELIVERABLES

Evaluation

1. The QAPP will specify what information should be submitted as part of the data package. Determine whether the data package submitted is complete as described in the QAPP. Usually the following will be required:
   - Method used,
   - Calibration/standardization information,
   - Bench records tabulating the order of analysis of samples, standards, blanks, duplicates and spikes with the resulting instrument readouts and final concentrations. Records submitted should be complete enough such that all results could be recalculated from this raw data.
   - Photocopy of all instrument readouts (strip charts, printer tapes, etc.)
   - Reference (QC) samples to be identified by source, lot number and sample number, with corresponding true values and 95% confidence limits provided.
   - Custody records

Action

1. If the data package is not complete, contact the data management coordinator. The data management coordinator will contact the appropriate laboratory or project manager and obtain the information necessary to complete the evaluation process.

C. DETECTION LIMITS

Evaluation

1. Evaluate whether the detection limits required in the QAPP were achieved by the method used.

Action

1. If detection limits were not reported at least to what was required in the QAPP, review the raw data to determine if the detection limits were achievable. If they were, recalculate the data and report results to the required detection limits.

2. If detection limits were not achievable by the laboratory, determine why (laboratory sensitivity problem or detection limits unobtainable for the method used. Inform the project manager of

the situation. The project manager will determine whether the evaluation should be completed.

D. HOLDING TIMES

Evaluation

1. Determine whether the established holding times were met. The holding time is established by comparing the DATE SAMPLED with the DATE OF ANALYSIS found on the raw data. Required hold times should be noted in the QAPP.

2. Examine the digestion/distillation logs to determine if samples were preserved at the proper pH.

Action

1. If hold times were not noted in the QAPP, use the EPA-recommended hold times and apply these hold times to both water and soil samples.

2. If holding times and preservation criteria are not met, qualify all results $>$ Instrument Detection Limit (IDL) and estimated (J) and results $<$ IDL as estimated (UJ).

3. Use professional judgement in cases where the holding time is grossly exceeded. The expected bias would be low and the reviewer may determine that results $<$ IDL are unusable (R).

E. CALIBRATION

Evaluation

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank. In cases where a curve set-up is not applicable (i.e. a titrimetric procedure), verify that the titrants were standardized as required in the procedure.

2. Verify that the correlation coefficient is ≥ 0.995 (unless otherwise specified).

3. Verify that the calibration verification standards (ICV/CCV) were run at the appropriate frequency and that results were within acceptable limits as stated in the QAPP.

4. Recalculate approximately 10% of the ICV/CCV using the following equation:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

Action

1. If the minimum number of standards as required in the QAPP were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

2. If the correlation coefficient is < 0.995 (or as specified in the QAPP), qualify results > IDL as estimated (J) and results > IDL as estimated (UJ).

3. If standardization of titrants was required and not performed, use professional judgement in qualifying the data. The reviewer should review ICV/CCV, EPA reference sample results and other criteria prior to determining if the data should be qualified as estimated (J if > IDL; UJ if < IDL) or unusable (R).

4. If the ICV/CCV %R falls outside the acceptable limits, use professional judgement to qualify associated samples. If possible, indicate the bias in the review. The following guidelines may assist the reviewer in qualifying the data:

   - If the ICV/CCV %R is significantly greater than the upper control limit (UCL), qualify associated results > IDL as unusable (R); results < IDL are acceptable.

   - If the ICV/CCV %R is significantly lower than the lower control limit (LCL), qualify all associated data as unuseable (R)

   - If the ICV/CCV %R are outside the acceptable limits, yet relatively close to the limits, qualify associated results as estimated (J if > IDL; UJ if < IDL and the %R is below the LCL).

F. BLANKS

Evaluation

1. Review the raw data for all blanks and verify the results were accurately reported.

2. No contaminants should be found in the blanks (i.e. all blank results should be < IDL).

3. Positive blank results must not be corrected by subtracting off the blank value from the sample results.

Action

1. Sample results ≥ IDL but less than 5 times the maximum concentration found in any blank should be qualified as not detected (U).

2. Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine if the sample data is biased in any way.

G. LABORATORY CONTROL SAMPLES (EPA REFERENCE SAMPLES)

Evaluation

1. The laboratory control sample (LCS) serves as a monitor of the overall performance of the analysis including all preparation steps. Review the LCS and verify that the results fall within the control limits required. (If no limits noted, use 80-120% for the control limits.)

2. Check the raw data to verify the reported recoveries. Recalculate one or more recoveries (%R) using the following equation:

$$\text{LCS } \%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Action

1. If results are < IDL and the LCS recovery is above the UCL, the data are acceptable.

2. If the LCS recovery for any analyte falls within the range of 50-LCL, or > UCL, qualify results > IDL as estimated (J).

3. If results are < IDL and the LCS recovery falls within the range of 50-LCL, qualify the affected results as estimated (UJ).

4. If the LCS recovery results are < 50%, qualify the data for the affected analytes as unusable (R).

5. If a LCS was required and not run, use professional judgement to determine whether the data is estimated (J) or unusable (R).

H. LABORATORY DUPLICATES

Evaluation

1. Review the duplicate data and verify that the results fall within the criteria required. If no criteria exist, use the limits of 20% for the Relative Percent Difference (RPD) or ± IDL if results are < 5x IDL (35 RPD or ± 2x IDL if > 5x IDL for soils).

2. Review the data and verify that the duplicate analysis was <u>not</u> performed on a field blank.

3. Check the raw data and recalculate one or more RPD using the following:

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

where, S = Original sample value
D = Duplicate sample value.

Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same sample matrix as estimated (J).

2. If the field blank was used for duplicate analysis, carefully evaluate all other quality control data and use professional judgement in evaluating the data.

I. MATRIX SPIKES

Evaluation

1. Review the matrix spike data and verify that the results fall within the limits specified. If no criteria exists, use the limits of 75-125% for both water and soil matrices.

2. Check the raw data and recalculate the % Recovery of at least 10% of the data. Use the following equation to verify that the results were calculated correctly:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Review the data and verify that the field blank was not used for the spike analysis.

Action

1. If the spike recovery is > 125% (or > UCL stated in the procedure) and the sample results are < IDL, the data is acceptable for use.

2. If the spike recovery is > 125% or < 75% (> UCL or < LCL stated in the procedure) and the sample results are > IDL, qualify the data for these samples as estimated (J).

3. If the spike recovery falls within the range of 30-74% (30-LCL) and the sample results are < IDL, qualify the data for these samples as estimated (UJ).

4. If any spike recovery results are < 30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

5. In cases where more than one spike analysis was performed and one or more %R results were outside acceptable ranges, use professional judgement when qualifying the data. Matrix spikes should be performed on each sample matrix. Qualify data of similar matrix as the spiked sample if different matrices in the data package.

6. If the field blank was used for the spike analysis, carefully evaluate all other quality control criteria and use professional judgement in evaluating the data.

J. SAMPLE RESULT VERIFICATION

Evaluation

1. Examine the raw data to verify the analyte quantitation was calculated as stated in the procedure. Re-calculate a minimum of 10% of the sample results to verify results were calculated correctly.

2. Examine the raw data for anomalies such as baseline drift, negative absorbances (indicative of interfences), omissions, etc.

3. Verify there are no transcription or reduction errors (dilutions, percent solids, sample weights) on at least 10% of the samples.

4. Verify that all results fall within the calibrated range.

Action

1. If any discrepancies are found and can be corrected, make the corrections and note. Increase the level of quality control if necessary. If the discrepancy cannot be resolved by the reviewer, contact the data management coordinator. The laboratory or appropriate agency will be contacted and the information necessary to complete the evaluation will be requested. The project manager should be informed of the delay in the validation process. If any discrepancy remains unresolved, use professional judgement in qualifying the data.

K. FIELD DUPLICATES

Evaluation

1. Field duplicate samples measure field and lab precision and therefore the results may have more variability than lab duplicates. Review the field duplicate data and calculate the RPD. No criteria exists for evaluating the data.

Action

1. If the RPD is > 50%, note in the narrative. Use professional judgement in qualifying any data. Review the raw data to verify that no reduction errors exist. This is to verify that the discrepancy is due to sampling techniques (sampling, preservation,

filtering, etc.) rather than laboratory error. Make notation of the discrepancy in the narrative.

L. OTHER (as specified in the procedure)

Evaluation

1. Review the procedure for any other quality control criteria not covered under the above sections. Examples of additional information required may be: ICP interference check samples, special AA quality control checks, distilled/digested blanks and standards, standardization, specific methodology requests for different concentration levels, etc..

Action

1. If specific quality control criteria were required and not followed, the project manager should be informed.

2. If the data was outside the acceptable ranges, use professional judgement in qualifying the data. Clearly identify your action and justification of the action in the narrative.

M. OVERALL ASSESSMENT FOR A CASE

1. It is appropriate for the reviewer to make professional judgements and express concerns and comments on the validity of the overall data package. This is particularly appropriate when several QC criteria are outside specifications. It is the reviewers responsibility to thoroughly document and explain all data validation qualifiers added to the data.

2. The following is a summary of data validation qualifier definitions which can be used in evaluating the data:

   U The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

   J The associated value is an estimated quantity due to quality control criteria not being met.

   R The data are unusable. The analyte may or may not be present.

   UJ The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

5. Once the data has been evaluated against all of the points described in part 4, the evaluator can prepare the data validation narrative.

The data validation narrative should be a summary of the data quality indicating any problems or deficiencies found with the data set. If

possible, on data qualified as estimated, note if the data is biased high or low. It is easiest to discuss each analyte separately. Refer to Figure 2 for the form to be used when writing the data validation narrative.

6. Record any data validation qualifiers (DVQ) to the left of the results on the analytical report. The laboratory qualifiers (LQ) will usually recorded to the right of the result by the laboratory who performed the analytical work. A copy of the lab qualifier definitions should be supplied with the data. Data entry people need to be able to distinguish between DVQ and LQ, so note location of qualifiers if located other than above.

7. Record the sample ID and date sampled as noted on the Chain of Custody on the analytical report.

8. Hilight POSITIVE-HIT data with a hilighter.

9. Stamp all of the analytical reports "VALIDATED". A "VALIDATED" stamp can be obtained from the data management coordinator.

10. Sign and date the Data Validation Request Form.

11. A quality control review needs to be performed on the validated data package. The entire validated data package (including raw data, evaluator's notes and narrative) should be submitted to the QC reviewer for a final review.

Quality Control Review:

1. The purpose of this quality control review is to verify that the data were validated according to the guidelines above, that all data validation qualifiers were added and also that the narrative is complete.

2. Review the validated data package for completeness. The data package should contain the following:

   - Data Validation Request Form
   - Inorganic Data Validation Narrative
   - Validated Analytical Results

3. Verify that results exist for all samples and all analytes requested in the data package.

4. Review the data validation narrative with the validated results for consistency, i.e., if the narrative stated that a particular analyte was qualified, verify that the qualifier has been recorded with the associated data. Check approximately 10% of the qualifiers. If discrepancies are found, increase the level of QC checks.

5. Review the narrative; check that the data has been evaluated according to the quality control criteria specified in the QAPP.

6. Verify that all positive-hit data have been hilighted and that the sample descriptions and sampling dates have been recorded on every result page.

7. Verify that the reviewer has signed and dated the validated data package.

8. If no discrepancies exist, sign and date the Data Validation Request Form. If the data package requires clarification, review the raw data and/or obtain clarification from the reviewer.

9. Deliver the final validated data package to the Data Management Coordinator when the quality control review is completed.

10. Recycle any raw data. Raw data is not submitted with the final validated data package.

INORGANICS
DATA VALIDATION NARRATIVE

Site Name _____ Project # _____

SMO Case # (SAS #) _____ #Samples/Matrix _____

Laboratory _____ Hours for Review _____

SOW # _____ EPA Validation Guidelines # _____

Sample Numbers _____

Validated By _____ Date _____

Reviewed By _____ Date _____

SUMMARY OF REVIEW:

CAW/dlk/KJD
[dlk-601-22]
60721-MD