July 20, 1990

CHEROKEE COUNTY, KANSAS, CERCLA SITE
BAXTER SPRINGS/TREECE SUBSITES

STANDARD OPERATING PROCEDURES
FOR
REMEDIAL INVESTIGATION/FEASIBILITY
STUDY AND RISK ASSESSMENT

DAMES & MOORE

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INTRODUCTION

This document presents Standard Operating Procedures (SOPs) for environmental sampling conducted by Dames & Moore. The SOPs contained in this document are:

SOP-1 Drilling Methods and Sampling Procedures
SOP-2 Monitoring Well Design, Installation and Development
SOP-3 Ground-Water Sampling
SOP-4 Handling and Disposal of Field Program Residuals
SOP-5 X-Ray Fluorescence Spectrometer Use and Calibration
SOP-6 Batch Leach and Column Testing
SOP-7 Solid Substance Sampling Methods
SOP-8 Random Sampling for Area Composites
SOP-9 Sample Preservation, Handling, Labeling, Shipping, and Documentation
SOP-10 Control of Measuring and Test Equipment
SOP-11 Surface-water and Streambed Sediment Sampling
SOP-12 Stream Flow Measurement

The procedures have been developed from governmental and industrial guidance documents, Dames & Moore field experience, and the standard methods and procedures as practiced throughout the industry. Referenced documents include:


Dames & Moore (1975), Manual of Technical Practice for Subsurface Investigation, Dames & Moore, Los Angeles, California.


New Jersey Department of Environmental Protection (1985), Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, DWM, HSMA, Trenton, New Jersey.
These SOPs provide only general methods and procedures which, by necessity, may be changed to suit a particular phase of the project. Preparation of the SOPs does not represent a firm commitment to utilize each and every procedure in the Step II RI program. The data needs assessment and Sampling and Analysis Plan (SAP) (with laboratory plan) will determine the scope of Step II.

The SOPs are intended to augment the SAP. Detailed procedures may be described in the SAP which go beyond those outlined in the SOP's as a result of specific requirements of the project. In any conflict between a procedure specified in the SOP and one specified in the SAP, the procedure specified in the SAP will rule.
STANDARD OPERATING PROCEDURE NO. 1
DRILLING METHODS AND SAMPLING PROCEDURES

This Standard Operating Procedure describes the techniques to be implemented for soil and rock coring, lithologic logging, and sample compositing. Equipment necessary for the implementation of these procedures is also summarized.

DRILLING METHODS

Several alternative methods for the collection of subsurface soil samples are described below. Reference to EPA, 1986a; EPA, 1987; Johnson, 1986; and appropriate ASTM guidance should be made for more detailed descriptions of the procedures presented below and other alternative methods.

For each drilling method, a boring log, based upon continuous, interval, or grab sampling, will be prepared in accordance with the lithologic logging procedures outlined in a later section of this SOP.

Where appropriate, temporary surface casing may be installed to preserve the integrity of the borehole and/or to prevent migration of ground water through the borehole. Sealing of the surface casing will be specified based upon site geology and aquifer characteristics.

Field residuals will be handled in accordance with the procedures outlined in SOP-4, Handling and Disposal of Field Program Residuals.

Hollow-Stem Auger Method

Hollow-stem auger methods will be used in cases where shallow penetrative work is to be conducted in unconsolidated...
sediments. The hollow-stem auger drilling method utilizes hollow-stem augers with lock-bolt joined 5-foot sections, drill rod, plug, and bit assembly. Auger diameter will be selected based upon auger availability, drilling rig capability, anticipated boring depth, sampler type, and intended boring use.

Drilling fluids will be used only in cases where required to maintain borehole integrity. For example, if the drill rod assembly within the auger should become bound by flowing sands, potable water will be pumped to the top of the drill rod assembly and the assembly will be raised and lowered through the augers until they become unbound. The creation of a positive hydraulic gradient between the inside and outside of the augers could allow the completion of a borehole in flowing sands. The source, time and duration of use, and rate of flow of drilling water will be recorded on the boring log. A sample of the water used in drilling may be collected to evaluate the potential for contaminant introduction through the use of water in the drilling process.

Rotary Drilling Method

Rotary drilling methods will be used in cases where deep penetrative work is to be conducted in unconsolidated sediments or where shallow penetrative work is to be conducted in competent bedrock or in deposits containing cobbles or boulders.

Where rotary drilling methods are specified, drill pipe fitted with a roller, drag bit, or drive shoe will be used. If rock coring is specified, the rotary drill rig will be fitted with wireline coring equipment of NX or larger size. The type and diameter of the bit or shoe will be specified based upon availability, formation characteristics, drilling rig capability, anticipated boring depth, sampler type, and intended boring use (i.e., for monitoring well installation).
Grab samples of cuttings are obtained by use of a strainer or other suitable means to catch cuttings as they exit the boring annulus. Cuttings are rinsed in water and are used to assess lithologic characteristics, and formation depths and thicknesses.

Potable water or a low-solids drilling mud will be the only drilling fluid used in direct rotary applications. The rate of necessary drilling water make-up will be recorded on the boring log. A sample of the water used in drilling may be collected to evaluate the potential for contaminant introduction through the use of water in the drilling process.

Air-Rotary Method

Air rotary methods may be used in cases where deep or shallow penetrative work is to be conducted in moderately hard to soft rock. Where air rotary methods are specified, drill pipe fitted with a roller, drag or button bit will be used. The type and diameter of the bit will be specified based on availability, formation characteristics, drilling rig capability, anticipated boring depth, sampler type, and intended boring use (i.e., for monitoring well installation).

Grab samples of cuttings are obtained by use of a strainer or other suitable means to catch cuttings as they exit the boring annulus. Cuttings are rinsed in water and are used to assess lithologic characteristics, and formation depths and thicknesses.

Water and/or foam may be added to the air stream to allow for enhanced circulation and cuttings recovery. Only potable water will be used. Foam manufacturer and composition will be
noted on the boring log. The condition of the compressor and quality of the air stream will be evaluated by field observation and recorded on the boring log.

Downhole Hammer Method

Downhole hammer methods may be used in cases where deep or shallow penetrative work is to be conducted in very hard to moderately hard rock.

Grab samples of cuttings are obtained by use of a strainer or other suitable means to catch cuttings as they exit the boring annulus. Cuttings are rinsed in water and are used to assess lithologic characteristics, and formation depths and thicknesses.

Water and/or foam may be added to the air stream to allow for enhanced circulation and cuttings recovery. Only potable water will be used. The condition of the compressor and quality of the air stream will be evaluated by field observation and recorded on the boring log.

SUBSURFACE SAMPLING METHODS

Drive-sampling methods will be used in the execution of hollow-stem auger and direct rotary drilling work in unconsolidated deposits. Interval and some continuous sampling of unconsolidated material can be performed with drive sampling devices such as split spoon or Dames & Moore Type U samplers (Figure SOP-1.1). The drive-sampling device will be driven 24 inches or until refusal by a 140 pound hammer free-falling 30 inches. Samples recovered from the standard split spoon are altered by the driving action and are considered "disturbed" samples (for the purpose of geotechnical evaluation). The Dames &
Moore Type U sampler recovers relatively undisturbed samples in brass or stainless steel liners (see thin wall sampling tube methods).

The sampler is removed from the borehole after driving and split open. The sample is then logged and removed and the sampler is decontaminated before reassembly. If the standard split spoon samples are to be retained for future reference, they will be placed in sealable plastic bags and labeled according to standard procedures.

Thin-wall sampling tube methods (Dames & Moore Type U Sampler) will be used where undisturbed samples of fine-grained material are to be obtained for saturated vertical hydraulic conductivity or other geotechnical analysis. Where a thin wall sampler (i.e., Shelby tube) is to be used, the sample may be obtained by pushed tube, piston sampler, Denison sampler, or Pitcher sampler methods.

In the case of the pushed-tube method, the tube will be attached to the Dames & Moore Type U sampler and pushed, in one continuous motion, into the material to be sampled. If the sample is to be retained for future reference or analysis, it will be trimmed, logged, and placed in sealable plastic bags. The sample should then be placed "deep" end down in a plastic sample container, sealed, and labeled. Once the tube is received at the laboratory, slough material will be removed from the tube and the actual sample length will be measured.

The bottom and top of the tube will be indicated on the tube in indelible marker. The following information will be noted on the side or top of the tube:

- Project Number and Name
- Sampling Date and Time
- Boring and Sample Numbers
Sample Depth

The tube will then be transported to the laboratory. Efforts will be made to keep the tube vertical and avoid disturbance of the tube at all times.

CME continuous sampling system methods may be specified where continuous soil cores are to be recovered by hollow-stem auger drilling methods. The continuous sampling system utilizes a core barrel which recovers a soil core from the interval drilled. The core barrel is recovered after each interval is drilled.

Representative samples of the soil cores can be placed in 8-ounce glass jars for future analysis. The entire length of the soil cores can be placed in plastic-lined core boxes should it be necessary to retain them.

Rock coring methods may be specified. Rock coring will be conducted with an NX or other available core barrel. Depending on subsurface conditions, the circulation media will either be water, air, or mud.

The cores will be removed from the core barrel, washed of drilling-related sediment, and placed in a properly labeled core box (Figure SOP-1.2). The cores will then be logged in accordance with the lithologic logging criteria for rock presented below.

During rotary and downhole hammer drilling, grab samples of drill cuttings will be collected for examination by use of a strainer or other suitable means.
### TOP VIEW (CLOSED)

<table>
<thead>
<tr>
<th>BORING NUMBER</th>
<th>RUN NUMBER</th>
<th>DEPTH FROM TO</th>
<th>INCHES RECO'D</th>
<th>PERCENT RECO'D</th>
<th>&lt; 4&quot; RECO'D</th>
<th>REQ'D</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

### TOP VIEW (OPEN)

#### END VIEW

1682-019
Springfield, Illinois
Box 2 of 4

DAMES & MOORE

Arrangement of Rock Core and Labeling of Rock Core Boxes

Figure SOP-1.2
Samples For Laboratory Analysis

Sampling procedures to be followed in the collection of all lithologic samples for laboratory analysis are as follows:

- Prepare field record sheets and record relevant data in logbook.
- All equipment or material to be used for sampling will be kept from contact with the ground or other potentially contaminated material by the use of plastic sheets. Any sampling equipment that comes in contact with the ground or other potentially contaminated material will be suitably decontaminated.
- Use drilling methods discussed in this SOP to reach the desired sampling point.
- Decontaminate sampling equipment.
- Collect sample using appropriate sampling method described in this SOP.
- Make appropriate entries on lithologic log.
- Place sample in appropriate sample container.
- Enter appropriate information on sample container label and mount label on sample bottle.
- Place the properly labeled sample container in an appropriate carrying container at required temperature.
- Either discard or decontaminate all items which contact the sample such as scoop, gloves, and beakers before proceeding to the next sampling location.
- Enter information pertaining to the sample to the chain-of-custody record, analysis request form, and field logbook.

Sample compositing will be conducted in accordance with the mixing bowl compositing procedures described in EPA (1983). The procedures are as follows:
A stainless steel mixing bowl and stainless steel scoop are decontaminated as described in this SOP.

The sample fractions are placed in equal volumes into the mixing bowl.

The resultant composite sample is stirred until homogeneous (it may be necessary to break up soil aggregates with the scoop or a decontaminated stainless steel knife or spoon).

The mixed sample is then spread evenly in the bottom of the bowl and quartered.

Equal amounts of sample are taken from each quarter to fill each of the sample bottles.

LITHOLOGIC LOGGING

The lithologic logging procedures presented below are modified for environmental sampling from the 1975 Dames & Moore Manual of Technical Practice for Subsurface Investigations.

Soil Boring Logs

The standard Dames & Moore boring log form will be used (Figures SOP-1.3A, SOP-1.3B). The procedures which will be followed in completing the boring logs are outlined below:

On the first page of the field log for each boring, the identifying information at the top and left margin of the boring log will be filled in completely. The following information will be included:

- Job Number
- Owner’s Name
- Job Location
- Boring Number
- Drilling Contractor
- Operator’s Name
## EXAMPLE BORING LOG

**DAMES & MOORE**

**LOCATION OF BORING**

**Location:** Bridgewood, NJ

**Area:**
- **T2E**
- **R23 N**
- **Sec 20**
- **Towhee Road**

**Date:** 11/20

**Surface Condition:**
- Corner of grass-covered pasture, gently sloping to southeast

**Drilling Method:** 4" auger and rotary well with 3½" tri-cone bit
- **Mob. B-61 rig**
- **Sampling Method:** U, TW, SS, ST, NQ

**Drilling Details:**
- **U" = 340 lb hammer, 24" drop**
- **SS = 180 lb hammer, 30" drop**

**Water Level:**
- **Time:** 1 PM
- **Time:** 1:00 PM

**Elevation:**
- **Depth:** 18.0

**Log Sample Data**

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Depth</th>
<th>Material</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>U-1</td>
<td>15'</td>
<td>U-1</td>
<td>15'</td>
</tr>
<tr>
<td>TW</td>
<td>16</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>TW</td>
<td>18</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>CL</td>
<td>18</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>LT BR</td>
<td>15'</td>
<td>LT BR</td>
<td>15'</td>
</tr>
<tr>
<td>CL</td>
<td>18'</td>
<td>CL</td>
<td>18'</td>
</tr>
</tbody>
</table>

**Log Description**

- **U-1:**
  - Description: BZ ORG CL 51 - Tension (sp?)
  - Comments: Pull to 12"

- **TW:**
  - Description: Moat BR + GR CL 51
  - Comments: Lowess

- **ML:**
  - Description: DTW 2" at TW
  - Comments: Sample test

- **OK GR CL 51:**
  - Description: CO SP (Stiff to very stiff) 4:2
  - Comments: PSS 250 psf

- **CL:**
  - Description: GDS w/occ 9V
  - Comments: PSS 2000 psf

- **SP:**
  - Description: LT BR FI to MED 51 UH 51
  - Comments: PSS 1000 psf

**Additional Notes:**
- Wash w/mud
- Core and test drilling
- Could not be used (soil would be used for pavement cover)

**Drill Log:**

**Sample:**
- **LT BR FI to MED 51 UH 51**
- **1/1850**

**Other Details:**
- **15 ft of 5"**
- **casing installed**
- **after Sample 5**
- **Core is subm.**

**Figure:**

**Figure SOP-1.3A**
### EXAMPLE BORING LOG (SHEET 2)

- **Location:** Braidwood, Ill.
- **Job No.:** 5643-03d
- **Client:** Comm. Edison

#### Datum

<table>
<thead>
<tr>
<th>No.</th>
<th>Datum</th>
<th>Elevation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1000</td>
<td>0.0</td>
</tr>
<tr>
<td>2</td>
<td>1001</td>
<td>1.0</td>
</tr>
<tr>
<td>3</td>
<td>1002</td>
<td>2.0</td>
</tr>
</tbody>
</table>

#### Sampling Method

- **Method:** DMS Co
- **Date:** 3/15/73

#### Drilling Method

- **Method:** Casing with a 30'-long pipe

#### Surface Conditions

- **Description:** Casing ignition

#### Geology

- **Material:** Sandstone
- **Depth:** 27.4' to 32.7'

#### Water Level

- **Depth:** 32.5' to 35.4'
- **Pressure:** Fresh
- **Water Level:** 37.4'

#### Boring Completion

- **Date:** 11/6/73
- **Depth:** 39.4 feet

#### Additional Notes

- **Casing used to a depth of 15 feet**

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*FIGURE SOP-1.3B*
- Rig number, if there is more than one rig.
- Name of Person Conducting the Logging
- Elevation and Reference Datum
- Complete and accurate location sketch with dimensions to landmarks. Important features such as road intersections, corners of large buildings, or other man-made structures are best used. A north arrow will be included.
- Water levels will be recorded each day prior to drilling start-up and at other times as appropriate. The casing depth and date and time of measurement will be noted for each water level reading. Drilling mud use will be noted. When using electric water level indicators, the depth marks should be checked against an accurate tape measure. Water levels will be referenced to ground surface, unless otherwise noted.
- The exact drilling and sampling methods used will be indicated, as well as the bit type and size, drill rod size, hammer weight and drop, types of samplers, and the rig type.
- Surface conditions will be noted, including vegetation or ground cover and land use, general topography (rolling, level, etc.), and physical features (hillside, river bottom, etc.).
- Sheet (Page) Number
- The time and date of the commencement and conclusion of the boring. (All down time and end/start of shifts should be noted at the corresponding depth).

Each succeeding page of the field log should contain at a minimum:
- Job Number
- Client Name
- Boring Number
- Sheet Number
- Any Water Level Measurements
The following standard completion note will be included at the bottom of the last page of each boring log:

- Boring Completed at (Depth) Feet on (Date)
- Casing Used to a Depth of (Depth) Feet on (Date)
- Ground-Water Level Recorded at (Depth) Feet on (Date)
- The casing size and depth (all depth data will be recorded in decimal feet)
- Well construction details
- Well development details

The left side of the boring log will be filled out accurately and completely for each sample and sampling attempt. The following information will be included:

- The sampler type. When using the Dames & Moore Type U sampler, the type of bit (U-1, U-2, or U-3) will be shown. The abbreviations for other sampler types are TW for thin-wall, P for piston, ST for Shelby Tube, and SS for standard split spoon. These abbreviations, or notations as to other sampler types, will be used in the "sampler type" column.
- The inches driven and recovered for each sample attempt.
- The depth of casing for each sample attempt.
- The sample number and depth. The sample depth is always measured to the top of the sample. A sample attempt where no sample is recovered will not be numbered, but the fact that a sampling attempt was made must be noted.
- The blow count for each 6-inch interval will be recorded opposite the appropriate interval on the depth scale.
- When geotechnical sampling is conducted, the number of rings of sample retained for each sample. When more than the normal six rings are retained, this will be noted by designating the samples as "A" and "B" and recording the number of rings for each.
The sampling attempts will be marked in the "depth in feet" column as follows:

A vertical line will be drawn through the sampling interval.

For the Dames & Moore Type U, thin-wall, piston, Shelby tube, or other low-disturbance samples, the depth interval corresponding to the sample will be blacked-in completely.

A sample which cannot be used for geotechnical testing or chemical analysis will be marked with an "X".

For disturbed samples collected for chemical analysis, the depth interval corresponding to the sample will be half blacked-in along a slanted line.

A "missed" sample will be marked with an open box.

The right side of the log will contain a detailed description of the soil strata encountered and all pertinent information regarding drilling operations and estimated soil and ground-water properties. It is helpful to draw a vertical line on the right side of the log to separate the soil descriptions from the drilling information.

- Soil will be classified according to the Unified Soil Classification System (Figure SOP-1.4). The Unified Soil Classification symbol (ML, CH, SP, etc.) will be written in the "soil graph" column within the depth interval corresponding to the appropriate soil type. The soil descriptions will be neat and orderly.

- Abrupt soil changes will be marked by a solid horizontal line at the appropriate depth in the "soil graph" column. Gradational soil changes will be marked by a dashed line. Diagonal lines should not be used. (Field personnel are best qualified to estimate the depth of changes. This task will not be delegated to office personnel, who have not observed the drilling operation.)

- Soil color descriptions will be consistent with the Munsell Soil Color Chart.

- Abbreviations will be used on the logs to save space for editing purposes.
### Major Divisions

<table>
<thead>
<tr>
<th>Gravel and Gravelly Soils</th>
<th>Clean Gravels (Little or No Fines)</th>
<th>GW</th>
<th>Well-graded Gravels, Gravel-Sand Mixtures, Little or No Fines</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean Gravels with Fines (Appreciable Amount of Fines)</td>
<td>GM</td>
<td>Silty Gravels, Gravel-Sand-Silt Mixtures</td>
<td></td>
</tr>
<tr>
<td>Clean Gravels (Little or No Fines)</td>
<td>GC</td>
<td>Clayey Gravels, Gravel-Sand-Clay Mixtures</td>
<td></td>
</tr>
<tr>
<td>Sand and Sandy Soils</td>
<td>Clean Sand (Little or No Fines)</td>
<td>SW</td>
<td>Well-graded Sands, Gravelly Sands, Little or No Fines</td>
</tr>
<tr>
<td>Sand with Fines (Appreciable Amount of Fines)</td>
<td>SP</td>
<td>Poorly-graded Sands, Gravelly Sands, Little or No Fines</td>
<td></td>
</tr>
<tr>
<td>More Than 50% of Coarse Fraction Retained on No. 4 Sieve</td>
<td>More than 50% of Coarse Fraction Passing No. 4 Sieve</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coarse Grained Soils</td>
<td>Silts and Clays</td>
<td>Liquid Limit Less Than 50</td>
<td>ML</td>
</tr>
<tr>
<td>More Than 50% of Material is Larger Than No. 200 Sieve Size</td>
<td>Silts and Clays</td>
<td>Liquid Limit Greater Than 50</td>
<td>CH</td>
</tr>
<tr>
<td>Fine Grained Soils</td>
<td>Organic Silts and Organic Silty Clays of Low Plasticity</td>
<td>OL</td>
<td>Organic Silts and Organic Silty Clays of Low Plasticity</td>
</tr>
<tr>
<td>More Than 50% of Material is Smaller Than No. 200 Sieve Size</td>
<td>Organic Clays of Medium to High Plasticity, Organic Silts</td>
<td>OH</td>
<td>Organic Clays of Medium to High Plasticity, Organic Silts</td>
</tr>
<tr>
<td>Highly Organic Soils</td>
<td>Peat, Humus, Swamp Soils with High Organic Contents</td>
<td>PT</td>
<td></td>
</tr>
</tbody>
</table>

**Note:** Dual symbols are used to indicate borderline soil classifications.

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**Unified Soil Classification System**

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*Dames & Moore*
Comments on the field log are extremely important. Some important aspects of the drilling operation that will be recorded follow:

The organic content of the soil and the depth of topsoil and roots will be noted.

Any sudden change in the speed, sound, or penetration rate of the drill rig will be noted.

If sampling is not continuous, drill cuttings will be used to complete the log of the stratigraphic section.

Loss or gain of drilling fluid, change in fluid color, or change in fluid consistency will be recorded.

Any sample that is suspected of being disturbed, contaminated, or chemically or physically altered during the drilling process will be noted.

Rock Coring Logs

The purpose of a rock coring log is to present all the relevant information obtained during drilling and from examination of the cores. The standard Dames & Moore soil boring logs can be adapted to a rock coring log. In some cases, more specialized rock coring log sheets may be used on a job. Regardless of the job, the same basic information should be included on all rock coring logs. The following discussion outlines the information to be included on Dames & Moore log forms that have been adapted for rock coring. If another log form is used, the following information will also be included on the form in an organized and systematic manner.

The identifying information on the first page of each boring log will be filled out completely in a manner consistent with that described previously for soil sampling above.
Each succeeding page of the field log will contain at a minimum:

- Name of Field Personnel
- Job Number
- Client Name
- Boring Number
- Sheet Number
- Any Water Level Data

The following standard completion note will be included at the bottom of the last page of each boring log:

- Boring Completed at (Depth) Feet on (Date)
- Casing Used to a Depth of (Depth) Feet on (Date)
- Ground Water Level Recorded at (Depth) Feet on (Date)
- The size and depth of casing
- Well construction details
- Well development details

The left side of the log will be filled out completely, including the following information:

- At the depth rock is encountered, the column headings will be relabeled as shown in Figure SOP-1.2B.

- All core runs will be numbered consecutively each time the bit is advanced and the sample is removed from the hole. A run with no recovery will be numbered.

- The total length the bit was advanced (run length) will be recorded.

- The length of the core recovered during the run will be recorded. It is usually advantageous to measure the recovery in the core barrel prior to removal. If for some reason this cannot be done, extreme care will be taken in measuring the total length of run in that broken and fractured core often appears much longer than its true length.

- The percent recovery will be calculated and recorded for each core run. The percent recovery will be computed by dividing the core recovery by the length of the core run, and multiplying by 100.
Unrecovered core from one run that is recovered on the succeeding run will be included in the first run for the sake of recovery and Rock Quality Designation (RQD). Such occurrences should be noted on the boring log.

- The RQD will be calculated and recorded for each run.

- The RQD is calculated by measuring the total length of all sound pieces of core 4 or more inches in length, dividing this total length by the length of the core run, and multiplying the result by 100. Both the total length of all core pieces 4 inches or longer and the percent RQD are recorded on the log.

- When considering breaks in the core, the following procedure should be followed:

  Mechanical breaks, whether intentional or unintentional, are overlooked and treated as sound rock.

  Pieces of core 4 or more inches in length with a vertical fracture or joint are counted as sound rock in the modified core recovery.

  Angle fractures are measured to the center of the angle.

Qualitative terms have been developed to describe rock quality as computed by the RQD and are listed below.

<table>
<thead>
<tr>
<th>Percent</th>
<th>Rock Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-25</td>
<td>Very Poor</td>
</tr>
<tr>
<td>25-50</td>
<td>Poor</td>
</tr>
<tr>
<td>50-75</td>
<td>Fair</td>
</tr>
<tr>
<td>75-90</td>
<td>Good</td>
</tr>
<tr>
<td>90-100</td>
<td>Excellent</td>
</tr>
</tbody>
</table>

- On many jobs, it is helpful to record the penetration rate in minutes per foot. The penetration rate is useful in comparing relative "drillability". The penetration rate is affected by rock hardness, density, drilling rpm, pull-down pressure, circulating water or air pressure, and drill bitwear and design. Thus, it is important to note all changes in drilling procedure on the log.
On some jobs, it is advantageous to calculate the fractures per foot and record it on the logs. The core is divided into intervals based on the predominant frequency of fracture spacing. The fractures in each interval are counted and the total number is divided by the length of the interval. The limits of each interval are not made to correspond to a particular footage or to the ends of each run. Preferably, a distinction is made between natural fractures and fractures due to drilling and handling (mechanical fractures). Where this is possible, only the natural fractures are included on the log. In some cases, such as deep holes in fresh rock, where it is not possible to distinguish natural fractures, all fractures are included. The method that is adopted should be specified on the log.

The run interval and run number should be displayed graphically in the "depth in feet" column. The depth of the hole should be determined after each run. Field personnel should constantly be aware of the depth and should check the depth measurement made by the driller.

The right side of the boring log should contain a detailed description of the rock units and all pertinent information regarding drilling operations. The following information should be included:

- The system of rock classification will be determined by the Project Manager or Principal Investigator before field operations commence. There are many good, similar systems of rock classification in print; however, it is recommended that Travis (1955) be used.

- Rock descriptions should be listed in the following order:
  
  Lithology
  Color as determined by Munsell Soil Color Chart
  Grain size or crystallinity
  Bedding
  Other observations such as vug percentage, mineralization, fossil content, or weathering.

- The degree of weathering of the core is estimated as detailed below. This classification is applicable to rock materials that are relatively strong in the unweathered state. For materials such as shale, which are weak in the unweathered
state, it is often difficult to assess the degree of weathering. In these cases, a simplified classification using only the terms "fresh", "partly weathered", and "extremely weathered", is used. Where "fresh" and "extremely weathered" have the same definitions as that below, "partly weathered" is used for anything intermediate.

### Degree of Weathering

<table>
<thead>
<tr>
<th>Term</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh</td>
<td>The rock shows no discoloration, loss of strength, or any other effect of weathering.</td>
</tr>
<tr>
<td>Slightly Weathered</td>
<td>The rock is slightly discolored, but not noticeably lower in strength than the fresh rock.</td>
</tr>
<tr>
<td>Moderately Weathered</td>
<td>The rock is discolored and noticeably weakened, but 2-inch diameter drill cores cannot usually be broken up by hand, across the rock fabric.</td>
</tr>
<tr>
<td>Highly Weathered</td>
<td>The rock is usually discolored and weakened to such an extent that 2-inch diameter cores can be broken up readily by hand, across the rock fabric. Wet strength is usually much lower than dry strength.</td>
</tr>
<tr>
<td>Extremely Weathered</td>
<td>The rock is discolored and is entirely changed to a soil, but the original fabric of the rock is mostly preserved. The properties of the soil depend upon the composition and structure of the parent rock.</td>
</tr>
</tbody>
</table>

Bedding characteristics are defined as presented below:

<table>
<thead>
<tr>
<th>Average Bed Thickness</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.001 Foot</td>
<td>Thinly Laminated</td>
</tr>
<tr>
<td>0.001 to 0.01 Foot</td>
<td>Laminated</td>
</tr>
<tr>
<td>0.01 to 0.1 Foot</td>
<td>Thin Bedded</td>
</tr>
<tr>
<td>0.1 to 1.0 Foot</td>
<td>Medium Bedded</td>
</tr>
<tr>
<td>&gt; 1.0 Foot</td>
<td>Thick Bedded</td>
</tr>
</tbody>
</table>
Quantification of descriptive terms is highly desirable. For instance "some small vugs" would better be described as "10% to 15% vugs, average size 0.1 cm ranging up to 0.5 cm."

Because rock defects severely modify the characteristics of the rock mass, the description of rock defects is a very important aspect of the coring log. Common defects requiring description are solution cavities, lava tubes, soil seams, shear zones, joints, and localized zones of weathering or hydrothermal alteration. If a planar defect (joint, shear zone, and so forth) is being described, its orientation relative to a plane perpendicular to the axis of the core is included. The condition of fracture of joint surfaces also is described (open, calcite-lined, closed, and so forth). If planar defects are filled with material of different composition than the parent rock, the type of filling material is described (calcite, kaolinite, sandy clay, chlorite, and so forth).

It is useful to construct a vertical line on the right side of the boring log to separate the rock description from the comments on drilling operation. The comments should include procedural drilling operations and any observations that might relate to the geologic, hydrologic, or engineering characteristics of the rock mass. Typical items to be included are:

Sequence of casing operations, including casing diameter and depth.

Depth at which noticeable changes in the operation of the drill occur. Includes sudden changes in pitch, rpm, vibration, pump pressure, or rate of penetration.

Bit drops or suspected voids.

Gain, loss, color change, or change in consistency of the circulating fluid.

Evidence of mineralization or oil or gas.

Use of drilling mud or other fluid activities.
Amount of cement used and depth interval of the set-up cement, if a hole has to be stabilized by cementing.

Explanation for the probable cause of core losses.

Field Photography

Color photographs will be taken of all full core boxes. A scale, preferably a rule extending the length of the core box marked in one-inch increments will be placed along the core box seam for the photograph. The open core box lid will be labeled as indicated in Figure SOP-1.1.

TEST BORING AND WELL ABANDONMENT

Monitoring wells and test holes could potentially provide contamination avenues for vertically segregated aquifers. The following guidelines will be adhered to in order to ensure that test holes and wells are abandoned properly. These guidelines adhere to Kansas State requirements for well abandonment.

ABANDONMENT OF TEST HOLES

Test borings penetrating water-table conditions will be abandoned by backfilling, from total depth to the ground surface, with Holeplug, Enviroplug, Volclay, or a cement-bentonite grout slurry.

- Holeplug and Enviroplug bentonite do not require mixing but are poured into the hole dry and charged with clean water.

- Volclay and cement-bentonite grout (herein termed cement grout) require mixing with clean, potable water. The mix should not contain more than 5 percent bentonite.
Backfilled holes will be checked after a period of time for settlement. If settlement has occurred, additional material will be added to bring the level to the surface.

Test holes that penetrate two or more aquifers (i.e., alluvial aquifer and bedrock aquifer) will be plugged with cement grout from total depth to ground surface.

ABANDONMENT OF MONITORING WELLS

Monitoring wells not intended for long-term use will be sealed to prevent contamination of aquifers. Materials used for abandonment (cement, bentonite, potable water) will be clean and free from contaminants. Upon completion of abandonment procedures (described below), exposed casing or well pipe will be cut off at a level which allows normal activity at the ground surface.

Unconfined Monitoring Wells

Wells completed in unconfined aquifers will be abandoned by grouting from total depth to ground surface, using cement grout or by backfilling with bentonite pellets (Enviroplug or similar). The casing or well pipe will not be pulled; cement grout will be pumped directly into the well itself or bentonite pellets will be poured down the well casing. Settlement of the grout or bentonite column is anticipated and additional cement grout or bentonite will be added, after a period of time, to bring the level to the surface.

Bedrock Aquifer Monitoring Wells

Wells completed in bedrock aquifers will be abandoned by plugging with cement grout from total depth to ground surface.
STANDARD OPERATING PROCEDURE NO. 2
MONITORING WELL DESIGN, INSTALLATION AND DEVELOPMENT

This Standard Operating Procedure (SOP) describes the techniques which will be implemented for construction and installation of ground-water monitoring wells at the Baxter Springs/Treece subsites. Materials and equipment necessary for the implementation of these procedures are also summarized.

MONITORING WELL DESIGN SPECIFICATIONS
Wells Completed in Unconsolidated Materials

Well design specifications for monitoring wells completed in unconsolidated materials are presented below. A schematic diagram of monitoring well design in unconsolidated materials is depicted in Figure 2.1.

Unless otherwise specified, monitoring wells will be constructed with 2-inch diameter, flush-threaded, Schedule 40, NFS certified, polyvinyl chloride (PVC) casing and factory-slotted screens. The length of the screened interval will depend on site-specific conditions. It is anticipated that competent bedrock will be encountered at depths less than 50 feet; therefore, the screened section would span the entire saturated thickness. A 2-foot long sediment sump (trap) will be constructed of blank casing at the bottom of the screened interval. Casing stickup above the ground surface will be approximately 1.8 feet.

Screen-slot size will be designed to retain approximately 90 percent of the filter pack material. The grain size of the filter pack will be specified to be between 4 and 6 times the effective grain size of the formation. The effective grain size of the formation will be established by conducting a sieve-analysis on the geologic unit with the finest grain size within the depth range to be screened. A curve generated with these data will be plotted
CONCRETE SURFACE
PAD 3'x3'x0.5'
2' SILT TRAP
(BLANK PVC CASING)

GROUND SURFACE

TOP OF 6" ø STEEL PROTECTIVE
CASING WITH LOCKING CAP

TOP OF WELL CASING
WITH CAP

Cement/Bentonite
(95%-5%)

2" ø FLUSH THREADED SCH. 40
NFS CERTIFIED PVC CASING

Bentonite Seal

10'-20 GRADED SILICA SAND
(COLORADO SILICA SAND OR
EQUIVALENT) FILTER PACK

10'(A) OR 5'(B) 0.01" OR 0.02"
FACTORY SLOTTED PVC SCREEN

10" (A) OR 5" (B) 0.01" OR 0.02"
FACTORY SLOTTED PVC SCREEN

BOTTOM CAP OR PLUG

TOTAL DEPTH

10" (A) OR 5" (B) 0.01" OR 0.02"
FACTORY SLOTTED PVC SCREEN

BENTONITE SEAL

10'-20 GRADED SILICA SAND
(COLORADO SILICA SAND OR
EQUIVALENT) FILTER PACK

BOTTOM CAP OR PLUG

TOTAL DEPTH

DAMES & MOORE

Construction Detail
Unconsolidated Monitoring Well

Figure SOP-2.1
on a grain size/percent passing diagram. Curves 4 and 6 times the formation particle size will be plotted as well. The filter pack material will be selected such that the grain size distribution curve for the filter pack falls between the 4 and 6 times formation size curves.

The filter pack material will be graded silica sand (Colorado Silica Sand or equivalent). The sandpack will extend from the bottom of the screened interval to about 2 feet above the top of the screen section.

Annular seals above the screened interval will be composed of bentonite pellets or powder. The seals will be 1 to 2 feet in thickness. Formation cuttings will not be used as backfill. The borehole annulus will be grouted with a cement-bentonite grout mix which will extend to within approximately 2 feet of the ground surface. The top two feet will be filled with a concrete mix used to make the concrete pad and secure the protective casing.

Protective steel casing with a locking cap will be placed over the PVC stickup with the top of the casing being approximately 2 feet above the ground surface. Protective casing will be secured by means of a padlock which will be installed at the time of protective casing installation. All protective casing will be cleaned prior to placement, free of extraneous openings and devoid of any asphaltic, bituminous, encrusting, and/or coating materials (except for paint or primer applied by the manufacturer). If the protective casing is to be painted, this will be accomplished prior to installation. Well designations will be painted or permanently attached on the protective casing for all wells. An elevated concrete pad will be poured into place around the well sloping away from and forming a surface seal around the well head.
Wells in Competent Bedrock

Well design specifications for monitoring wells completed in shallow and deep bedrock aquifers are similar. Steel surface casing will be sealed to the formation with cement-bentonite grout to prevent the infiltration of surface water. It is anticipated that the formation rock in both the shallow and deep aquifers is sufficiently competent to maintain the integrity of an open borehole. Thus, in the aquifer interval to be monitored, monitoring wells will be completed with an interval of open (uncased) borehole.

Monitoring wells in the shallow bedrock aquifer will be completed with an open borehole from the bottom of the surface casing to the total depth of the borehole. Monitoring wells in the deep bedrock aquifer will be cased through the Mississippian/Devonian aquitards with an open borehole in the deep aquifer.

If the competency of the formation rock is deemed insufficient to maintain an open-borehole completion, factory-slotted liner casing will be installed in the open-borehole interval. To prevent inter-aquifer communication, an approved packer will be installed on the liner casing above the interval to be monitored and the liner casing will be sealed to the formation and outer casing with grout. Schematic diagrams of shallow and deep bedrock monitoring well designs are shown in Figures 2.2 and 2.3, respectively.

Unless otherwise specified, bedrock monitoring wells will be constructed with steel surface casing that extends through overlying unconsolidated materials and weathered bedrock and a minimum of 5 feet into bedrock. The surface casing will be sealed into place with cement-bentonite grout to within two feet of the ground surface.
Construction Detail
Shallow Aquifer Monitoring Well

Figure SOP-2.2
CONCRETE SURFACE
PAD 3'x3'x0.5'

TOP OF PROTECTIVE CASING
WITH LOCKING CAP

TOP OF WELL PIPE
WITH CAP

GROUND SURFACE

UNCONSOLIDATED
MATERIAL

COMPETENT BEDROCK SURFACE

MISSISSIPPIAN CARBONATES
(SHALLOW AQUIFER)

MISSISSIPPIAN/DEVONIAN
AQUITARDS

ROUBIDOUX FORMATION
(DEEP AQUIFER)

STEEL SURFACE CASING
APPROXIMATE 8"-10" ø

APPROXIMATE 6"-8" ø
STEEL CASING

APPROXIMATE 6" ø
OPEN BOREHOLE

TOTAL DEPTH
Protective casing will be installed by extending the surface casing or by installing a separate steel casing to a height above the ground surface of approximately 2 feet. The protective casing will be fitted with a locking cap attached to the top of the surface casing. The cap will be secured by means of a padlock which will be installed at the time of well completion. All surface casing will be cleaned prior to placement and will be free of extraneous openings and devoid of any asphaltic, bituminous, encrusting, and/or coating materials (except for paint or primer applied by the manufacturer). If the protective casing is to be painted, this will be accomplished prior to installation. Well designations will be painted or permanently attached on the protective casing for all wells. An elevated concrete pad will be poured into place around the well.

MONITORING WELL INSTALLATION
Installation of Surface Casing

Surface casing will be installed to contain unconsolidated deposits above competent bedrock and to prevent the infiltration of surface water. Where surface casing is installed, the casing will be set by the selected method (spinning, driving, under-reaming, etc.) to a depth of 5 feet below the bedrock surface. A cement-bentonite grout slurry will be placed in the surface casing/borehole annulus from the bottom of the casing to the ground surface to ensure a proper seal. Once the grout has set, drilling will continue into the underlying bedrock.

Installation of Filter Pack and Bentonite Seal

Wells completed in unconsolidated deposits will require a filter pack placed around the screened interval and a bentonite seal placed above the filter pack. The filter pack and bentonite materials will be stored so as to avoid contact with precipitation.
or foreign material. Any sand or bentonite exposed to the ground surface will not be used in the completion of monitoring wells.

Where wells are constructed with total depths greater than 50 feet, the filter pack material will be placed in the borehole by use of a tremie pipe. The tremie pipe will be inserted to the bottom of the borehole. Sand will be poured into the tremie pipe and allowed to flow into the open annular space in the borehole. Where wells are constructed with total depths less than 50 feet, the filter pack material will be poured directly into the borehole. The top of the filter pack will be measured with a weighted measuring tape periodically to confirm the depth and distribution on the filter pack. In addition, the well will be bailed during placement to assist with the settlement of the filter pack.

When the hollow stem auger method of drilling is used, no more than a 3-foot interval of sand will be placed into the borehole at one time. After a 3-foot layer of sand has been installed, approximately 2.5 feet of the drilling steel casing or augers will be removed from the borehole. This sequence will be repeated until the sandpack is above the top of the screen. The depth to the top of the filter pack will be measured with a weighted measuring tape periodically to confirm the depth and even distribution of the filter pack.

After the filter pack is in place, the bentonite seal will be placed in the borehole by the following methods:

- If the depth for bentonite placement is above the water table, bentonite pellets will be poured slowly into the borehole until the required depth is measured.

- If the depth for bentonite placement is greater than 30 feet, then a bentonite slurry will be mixed and placed by pumping down a tremie pipe.
If the borehole contains thick fluid (drill cuttings and water), bailing the fluid from the cased hole prior to adding the sand or casing may facilitate well construction. Dilution of the fluid may be necessary. The volume and source of water added or removed will be recorded on the boring log.

Grout will be placed directly above the bentonite seal and will extend within two feet of the ground surface. This will inhibit water flow through the annular space between zones of the aquifer system and from the ground surface. In cases where the depth to the upper surface of the bentonite seal is less than 50 feet and ground water is not present above the bentonite seal, the bentonite cement slurry can be poured directly into the borehole. Where the depth to the upper bentonite surface is greater than 50 feet or ground water is present above the bentonite seal, the grout will be tremied into the borehole. The bottom of the tremie pipe will be positioned approximately 1 foot above the bentonite seal during grout installation.

When the hollow-stem auger method of drilling is used, no more than a 3-foot interval of grout will be placed into the borehole at one time. After a 3-foot layer of grout has been installed, approximately 2.5 feet of the drilling steel casing or augers will be removed from the borehole. This sequence will be repeated until the upper grout surface is 2 feet below the ground surface. The top of the grout will be measured with a weighted measuring tape periodically to confirm the depth and even distribution of the grout.

The grout will then be allowed to settle. If significant settlement of grout occurs, additional grout will be added to the prescribed depth of approximately 2 feet below ground surface prior to the installation of the protective casing and the concrete pad.

Installation of Protective Casing and Cement Pad
The protective casing will be placed over the casing stickup. A spacer will be temporarily installed so that the protective casing is 0.2 feet higher than the top of the well casing. The dimensions of the concrete pad will be 3 feet x 3 feet x 0.5 feet and will be sloped so as to provide positive drainage away from the steel casing.

**MONITORING WELL DEVELOPMENT**

Wells will be developed for a minimum of one-half hour. During development, the discharge water will be monitored for pH, specific conductance, and temperature. Development will continue until the field parameters stabilize. The water removed in the course of well development will be handled in accordance with the procedures developed for Handling and Disposal of Field Program Residuals for the project (SOP-4). The volume of water removed, method used, time of development, and other measurements will be recorded in the field logbook. In general, the method which will provide the highest flow velocity into and out of the filter pack will be used.

Surface centrifugal pump development may be used in 2-inch diameter wells where the depth to the bottom of the screened interval is less than the limit of surface centrifugal pump lift (approximately 25 feet). Submersible pump development may be used in cases where 4-inch diameter wells are present. Airlift development may be conducted in 2-inch diameter wells where the depth to the bottom of the screened interval is greater than the limit of surface centrifugal pump lift. Bladder pump development may be conducted in conjunction with surge block methods in 2-inch diameter wells where the depth to the bottom of the screened interval is greater than the limit of surface centrifugal pump lift. Bailer development may be conducted in conjunction with surge block methods in 2-inch diameter wells where the depth to the bottom of the screened interval is greater than the limit of surface centrifugal pump lift.
EQUIPMENT

All equipment should be assembled, calibrated, and tested prior to sampling. All downhole drilling equipment will be decontaminated prior to use at each drilling location. Equipment that may contact samples or ground water will be prevented from contact with the ground surface and other potentially contaminated materials. If a truck tailgate is used as an equipment bench, it will be covered with plastic sheeting. Field instruments and equipment should be kept out of direct sunlight to avoid temperature fluctuations.

DECONTAMINATION PROCEDURES

New well-screen and casing materials that are not pre-cleaned and wrapped will be decontaminated before use by means of high-pressure hot-water spray equipment with potable water as the cleaning medium.

If a pump is necessary to properly develop a well, it will be cleaned in the following manner:

- Clean two buckets with potable water and detergent (e.g., Alconox), rinse with tap water, and final rinse with distilled water before cleaning equipment.
- Fill first bucket with detergent and potable water.
- Fill second bucket with potable water.
- Submerse the pump intake device into the detergent water and operate for 5 minutes.
- Submerse the pump intake device into the potable water and operate for 5 minutes.
These procedures describe the techniques to be implemented for water-level measurements, well evacuation, sample withdrawal, and field measurements from monitoring wells. Equipment necessary for the implementation of these procedures is also summarized. Water samples collected from mine shafts will require slightly different procedures which are noted in the discussions below.

The following procedures should be followed in the collection of all ground-water samples:

- Note condition of well or shaft.
- Prepare Liquid Substance Sampling Form (Figure SOP-9.6) and record data in sample logbook.
- All equipment or material to be introduced into a monitoring well or mine shaft for ground-water sampling purposes will be kept from contact with the ground and other potentially contaminated materials by the use of plastic sheeting or other methods. Any such equipment which contacts the ground or other potentially contaminated materials will be suitably decontaminated before it is used in the well.
- Lay out equipment on plastic sheeting adjacent to sampling location to prevent direct contact with the ground surface.
- Measure depth to water in all wells/shafts prior to pumping or bailing of any wells. Record water depths, and time of measurements in field notes.
- If sampling point is a well, calculate bore volume based on well bore diameter, sandpack porosity, well diameter, and length of water column. Record calculation in field notes. Purge the well by evacuating water as described in the section below. Record volume, rate, duration, and time of purge in field notes and monitoring well purge/sampling field form (Figure SOP-3.1)
MONITORING WELL PURGE/SAMPLING FIELD FORM

Client: ________________________________ Project No: ___________________________

Location: ________________________________

MONITORING WELL NO: _________________________ SAMPLE NO: ________________________

Total Depth of Well _________________ (TD)

Well Diameter ____________________________

Construction Material ____________________________

Static Water Level Before Purge (**) __________________ (SWL) Time: _________________

Static Water Level Before Sampling (**) __________________ Time: _________________

Gallons of Standing Water (See Below) __________________ (V)

Total Purge Before Sampling (See Below) __________________ (TV)

TD - SWL = H

H(FT) x C(GAL/FT) = V(GAL)

3 x V = TV

Turbidity: __________________ Color: __________________ pH: / / / / _____________

Temperature: __________________ Conductivity: / / / / _____________

Recharge Characteristics: __________________

COMMENTS

H = Height in Feet of Water Column in Well
C = Conversion Equivalent (Gal/FT)
V = Water Volume in One Purge
* = Nearest Tenth of Foot. Point of Reference

Conversions (C) (GAL/FT)

Well Diameter | Equivalents
------------- | ------------
2"           | 1' = 0.16 Gal
4"           | 1' = 0.85 Gal
6"           | 1' = 1.47 Gal
8"           | 1' = 2.81 Gal

Dames & Moore

FIGURE SOP-3.1
If well recovery is insufficient to meet the three bore-volume standard, evacuate to dryness and allow well to recover (USEPA, 1986b). If recovery exceeds two hours, samples can be taken as soon as sufficient volume is available for a sample for each parameter.

Collect samples from the well/shaft using the appropriate device (i.e., bailer, submersible pump, depth discrete sampler).

Measure pH, specific conductance, dissolved oxygen and temperature.

Filter and/or preserve the sample as per SOP-9.

Fill necessary sample bottles.

Label sample bottle with appropriate labels, being sure to provide required information. Complete chain-of-custody and sample analysis request form. Record times and data pertaining to sampling and filtering in field logbook.

Place the properly labeled sample bottle in an appropriate carrying container on ice (4°C).

Either discard or decontaminate all items which contact the sample, such as bailers, pumps, cables, tubing, probes, gloves, and beakers, before proceeding to the next sampling location.

Sample protocol will be strictly followed to obtain representative ground-water samples and to maintain sample integrity.

PROCEDURES FOR WATER-LEVEL MEASUREMENT

Water-level measurements will be referenced to a known elevational datum. The measuring point at the top of the casing or shaft will be permanently marked and surveyed. Water-level measurements will be consistently taken from the same marked point.
Water levels will be measured in all wells prior to well evacuation. Water levels will be measured with an electrical sounding device, tape measure, or equivalent device.

Water levels will be measured by lowering the probe or tape measure into the well/shaft until contact with the water surface is indicated. The electric tape will be marked at the reference measuring point and partly withdrawn; the distance from the mark to the nearest tape band will be measured and added to (or subtracted from) the band reading to obtain the depth to water below the reference point. Readings will be verified by repeating the procedure until successive measurements differ by less than .01 feet. Measuring devices will be decontaminated prior to and after each monitoring well.

PROCEDURES FOR WELL EVACUATION

At least three well-bore volumes of water will be evacuated from the well before sampling. If conditions permit (i.e., the well is rapidly recharged); evacuation will continue until the well water temperature, pH, and specific conductance are stabilized, or until at least three well-bore volumes are removed, whichever is first. Operation and calibration specifications for the pH and specific conductance meters are included below in the section on field measurements.

Bailing: Bailer construction will consist of stainless steel, teflon, or fluorocarbon resin material. Procedures are:

- Decontaminate equipment, record total depth and water level, and calculate the fluid volume in the well bore (include the water in the screen, casing, and gravel/sand pack, assuming a gravel/sand pack porosity of 30 percent).
- Collect bailed water for pH and conductivity measurements.
Evacuate the well in accordance with the above section.

Submersible Pump: The electrical pump housing and impellers will consist of stainless steel and/or teflon. Procedures for use are:

- Decontaminate equipment, record total depth and water level, and calculate the fluid volume in the well bore (include the water in the screen, casing, and gravel/sand pack, assuming a gravel/sand pack porosity of 30 percent).
- Collect purge water for pH and specific conductance measurements
- Evacuate the well in accordance with the above section.

Sample Withdrawal

When the well has been purged, sampling will begin. If recovery is slow, it may be necessary to wait until sufficient volume is available for all the necessary analyses.

When bailers are used to collect samples, sections of bailer cord or cable will be dedicated to each monitoring well to prevent cross-contamination of wells by re-used bailer cord.

EQUIPMENT PROCEDURES

All equipment should be assembled, calibrated, and tested prior to sampling. Items which may come in contact with the sample should be decontaminated according to the procedures described in SOP-1. Between sampling locations, all items which have come in contact with the sample will be either discarded or decontaminated.

At most sites, both filtered and unfiltered samples will be collected. Care will be taken to thoroughly agitate the sample container while pouring unfiltered water into the sample bottles for inorganic and metal analysis. Samples will be filtered at the time of collection through a 0.45 micron membrane filter using a
geo-filter peristaltic pump and filter. The initial 100 to 200 ml of filtrate will be used to rinse the sampling system and sample bottles and will be discarded prior to actually obtaining a filtered sample.

Equipment decontamination will be performed between each sample as per sample equipment decontamination procedures in SOP-1. Equipment which comes in contact with potential sources of contamination during sampling will be discarded or decontaminated prior to sampling the next site. The pump hose will be decontaminated between each sample by triple rinsing with deionized water. Equipment such as sample hoses will be replaced when visual staining not removed by triple rinsing is observed. Other equipment will be washed with a mild laboratory detergent and a pH 4 nitric acid solution if necessary to remove staining.

Field measurements at most sites will include pH, dissolved oxygen, temperature, and specific conductance. Specific conductance, temperature, and dissolved oxygen will be measured using YSI portable conductivity and dissolved oxygen meters or equivalent devices. Field pH will be measured using an Orion pH meter.

At a minimum, instruments will be calibrated at the beginning of each work day and at any time the calibration range is exceeded during measurements. The pH meter will be calibrated using a dual point calibration (buffers pH 4 and pH 10). The conductivity meter will use a one point calibration with a certified standard. The dissolved oxygen meter will be calibrated at each site using a calibration chamber. Standard solutions will be labeled with their laboratory measured values and current shelf life. Calibration and operational procedures for the field pH meter, conductivity meter and dissolved oxygen meter are included with the manufacturer’s
equipment specifications attached in Appendix A. Dissolved oxygen measurements will be made on samples obtained by bailer with care taken to avoid aeration of the sample.
STANDARD OPERATION PROCEDURE NO. 4
HANDLING AND DISPOSAL OF FIELD PROGRAM RESIDUALS

These procedures describe the techniques to be used for handling and disposal of residuals obtained from field drilling, well development and sampling. The goal of residual disposal is to minimize potential contamination due to exposure of residuals to the environment.

Decontamination

It is not anticipated that wash water or rinse water, used in decontamination procedures, will constitute a hazardous material. Therefore, wash and rinse water will be discarded on-site.

Solid Materials

The residual solid materials will consist primarily of drill cuttings. It is not anticipated that drill cuttings will constitute a hazardous material because existing mine waste materials are not hazardous wastes. Therefore, the cuttings will remain at each drill site. If a land owner objects to the disposal of non-hazardous drilling residue at a drilling site, that material will be transported to a Baxter Springs/Treece mine waste location for disposal where landowner permission has been obtained.

In the event that drill cuttings exhibit visible signs (e.g., odor or color) that they may have constituents or characteristics other than those related to mining wastes, these materials will be sampled and analyzed for hazardous characteristics. If laboratory analysis confirms that drill cuttings are hazardous, other arrangements will be made for the disposal of these materials.
Development and Purge Waters

It is not anticipated that waters obtained by the development and purging of wells will constitute a hazardous material. Development and purge waters will be discarded on-site. The discharge of development and purge waters will be channelized or otherwise controlled to prevent erosion or other damage to property. The pH of purge and development waters will be monitored. These waters will be containerized if the pH drops below 4.0 and transported to a central location to be held for treatment and/or disposal.
This Standard Operating Procedure (SOP) describes the operating procedures for the use of a field portable X-Ray fluorescence spectrometer (FPXRF) to analyze solid materials for metals content, i.e. soils, sediments, sand tailings and chat. Instruments that may be used are the X-MET 880 from Outokumpu Electronics, Inc., the Spectrometer ATX-100 from Enviro-Recovery Consultants or any equivalent field portable unit.

Some FPXRF units, particularly the X-MET 880, can use several types of sample probes with different configurations and X-Ray sources. The FPXRF unit selected for the Baxter Springs and Treece investigations will use a "surface analysis" probe. In order to obtain accurate measurements, the surface analysis probe must be placed flat against the surface of the sample material. The surface analysis probe measures only a small portion of the surface; therefore, several measurements will be taken at a sampled point. The X-Ray sources used in either type of probe must be of the proper energy to measure the concentrations of the metals of interest.

Proper operation, calibration and maintenance of an FPXRF unit are essential to obtaining usable data from this program. The operator of the FPXRF unit will be trained in proper operation, calibration and maintenance of the unit by attendance at a manufacturer's training program or by a minimum of 24 hours of field training by an experienced operator.

MEASUREMENT TECHNIQUES AND FIELD PROCEDURES

A field team will consist of at least two people, one to operate the FPXRF unit and the other to record readings and locations in the logbook and to cross check operations. All
readings taken with the FPXRF will be recorded in a field logbook for future reference even if the instrument is also storing the data. The field log will include the date, time, a location description and a material type indication for each reading. An example form showing required data is presented as Figure SOP-5.1.

Measurement Procedures - Surface Analysis Probe

The surface analysis probe measures metal concentrations in a very small volume of material (approximately 0.05 cubic inches). To compensate for possible nugget effects that would be increased by this small volume, several measurements will be taken in a pattern centered on the nominal measurement point. The measurement procedure will be as follows:

- Check to make sure that the FPXRF is calibrated for the matrix at the location that is under investigation.
- Place surface probe against nominal measurement point, take reading.
- Record reading in field logbook along with location coordinates and stake location number. Survey coordinates will be determined and survey stakes will be placed prior to conduct of the FPXRF program.
- Move surface probe 12 inches north of the nominal measurement point, take and record reading.
- Repeat measurements at 12 inches east, south and west of nominal measurement point.
- After every tenth reading, check the calibration by reading the low concentration and medium concentration check standards at least three times each.
- At every tenth measurement point, take a composite soil sample with a clean stainless steel spoon or trowel following instructions in SOP-7 and SOP-8. Place the composited samples in a clean sample jar, and store in a cooler at 4°C for shipment to an analytical laboratory for analysis of the selected metals. Label the sample jar as an FPXRF field QC sample, and record all pertinent information in the FPXRF field logbook, and Solid Substance Sampling form.
# Field Data Reporting Sheet

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**FIGURE SOP-5.1**
Measurement Procedures - SPEX Cup Analysis Probe

If samples are to be measured in the field with a probe using SPEX cups, the measurements will be limited to dry fine solid materials. It is not anticipated that the FPXRF will be used on samples needing drying and crushing. The measurement procedure will be as follows:

- Sieve the sample onto a large, fresh piece of kraft or butcher paper.
- Homogenize the sample by rolling it on to itself by lifting the corners of the paper alternately until each corner has been lifted at least 20 times.
- With a plastic teaspoon, remove approximately 5 grams of sample from the middle of the pile and place it in the SPEX cup and seal off with polypropylene film.
- Measure the concentration of the sample with the FPXRF and record all the data required as above and note that this was a SPEX cup measurement.
- Retain the excess material not used in the XRF measurement from at least every tenth sample for QA/QC check analyses. Place in a clean sample jar and label accordingly.

INSTRUMENT CALIBRATION AND PREVENTATIVE MAINTENANCE

Calibration and preventative maintenance procedures are specific to the instrument used. The field calibration procedure and preventative maintenance schedule and instructions from the users manual for the instrument chosen will be used. Full calibration will be performed, at a minimum, at the beginning and end of each field operations day and when the sample matrix changes. More frequent calibration may be performed if found necessary. In addition, after every tenth sampling point a low concentration check standard and a midrange calibration check standard will be analyzed in triplicate and the results recorded in the logbook.
Prior to the FPXRF program, at least ten samples will be collected from each type of sample matrix in which FPXRF measurements are expected to be made to serve as calibration samples. The samples will be sieved and split. One split from each calibration sample will be analyzed for the metals of interest by standard laboratory methods with full quality control and the other split will be placed in a SPEX cup to serve as the field calibration standard. The FPXRF may be used for up to four solid matrices: soils, sediments, chat and sand tailings. Initially, only one set of calibration samples will be gathered for each type of matrix to represent all of that matrix throughout the subsites. Sufficient field QA/QC samples (i.e. one QA/QC sample per 10 XRF readings), will be gathered to allow for adjustment of the calibration model if it is found that there are problems with the site-wide calibration.

If the range of concentrations of the calibration samples does not extend high enough to cover the possible range of concentrations that could be encountered in the field, calibration samples will be spiked with known amounts of lead, cadmium and zinc to cover the needed range of concentrations. The added metals will be in their expected compounds, i.e. as sulfides. The spiked samples will be analyzed by the same methods as the other calibration standards.

Calibration will be conducted separately for each type of matrix, i.e., soils, sediments, chat and sand tailings, and a separate set of calibration standards will be assembled for each matrix. Two of the calibration samples for each matrix will be carried in the field for periodic instrument checks. These will be a low concentration and a medium concentration calibration standard.
PREPARATION OF MATRIX SPECIFIC CALIBRATION SAMPLES

The method that will be used to prepare the matrix specific calibration standards is as follows:

- Using a stainless steel spoon, the sample is scooped into a pre-cleaned (acid washed) 8 ounce jar.

- Oven dry the sample at 60°C for a minimum of 6 hours. If mercury is to be analyzed, a separate split must remain undried, as heating may volatilize the mercury.

- Sieve the sample through a standard 10 mesh (2 mm) sieve. Push the peds through the screen but not to pulverize the sample. All that should remain behind on the screen is the coarse rock fraction.

- Homogenize the soil by placing the dried, sieved sample on a large (approximately 1.5 X 1.5 foot) piece of kraft or butcher paper. The sample at this point should be 150 to 200 grams. Lift each corner of the paper alternately rolling the soil over on itself and toward the opposite corner. Continue until the soil has been rolled toward each corner 20 times.

- One in five of the intrusive samples taken for corroboratory analysis are split to track the sample preparation variance.

- With a plastic teaspoon, remove approximately 5 grams (approximately one teaspoon) of soil from the middle of the pile, pour it into a 31 mm diameter SPEX cup and seal off with 0.2 mil polypropylene. The SPEX cup must be between one half and three quarters full. Remove approximately 30 grams (5 to 6 teaspoons) of soil from the pile and pulverize to minus 200 mesh (0.074 mm) and transfer to a clean container. Pour the soil remaining on the paper into a clean glass or plastic container, exercising care to tap the fine fraction that tends to remain on the upper into the container. The sample in the SPEX cup becomes the loose soil SSC standard used to calibrate the FPXRF instrument. One half of the pulverized sample is sent to an analytical laboratory for total digestion and ICP/AA inorganic characterization. The other half of the pulverized sample is pressed into a pellet for analysis on the mobile laboratory XRF instrument.
INTRODUCTION

This Standard Operating Procedure (SOP) describes the procedures to be followed in conducting batch leach and column leach testing of surficial wastes and other materials from the Baxter Springs and Treece subsites with site waters or distilled water. The data from these tests will be used if the investigation of aqueous mobility of metals from the mining wastes on these sites is necessary.

Procedures are outlined for two types of column leaching. One simulates exposure of waste materials to rain under unsaturated surface conditions. The other simulates waste materials saturated by ground water that is occasionally replaced by infiltration due to rainfall.

BATCH LEACH TESTING

The batch leach testing will follow the procedures outlined in ASTM Standard Method D 4319-83 "Distribution Ratios by the Short-Term Batch Method" which is attached. No significant deviations from that method are anticipated. The procedure is self-explanatory and will not be restated in the text of this SOP.

COLUMN LEACH TESTING - RAIN WATER SIMULATION

Column leach testing will generally follow procedures that have been developed and tested on mining wastes from other subsites in the Cherokee County area. These tests are intended to simulate exposure of mining waste materials on the ground surface to rain.
Total leach test time will be 28 days with simulated rain fall events on the third, fifth, ninth, fifteenth and twenty-eighth days.

Column Construction

Columns will be constructed with a height to diameter ratio of three to one, and be of sufficient volume to hold at least 80 pounds of chat or sand tailings (0.8 cubic feet at 100 lb/cu ft). The columns will be constructed with a distribution system at the top to spread water additions evenly across the test material. Water will be collected at the bottom of the column.

Test Procedures

- If the sample contains material coarser than 1", the sample will be crushed to pass 1". The sample will be blended and a split taken for pre-leach chemical characterization.

- The column will be filled with sample, and the weight of sample added will be recorded. In addition the total weight of the column and sample will be recorded for pore volume calculations.

- The leaching fluid may be rain water collected on the site, simulated rain water or distilled water. A single batch of water will be used throughout one test. It will be stored at 4°C to minimize any change during the test program. An aliquot of the water will be taken at the beginning of the test program for pre-leach chemical characterization.

- The column will be flooded with leaching fluid to the top of the solid sample and allowed to sit for one hour to allow air to escape. After ensuring that the water is still at the top of the sample, the column will be weighed to measure the amount of water added which will be used to estimate the pore volume of the sample.

- The column will then be drained and the water collected. The volume of water collected, its specific conductance, pH and temperature of the water will be measured and recorded, and sample will be taken and stored at 4°C for later analysis.
The column will be sealed to maintain humidity and the sample allowed to stand wet but unsaturated for two days.

After two days, an aliquot of the leaching fluid, equal to the volume of 2" of height of the column, will be distributed evenly across the material in the column at a rate that will allow distribution of the aliquot in two hours.

The water will be allowed to percolate through the sample and collected at the bottom of the column. Volume versus time measurements of leachate flow will be made in sufficient detail to calculate a yield curve (volume measurements at maximum time intervals of 5 minutes).

The total volume, specific conductance, pH and temperature of the water collected will be measured and recorded. A sample will be saved and stored at 4°C for later analysis.

The last three steps above will be repeated on the fifth, ninth, fifteenth and twenty-eighth days, with the column being kept sealed between leaching days.

COLUMN LEACH TESTING - SATURATED GROUND-WATER SIMULATION

These tests are intended to simulate exposure of mining waste materials to ground water with displacement by infiltration after rainfall events. Total leach test time will be 28 days with simulated rainfall events on the third, fifth, ninth, fifteenth and twenty-eighth days.

Column Construction

Columns will be constructed with a height to diameter ratio of three to one, and be of sufficient volume to hold at least 80 pounds of chat or sand tailings (0.8 cubic feet at 100 lb/cu ft). The columns will be constructed with the inlet at the bottom and the outlet at the top. The inlet will be connected to a pressurized reservoir large enough to hold a volume of water equal to the volume of 2 inches of length of the column. The outlet will be
connected to a container that can hold at least the same volume as the inlet reservoir. The outlet line will be provided with a vacuum break to prevent syphoning.

Test Procedures

- If material sample contains material coarser than 1", the sample will be crushed to pass 1".

- The column will be filled with sample, and the weight of sample added will be recorded. In addition the total weight of the column and sample will be recorded for pore volume calculations.

- The leaching fluid may be rain water collected on the site, simulated rain water or distilled water. A single batch of water will be used throughout one test. It will be stored at 4°C to minimize any change during the test program. An aliquot of the water will be taken at the beginning of the test program for pre-leach chemical characterization.

- The column will be flooded with leaching fluid to the top of the solid sample and the reservoir height adjusted to maintain saturation of the sample.

- After initial flooding the sample will be allowed to stand flooded two days.

- After two days, an aliquot of the simulated rain water equal to the volume of 2" of height of the column will be added to the reservoir and allowed to percolate through the sample at a rate that will allow the aliquot to move through the sample in two hours.

- The water percolating through will be collected at the top of the column. The pressure required to produce flow through the column at the rate specified will be measured and recorded.

- The total volume, specific conductance, pH and temperature of the water collected will be measured and recorded. A sample will be taken and stored at 4°C for later analysis.

- The last three steps above will be repeated on the fifth, ninth, fifteenth and twenty-eighth days, with the column being kept saturated between leaching days.
After the sample for the twenty-eighth day is collected, the remaining solution will be drained from the column.

The column will be sealed to maintain humidity and allowed to stand for seven more days.

After seven days the column will be flooded at the same rate used above, until a volume of leachate equal to the volume of 2 inches of column height is collected at the overflow. This sample will be saved and stored at 4°C for later analysis.
INTRODUCTION

As an aqueous fluid migrates through geologic media, certain reactions occur that are dependent upon the chemistry of the fluid itself and upon the chemistry and geochemistry of other fluids and solid phases with which it comes in contact. These geochemical interactions determine the relative rates at which chemical species in the migrating fluid (such as ions) travel with respect to the advancing front of water. Processes of potential importance in retarding the flow of chemical species in the migrating fluid (movement of species at velocities less than the ground-water velocity) include ion exchange, adsorption, complex formation, precipitation (or coprecipitation, for example $^{39}\text{Sr}^{2+}$ and $^{88}\text{Sr}^{2+}$ coprecipitating as the sulfate), oxidation-reduction reactions, and precipitate filtration. This test method applies to situations in which only sorptive processes (adsorption and ion exchange) are operable for the species of interest, however, and is restricted to granular porous media.

It is difficult to derive generalized equations to depict ion exchange-adsorption reactions in the geological environment. Instead, a parameter known as the distribution coefficient ($K_d$) has been used to quantify certain of these sorption reactions for the purpose of modeling (usually, but not solely, applied to ionic species). The distribution coefficient is used to assess the degree to which a chemical species will be removed from solution as the fluid migrates through the geologic media; that is, the distribution coefficient provides an indication of how rapidly an ion can move relative to the rate of ground-water movement under the geochemical conditions tested.

This test method is for the laboratory determination of the distribution ratio ($R_d$), which may be used by qualified experts for estimating the value of the distribution coefficient for given underground geochemical conditions based on a knowledge and understanding of important site-specific factors. It is beyond the scope of this test method to define the expert qualifications required, or to justify the application of laboratory data for modeling or predictive purposes. Rather, this test method is considered simply a measurement technique for determining the distribution ratio or degree of partitioning between liquid and solid, under a certain set of laboratory conditions, for the species of interest.

Justification for the distribution coefficient concept is generally acknowledged to be based on expediency in modeling-averaging the effects of attenuation reactions. In reference to partitioning in soils, equilibrium is assumed although it is known that this may not be a
valid assumption in many cases. Equilibrium implies that (1) a reaction can be described by an equation and the free energy change of the reaction, within a specific system, i.e., and (2) any change in the equilibrium conditions (T, P, concentration, etc.) will result in an immediate reaction toward equilibrium. The concept is based upon reversibility of reactions. Measured partitioning factors may include adsorption, coprecipitation, and filtration processes that cannot be described easily by equations and, furthermore, these adsorption mechanisms may not instantaneously respond to changes in prevailing conditions. Validity of the distribution coefficient concept for a given set of geochemical conditions should not be assumed initially, but rather should be determined for each situation.

This is a short-term test and the attainment of equilibrium in this laboratory test is presumed, although this may be so for certain systems (for example, strictly interlayer exchange reactions of clays). Consistent with general usage, the result of this test could be referred to as “distribution coefficient” or as “distribution ratio”; in the strictest sense, however, the term “distribution ratio” is preferable in that the attainment of equilibrium is not implied.

The distribution ratio ($R_d$) for a specific chemical species may be defined as the ratio of the mass sorbed onto a solid phase to the mass remaining in solution, which can be expressed as:

$$R_d = \frac{\text{mass of solute on the solid phase per unit mass of solid phase}}{\text{mass of solute in solution per unit volume of the liquid phase}}$$

The usual units of $R_d$ are mL/g (obtained by dividing g solute/g solid by g solute/L solution, using concentrations obtained in accordance with this test method).

Major difficulties exist in the interpretation, application, and meaning of laboratory-determined distribution ratio values relative to a real system of aqueous fluid migration through geologic media. Typically, only reactions between migrating solutions and the solid phases are quantified. In general, geochemical reactions that can result from interactions of the migrating fluid with another aqueous phase of a differing chemistry have not been adequately considered (interactions with other liquids can profoundly change the solute chemistry). Additionally, as noted above, the distribution coefficient or $K_d$ concept imposes an equilibrium condition for given reactions, which may not realistically apply in a natural situation because of the time-dependence or kinetics of specific reactions involved. Also, migrating solutions always follow the more permeable paths of least resistance, such as joints and fractures, and larger sediment grain zones. This tends to allow less time for reactions to occur and less sediment surface exposure to the migrating solution, and therefore preclude the attainment of local chemical equilibrium. Thus, the distribution coefficient or $K_d$ concept is only directly applicable to problems involving contaminant migration in granular porous material.

Sorption phenomena are also strongly dependent upon the thermodynamic activity of the species of interest in solution (chemical potential). Therefore, experiments performed using only one activity or concentration of a particular chemical species may not be representative of actual in situ conditions or of other conditions of primary interest. Similarly, unless experimental techniques consider all ionic species anticipated to be present in a migrating solution, adequate attention is not directed to competing ion and complexation effects, which may strongly influence the $R_d$ for a particular species.

Many “sorption” ion complexation effects are strongly influenced, if not controlled, by conditions of pH and Eh. Therefore, in situ conditions of pH and redox potential should be considered in determinations of $R_d$. To the extent possible, these pH and Eh conditions should be determined for field locations and must be approximated (for transition elements in the laboratory procedure).

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1. Scope

1.1 The of distribution technique primarily migration application...

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1. Scope

1.1 This test method covers the determination of distribution ratios of chemical species for site-specific geological media by a batch sorption technique. It is a short-term laboratory method primarily intended for ionic species subject to migration in granular porous material, and the application of the results to long-term field behavior is not known. Distribution ratios for radionuclides in selected geomedia are commonly determined for the purpose of assessing potential migratory behavior at waste repositories. This test method is also applicable to studies of intrusion waters and for parametric studies of the effects of variables and of mechanisms which determine the measured distribution ratios.
1.2 The values stated in acceptable metric units are to be regarded as the standard.
1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable Documents

2.1 ASTM Standards:
D 422 Method of Particle-Size Analysis of Soils
D 2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants
D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)
D 3370 Practices for Sampling Water

3. Description of Terms Specific to This Standard

3.1 distribution coefficient, K=a is identically defined as Ra for equilibrium conditions and for ion exchange-adsorption reactions only. To apply Ra values to field situations, an assumption such that Ra = K is necessary. The validity of such an assumption can only be determined by informed experts making a judgment (albeit uncertain) based on a detailed study of the specific site.

3.2 distribution ratio, Ra—the ratio of the concentration of the species sorbed on the soil or other geo-media, divided by its concentration in solution under steady-state conditions, as follows:

\[
Ra = \frac{\text{mass of solute on the solid phase}}{\text{per unit mass of solid phase}} / \frac{\text{mass of solute in solution per unit volume of the liquid phase}}
\]

By steady-state conditions it is meant that the Ra values obtained for three different samples exposed to the contact liquid for periods ranging from 3 to at least 14 days, other conditions remaining constant, shall differ by not more than the expected precision for this test method.

The dimensions of the expression for Ra reduce to cubic length per mass (L^3/M). It is convenient to express Ra in units of milliliters (or cubic centimeters) of solution per gram of geo-media.

3.3 species—a distinct chemical entity (such as an ion) in which the constituent atoms are in specified oxidation states.

4. Significance and Use

4.1 The distribution ratio, Ra, is an experimentally determined parameter representing the distribution of a chemical species between a given fluid and a geo-medium sample under certain conditions, including the attainment of a steady state. Based on a knowledge and understanding of the important site-specific factors, Ra values may be used by qualified experts for estimating the value of the distribution coefficient, K, for a given set of underground geochemical conditions. The K concept is used in mass transport modeling, for example, to assess the degree to which an ionic species will be removed from solution as the solution migrates through the geosphere. For applications other than transport modeling, batch Ra measurements also may be used, for example, for parametric studies of the effects of variables and of mechanisms related to the interactions of fluids with geo-media.

5. Apparatus

5.1 Laboratory Ware (plastic bottles, centrifuge tubes, open dishes, pipets, graduated cylinders, beakers, etc.)
5.2 Centrifuge
5.3 Laboratory Shaker/Rotator, ultrasonic cleaner (optional).
5.4 Environmental Monitoring Instruments: pH meter, electrometer and electrodes for B determination, conductance apparatus, and thermomter.
5.5 Analytical Balance.
5.6 Appropriate Equipment, necessary to maintain in situ conditions within the laboratory.
5.7 Analytical Instrumentation, appropriate for determination of the concentration of major constituents (cations and anions) and of the species of interest in the core geo-media.

6. Sampling

6.1 The samples shall be collected from in situ sites, based on expressed, specific guidance, procedures, and if necessary, using core geo-media.
6.2 The site to origin is at least a meter in depth of sampling.
6.3 A geo-medium sampling method, such as the core geo-medium method (Method D-4319), is necessary to avoid biases from stripping out contaminants. The method should be performed and the required precision shall be achieved within the laboratory. Where plateaux may have significant effect on the measurement, certain porous plastically should be avoided and the use of FEP TFE-fluorocarbon containers is recommended.
6.4 Sufficient detail and precision for the analysis is required. Specific guidance and standards are necessary to maintain in situ conditions and to avoid bias from stripping out contaminants. The method should be performed within the laboratory.

Note 1—It is extremely difficult to analyze un-consolidated soils or high-density reactive soil. This method is insufficient for detailed exploration programs, and, if explored, is extremely expensive. Specific guidance and standards are necessary to maintain in situ conditions and to avoid bias from stripping out contaminants.

7. Procedure

7.1 This test is conducted in a consolidated aggregated core. For the application of this standard, it is recommended that the use of core geo-media in the laboratory with the required precision.
Rf, is an empirical factor representing the fraction of the tracer that is removed from the solution through transport processes, relative to removal by transport processes only. The value of Rf is used for estimating migration rates. In certain cases, the migration rate may be estimated by curve fitting techniques or by a Monte Carlo technique. For example, in the study of migration rates of solutes through soils, Rf values may be determined by using a numerical technique such as the Monte Carlo technique.

6. Sampling

6.1 The samples of soil, rock, or sediment shall be considered to be representative of the stratum from which it was obtained by an appropriately accepted or standard procedure and based on expert judgment.

6.2 The sample shall be carefully identified as to origin in accordance with Practice D 2488.

6.3 A geological description shall be given of the core material used for the distribution ratio measurement, including particle-size analysis (Method D 422) for unconsolidated material, depth of sample, and boring location.

6.4 Sampling of representative ground water in the test zone for use as the contact liquid in this test method shall be accomplished in accordance with Practices D 3370, using sampling devices that will not change the quality or environmental conditions of the waters to be tested. Recommended methods include the use of Kemmerer samplers or inert gas pressure lifts (provided this does not alter the ground-water sample by stripping out carbon dioxide and raising the pH, for example) or submersible diaphragm-type pumps. Proper precautions should be taken to preserve the integrity of in situ conditions of the sampled water, and in particular to protect against oxidation-reduction, exposure to light for extended periods, and temperature variation.

NOTE 1—it is recognized that sampling is likely to be a major problem. Materials (or fractures) that the contaminants pass through are likely to be the most difficult part of the geologic section to sample. In addition, proper sampling entails determining the path of ground-water flow so that the critical materials can be sampled. This determination is often accomplished by using a neat, well-equipped method of inquiry in normal geologic site exploration programs. However, if it is attempted in some cases, the exploration program may become unacceptably expensive. Specific guidelines are beyond the scope of this test method, but it is recommended that geologic and water sampling procedures be carefully considered by personnel involved in the site examination.

7. Procedure

7.1 This test method can be applied directly to consolidated core material samples or to disaggregated portions of core material samples. For the applications intended for this test method, however, disaggregation of the samples is the recommended procedure. Disaggregate the sampled soil and friable core materials (this may be done by ultrasonic method although it should be noted that the effect of ultrasonics on the microstructure of geological materials may lead to higher sorption values in certain cases). If a sufficiently large-sized sample is available, separate 200-g portions through a "nonsuis" riffle splitter. Crush competent sedimentary rock materials to a desired particle size or equivalent soil texture anticipated to result from natural weathering processes (this is because surface area is controlled by sample particle size).

NOTE 2—A significant source of error may be introduced by disaggregating the sample in a batch test in that (a) disaggregation can mask a preferred flow path (either horizontal or vertical), (b) disaggregation can destroy the effect of preferred flow paths caused by fractures or perhaps thin sand stringers, and (c) disaggregation will tend to increase the available surface area of the geologic materials. It is for the purpose of achieving uniformity of application, however, that disaggregation is recommended for this test method. It should be realized by persons applying results from this method that inclusion of the disaggregating operations may for these reasons tend to maximize the values of the distribution coefficients (ratios) obtained from this test method.

7.2 In some cases, it may be desirable to remove organic material from the geomeidium (soil specimens) for comparative purposes. If this is so indicated, remove the organic material from the composite sample mixtures for selected samples by treatment with concentrated hydrogen peroxide (30% H2O2), using the procedure given in "Soil Chemical Analysis". In such a case, make duplicate runs using samples both with and without pretreatment to remove organics. It should be noted, however, that treatment with concentrated hydrogen peroxide could cause other changes in the geomeidium, for example, dissolution of hydrous metal oxides that may be important adsorbents.

7.3 Using standard analytical procedures, characterize the geologic specimens (without pretreatment and, if so done, with the pretreatment to eliminate organics) as considered appropriate. The analyses may include percent chemical composition of anhydrous oxides (for example, SiO2, FeO, MnO, CaO, Na2O, etc.), hydrous oxides (for example, Fe, Mn, and Al hydrous oxides), etc.1

and minerals that are present, and carbonate content, surface area (m²/g), and cation and anion exchange capacity (at specified pHs). Similarly, characterize the contact liquid obtained from the test zone as appropriate for interpreting the results. Chemical analysis of the liquid should include macro constituents (for example, Na⁺, Ca⁺⁺, K⁺, Mg⁺⁺, Cl⁻, HCO₃⁻/CO₃⁻, SiO₂, etc.) and redox-active and hydrolyzable species such as Fe and Mn ions. Likewise, determine the pH and Eh of the contact liquid, as well as the concentration (if present) of the chemical species of interest. Specific instructions for the Eh determination are not part of this test method, however, use of a referenced technique is advised (such as a platinum versus standard calomel electrode measurement). If the species of interest may exist in the contact liquid in a variety of valence or chemical states (for example, with studies of actinides), a method of determining speciation should be applied.

7.4 Pass each of the soil and rock (core sediments) fractions again through a “nonbias” riffle splitter and place four 5 to 25-g portions (record weight to nearest 0.1 g) in centrifuge tubes or bottles.

NOTE 3—Unless it is decided that the samples may be allowed to dry by exposure to the open air, record a moisture weight (for comparative purposes, a moisture content determination should be done with a separate sample). Some soils never dry in nature, and characteristics may be greatly altered when dried. This is especially true for originally anoxic sediments. If the samples are not to be allowed to dry before testing, follow Practice D 2217 (Procedure B) for maintaining a moisture content equal to or greater than the natural moisture content. In all cases, the contact liquid used in this test is the sampled ground water from the test zone.

7.5 If a radiotracer or spiked stable tracer determination of the distribution ratio is desired, pretreat the composite samples with exact solution (contact liquid) used in the determination but without the tracer present. This solution will be either the site-specific ground water or a selected intrusion water. Wash the composite soil and rock samples four times with the pretreatment solution. For the first three washes, stir the mixtures of soil and rock and pretreatment solution several times over a 15-min period, allow to settle, centrifuge at 1000 g or more for 5 min, and decant off the wash. Apply the fourth wash for at least 24 h with occasional stirring, and again separate the wash from the composite sample by centrifugation and decantation as before.

7.6 It may be advisable to pre-equilibrate the treatment solution (contact liquid) with the geomedia prior to the start of this test method. Proceed as in 7.5, using the fourth wash after centrifugation and decantation as the treatment solution. Unless otherwise noted, add 20 to 100 mL (exact value should be equal to four times the weight in g of the geomedia) to each 100 to 250 mL centrifuge tube or bottle, and thorough mix the contents by stirring action. Prior to contact, the treatment solution should contain the species of interest at a known concentration prepared by the addition of chemically pure reagents to the site-specific ground-water sample. (The species of interest may be at trace concentration; if it is a radioactive or stable tracer added to the treatment solution, the elemental concentration as well as the isotopic concentration must be known.) If tracers are used, first equilibrate the tracer with the ground-water (or intrusion-water) sample by allowing to stand overnight and then filter using a ≤0.45-µm pore size membrane filter. Following this step, analyze the contact solution and add to the soil and rock composite samples as indicated above. Measure the pH of the soil/rock-solution system; if the pH has changed or if other than the natural pH is desired, adjust by addition of N NaOH solution or HCl, or by an appropriate buffer. The in situ Eh should be maintained, if necessary, under an inert atmosphere.

NOTE 4—Experiments have shown that Rₛ will vary depending on the solution-to-geomedium ratio used in the test. If other ratios are indicated (which would more closely approximate the normal field situation), duplicate runs should be made, however, the ratio prescribed here should also be run as the reference case. Because Rₛ varies with the solution/medium ratio, it is strongly recommended that this measurement include determination of the isotherm by making several runs with different ratios of solution-to-geomedium than specified above.

7.10 Conductivity results should be compared to values to appropriate standard contact.

7.12 Filtered solution mixtures near dryer should be filters equivalent, through it the following by flow or using such a standard "in situ" contact.

7.7 Determine the specific conductance of each solution and report in units of microsiemens per centimeter at 20°C.

7.8 Run each set of samples at least in triplicate to demonstrate that steady state is attained in this short-term test. Stir the contents of each contact tube, then gently shake all of the soil/rock solution mixtures on a laboratory shaker/
Visible to pre-equilibrate the (contact liquid) with the geo-
media at untrace concentration. Prior to contact, the soil
solute should contain the chemically pure reagents and
water (or intrusion-water) at trace concentration; the
system must be at steady-state. If chemical standards
are used, first equilibrate the contact liquid, then
place the element concentration in the respective
contact solution. The concentration must be
known concentration prior to pre-equili-
ibration (chemical standard). If it is variable, of the
concentration must be
accurately determined using the appropriate standard analytical method.

7.11 If filtering is necessary or if desired for comparative purposes, use polycarbonate mem-
bane filters (0.002 to 0.02 μm pore size), or the
equivalent. Pretreat the filter disc by passing
through it approximately 50 mL of 1.0 N HCl,
followed by 50 mL of distilled water, by gravity
flow or suction to near dryness. Check the possibility of sorption of tracers onto the filter by a
standard "double filter" technique using the original
contact solution.

7.12 Filter the supernate from each soil/rock
solution mixture by gravity flow or suction to
near dryness. Determine the concentration and
speciation (chemical state), if it is variable, of the
species of interest in this solution by the appro-
riate standard analytical method. Make a blank
determination using the equivalent procedure
outlined here (7.6 through 7.12, except do not
add the soil/rock sample) with treatment solution
only. The use of tracers involves particular atten-
tion to corrections for blanks and potential
plateout of the tracer on container walls, filters, and
other surfaces as well as other losses. For
example, it should be ascertained that loss of
tracer to the blank vial walls is the same as for
the walls of the sample vial, etc.

7.13 If necessary or if desired for comparative
purposes or for a mass-balance determination,
determine the concentration of the species of
interest for each filtered solid residue. In this
case, note the necessity of removing the residual
solution from the solid phase, or correcting for
it, particularly for solids with low \( R_e \) values. If
this determination is made, a correction is re-
quired for the amount (if any) of the species of
interest to be found naturally present in the soil/
rock sample. Provided a satisfactory analysis is
accomplished for the species concentration in the
soil/rock residue, calculate \( R_e \) by dividing this
value (g solute per g solid residue) by the final
concentration in the contact solution (g solute
per mL solution), assuming the filter did not
remove tracer from the solution. An alternative
method is to compute \( R_e \) as shown on the Ex-
ample Calculation Sheet (Table 1).

8. Precision and Bias

8.1 In following this method with usual care
in analytical determinations, it is estimated that
an overall precision or reproducibility of 10 to
25 % should be obtained. In many cases, this
may be limited at very high or very low \( R_e \) values
by the difficulty in measuring either very small
residual concentrations or very small changes in
a higher concentration. In such cases, constancy
of \( R_e \) to within an order of magnitude may
be acceptable for certain applications. It should be
noted, however, that sampling difficulties and
inability to properly measure or control the rel-
levant in situ factors for determining the \( R_e \) of
interest can inject a substantial uncertainty into
the application of the obtained values in mass
transport or solute modeling-predictive exercises.
The distribution ratio is given by:

\[ R_d = \frac{(F_d)V_d}{(F_s)V_s} \]

where:

- \( R_d \) = distribution ratio, m/L,
- \( F_d \) = fraction of total activity in solution, which equals the total concentration in solution, assuming the activity coefficients of a given ion were the same before and after steady state was attained in contact of the solution with the soil/rock matrix (that is, the ionic strength is unchanged). Making this assumption, \( F_d \) is found by dividing the concentration of the ion after the solution has come to "equilibrium" (reaches steady state) with the soil/rock fraction by the concentration (of mass units) of the ion before the solution was allowed to come into contact with the soil fraction,
- \( F_s \) = fraction of activity sorbed onto the mineral or solid residue (correcting for the natural content of the species of interest initially present), or, making the same assumptions as to activity coefficients,
- \( V_d \) = volume of solution "equilibrium" with \( V_s \) mL and \( W_s \) = weight of minerals or solid residue, g.

In the case of a radioactive species of interest, where the radiocactivity of the solution and solid fraction are determined, the distribution coefficient is given by:

\[ R_d = \frac{(A_d)V_d}{(A_s)V_s} \]

where:

- \( A_s \) = activity of the mineral or solid residue, mCi, and
- \( A_d \) = activity of the solution "equilibrium" with \( W_s \) mCi.

### Table 1: Example Calculation Sheet

<table>
<thead>
<tr>
<th>TABLE 1 Example Calculation Sheet</th>
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</thead>
<tbody>
<tr>
<td>The distribution ratio is given by:</td>
</tr>
<tr>
<td>[ R_d = \frac{(F_d)V_d}{(F_s)V_s} ]</td>
</tr>
<tr>
<td>where:</td>
</tr>
<tr>
<td>( R_d ) = distribution ratio, m/L,</td>
</tr>
<tr>
<td>( F_d ) = fraction of total activity in solution, which equals the total concentration in solution, assuming the activity coefficients of a given ion were the same before and after steady state was attained in contact of the solution with the soil/rock matrix (that is, the ionic strength is unchanged). Making this assumption, ( F_d ) is found by dividing the concentration of the ion after the solution has come to &quot;equilibrium&quot; (reaches steady state) with the soil/rock fraction by the concentration (of mass units) of the ion before the solution was allowed to come into contact with the soil fraction,</td>
</tr>
<tr>
<td>( F_s ) = fraction of activity sorbed onto the mineral or solid residue (correcting for the natural content of the species of interest initially present), or, making the same assumptions as to activity coefficients,</td>
</tr>
<tr>
<td>( V_d ) = volume of solution &quot;equilibrium&quot; with ( V_s ) mL and ( W_s ) = weight of minerals or solid residue, g.</td>
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</tbody>
</table>

### Table 2: Example Report Sheet

<table>
<thead>
<tr>
<th>TABLE 2 Example Report Sheet</th>
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</thead>
<tbody>
<tr>
<td>Tabulated Results for Distribution Ratio Determination of Sample Number</td>
</tr>
<tr>
<td>Contact liquid: Site-Specific Ground Water</td>
</tr>
</tbody>
</table>

Site description, sampling methodology and core material description, analyses of core materials and of site-specific ground water or other contact liquid:

ATTACH SHEET

Species (ion) of interest | Initial Conc. in Solid (units) | Initial Conc. in Solution (units) | \( F_d \) | \( R_d \) | (mL/g) |

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years as if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.
INTRODUCTION

Techniques for sampling solid substances cover the collection of soils, stream sediments, sand tailings, waste rock, and chat. Once collected, it is anticipated that solid substance samples will be either submitted for analysis as a grab sample (representative of a single locality) or as a composite. Techniques for compositing subsamples are described in SOP-8, Random Sampling and Field Composites.

Solid substance samples collected during the field programs will be described in accordance with procedures outlined by the USDA Soil Conservation Service and the descriptions will be recorded in the appropriate field book and on Solid Substance Sampling Forms (Figure SOP-7.1).

SOIL SAMPLING

Soil samples will be collected by use of a scoop or trowel, sample trier, or soil bucket auger. The scoop is used to collect shallow soil samples, up to 3 inches in depth. The sampling trier and soil auger can be used to collect soil samples at depths greater than 3 inches. The trier is preferred to the auger for most sampling events. Samples taken with the trier tend to be less disturbed and allow for easier observation and characterization of the collected material. If the trier does not provide an adequate sample, then an auger will be used. The auger may also be used where larger sample volumes are required.
<table>
<thead>
<tr>
<th>SOLID SUBSTANCES SAMPLING FORM</th>
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<tbody>
<tr>
<td><strong>Project</strong></td>
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<tr>
<td><strong>Task No.</strong></td>
</tr>
<tr>
<td><strong>Site</strong></td>
</tr>
<tr>
<td><strong>Sample ID</strong></td>
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<td><strong>Sample ID</strong></td>
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**SUBSTANCE DESCRIPTION**

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Description</th>
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<tbody>
<tr>
<td>[ ] Soils</td>
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<tr>
<td></td>
<td></td>
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<tr>
<td>[ ] Sediments</td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>[ ] Other</td>
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</table>

**Sampling Procedures / Methods (Describe)**

<table>
<thead>
<tr>
<th>Sample Containers</th>
<th>Analyses</th>
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**Comments**

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</table>

(1) White - D&M Project File  
(2) Canary - D&M Duplicate  
(3) Pink - Originator
Soil samples will be collected using the following procedures:

1. Prepare a Solid Substance Sampling Form.

2. Clear surface vegetation, rocks, and other debris not designated for sampling from the sample point. Document the removal of this material.

3. Scoop or trowel - Collect sample with scoop or trowel.

   Trier - Insert a trier vertically and apply downward pressure. Rotate the trier to cut a core of material to the desired depth. Withdraw the trier and extract the core directly to a sample container or compositing bucket.

   Bucket Auger - A soil bucket auger consists of angled blades below a collection tube. As the auger is rotated clockwise by its T-handle, the soil is cut downward and is collected by carefully pulling the auger out of the hole. Auger until the desired depth is reached or the bucket portion is filled; then transfer the collected material to a sample container or compositing bucket. If the desired depth has not been reached, continue augering, emptying the auger as needed. If the material to be collected is too hard for the auger, a rock pick or spud bar will be used to collect the material.

4. Handle and preserve samples as outlined in SOP-9.

Sediments

Sediments will be sampled with the same equipment as soils. If the samples are under water as in pits, ponds, and lagoons, a pond sampler can be used. A pond sampler consists of a beaker attached to the end of a 12-foot telescoping aluminum tube. By combining several grab samples collected with the pond sampler, representative composite samples can be collected.

Where possible, a sludge sampler (similar to a soil trier) will be used to profile the sediment and collect samples up to 18-inches in depth. The sludge sampler consists of a plastic or steel tube which is shoved into the sediments. A cap or seal is then
placed over the top of the tube prior to extraction. The cap creates suction which helps to hold the sample material in the tube. Excess fluid will be decanted from sediment samples in the field.

Sand Tailings

The sand tailings will be sampled with soil sampling equipment, if possible. However, dry tailings may collapse easily and prevent all but shallow sampling with soil sampling equipment. In areas where the fine tailings exhibit high collapse potential, a standard hand-driven sampler will be used to collect samples. The following procedures will be used:

1. Drive one or two-inch diameter steel split tube sampler to desired depth. Extract sampling device.

2. Open tube, measure sample recovery and describe sample. If composite samples are required, consult SOP-7, Random Sampling and Field Composite.

3. Decontaminate sampler and backfill hole with excavated material.

4. Handle and preserve samples as outlined in SOP-9, Sample Handling and Preservation.

If the hand-driven sampler fails to provide the required samples, or cannot be driven to the depth needed, a drill rig (CME 45 or equivalent) may be used. Using the drill rig, the split-spoon sampler is usually driven 18 inches by a 140-pound hammer free-falling 30 inches (Standard Penetration Test). Sample removal and logging is the same as for hand-driven samplers.
Chat Piles

Chat is coarse to fine grained waste rock, composed primarily of chert. To characterize the upper surfaces of the chat piles, standard soil sampling equipment can be used (trowels, soil triers, shovels, etc.). Sampling the chat at depth may be accomplished using a split tube drive sampler. The sampler will be driven at an angle perpendicular to the bedding. Depending on the thickness of chat at a given location, a hand-driven tool or a split spoon driven by a drill rig or a backhoe may be used. With the drill rig, sampling at depth in even the very thick piles of chat is practical. Samples are collected with the slit-spoon at predetermined intervals (i.e., 2 or 5 feet) and mixed to form a composite (for that boring location) or retained as separate grab samples characterizing individual horizons.

In lieu of the drill rig, a backhoe may be employed to expose fresh chat for sampling. If the exposed material stands well, or at chat piles which are actively being quarried, the resulting steep face may allow sampling of the entire vertical dimension of the pile using "channel" sampling techniques. Using a sample scoop or trowel, a square or V-slot is cut into the material from top to bottom, taking care to keep the dimensions of the excavated slot consistent. All material cut from the slot is retained either in sample pans, buckets, or on plastic sheeting placed at the base of the work area. This material should then be thoroughly mixed and reduced, if necessary, using the sample splitter or cone and quarter methods (SOP-8, Random Sampling and Field Composites), prior to analysis.

Waste Rock (Development Rock)

The waste rock to be sampled consists of large blocks (greater than 6 inches in diameter) of shale and limestone, with little or no fines. A typical waste rock pile for a single excavated shaft
may be 30 feet in diameter and 20 feet high. To characterize the mineral content of the waste rock, a minimum of 15 large blocks will be collected. Each block will be struck with a sledge or rock pick to remove rock fragments, which will be collected on plastic sheeting. Approximately equal volumes of rock fragments will be removed from each of the blocks. The collected fragments will be crushed at the laboratory to minus 200. This material will be mixed and reduced, if necessary, using the sample splitter or cone and quarter methods.

DECONTAMINATION

Sampling equipment will be decontaminated between sample sites. Equipment will be washed with a warm detergent solution (e.g., Alconox), rinsed several times with potable water, triple rinsed with distilled water, and allowed to air dry. This procedure will be repeated as many times as necessary to remove visual staining. Equipment that cannot be decontaminated to the satisfaction of the sampling team will be replaced. Thorough decontamination will not be necessary between subsample stations for composite samples. Brushing or rubbing the sampling tool to remove material build-up is deemed adequate.
STANDARD OPERATING PROCEDURES NO. 8
RANDOM SAMPLING AND FIELD COMPOSITES

This SOP addresses procedures for collecting and processing solid substance samples when composite samples are called for. These procedures are to be used in conjunction with the basic solid substance sampling procedures described in SOP-7.

RANDOM SAMPLING

For large areas covered by a relatively homogenous material, such as sand tailings or chat, it is often times desirable to use random sampling methods to collect subsamples necessary to form a composite. Modified stratified random sampling methods will be used to provide representative subsamples. The procedure involves the division of the sampling area into about 10 equal-area polygons - each of these areas is further subdivided into six subareas to form a sample grid. A subarea is then randomly selected for sampling. The following procedure will be utilized:

1. Define the perimeter of the site and, if possible, construct a visible boundary.

2. By measurement or estimation, divide the site into about ten areas of approximately equal size. Show the site and these divisions on an existing map, and/or construct a map in the field logbook.

3. Assign a sequential number to each of the ten areas. Begin numbering in the northwest corner moving left to right and from north to south (see Figure SOP-8.1).

4. Divide each area into six numbered subareas. These will be the subsample locations within an area. Again, the numbering should begin in the northwest corner of the subarea and proceed left to right and from north to south. Place a surveyor's flag in the approximate center of each subarea.

5. Starting with area number 1, roll a die and record the result next to the area number. In this manner, a random number 1-6 will be generated for each of the ten areas.
LEGEND

1  Sub-Area Number

1  Area Number

- - - Sub-Area Boundaries

- - - Area Boundaries

Sampling Grid

DAMES & MOORE

Figure SOP-8.1
6. Consult the die roll result table (step 5) to determine in which subarea the subsample will be collected.

7. Go to the center of the selected subarea (marked with surveyor's flag - see step 4) and, using appropriate methods, collect a subsample for composite from that point.

In instances where it is desirable to collect a composite sample at a single site, the five-point method will be used to locate subsamples. The procedures are as follows:

1. Locate site to be sampled and mark center of area with surveyors flag.
2. Measure off subsample points at equal distances north, south, east and west of the center surveyor's flag.
3. Collect equal-volume subsamples at each compass-point location and at the center and combine in stainless steel bowl. Note procedures in logbook.
4. Collect representative composite sample and mix. If sample size is too large for containers, reduce volume using chute splitters or cone and quarter method.

FIELD COMPOSITING

Standardized field methods will be used to ensure that composite samples represent the sum of the various subsamples. Subsamples of approximately equal volume will be collected for compositing and placed in a stainless steel mixing bowl or plastic
compositing bucket. A trowel or spatula will be used to mix and disaggregate the sample. Following the initial mixing process in the bowls, the composite will be air dried (if required) then transferred to a portable sample splitter. A riffle or chute splitter (Gilson or equivalent) with stainless steel chutes and hopper will be used, following procedures listed below:

1. Pass entire composite through splitter three (3) separate times, retaining all split materials in pans. Material should be dry enough to pass easily through chutes.

2. Reduce sample quantity to desired volume by passing entire sample through splitter, retaining 50 percent of material on pan.

3. Dispose residuals as described in SOP No. 4, Handling and Disposal of Field Program Residuals.

4. Continue Step 2 process until material volume retained approximates sample volumes required.

5. Decontaminate sample splitter, material pans, and other associated sample equipment as described in SOP’s-3, 8, and 11.

CONE AND QUARTER

Samples which are agglomerating or are otherwise difficult to mix, will be reduced in size using the cone and quarter method. After the sample has been premixed in stainless steel bowls, and superfluous materials have been removed, the entire composite will be transferred onto a plastic sheet. The sample is mixed by alternately pulling one corner of the sheet over the opposite corner a minimum of 10 times. A conical heap is then formed and split in four similar parts by dividing with a spatula in a cross-shaped pattern. Two opposite quarters are removed and the remaining quarters are remixed. The quartering procedure is repeated until the required sample volume is obtained.
This procedure presents the requirements for handling, labeling, and transport of environmental and waste samples as well as field recording practices necessary for reconstruction of the sampling event.

SAMPLE HANDLING

The possession and handling of all samples collected will be traceable from the time of collection, through analysis, until final disposition. Documentation of the sample history is referred to as "chain-of-custody." Components of the chain-of-custody (sample seals, a field logbook, chain-of-custody record, and sample analysis request form) and procedures for their use are described in the following section.

A sample is considered to be under a person's custody if it is: 1) in a person's physical possession, 2) in view of the person after he or she has taken possession, 3) secured by the person so that no one can tamper with the sample, or 4) in a secure area. A person who has samples under custody must comply with the procedures described in the following sections.

Chain-of-Custody Records

To establish the documentation necessary to trace sample possession from the time of collection, a chain-of-custody record must be filled out in triplicate and must accompany every sample or group of individually identified samples. Chain-of-custody records will contain the following information:
Each person who has custody must sign the form. Samples cannot be left unattended unless they are secured and sealed.

Sample Labels

Sample labels are necessary to prevent misidentification of samples. Example sample labels are shown in Figure SOP-9.1. Gummed paper labels or tags will be used and will include at least the following information:

- Sample number (referenced to a sampling location) including a sample code that distinguishes field samples, duplicates, spikes, or blanks. The laboratory should not be cognizant of the code.
- Signature or initials of sample collector.
- Date and time of sample collection.
- Location of sample collection.
- Type of preservative used, or "None" as applicable.

Labels will be affixed to sample containers prior to or at the time of sampling. The labels will be filled out at the time of
<table>
<thead>
<tr>
<th>SOLID/WATER SAMPLE</th>
<th>Dames &amp; Moore</th>
</tr>
</thead>
<tbody>
<tr>
<td>Project #</td>
<td>Preserved:</td>
</tr>
<tr>
<td>Sample Id.</td>
<td>Location</td>
</tr>
<tr>
<td>Date</td>
<td>Time</td>
</tr>
<tr>
<td>By</td>
<td>HCL</td>
</tr>
<tr>
<td>Comments</td>
<td>HNO₃</td>
</tr>
<tr>
<td></td>
<td>H₂SO₄</td>
</tr>
<tr>
<td></td>
<td>NaOH</td>
</tr>
<tr>
<td></td>
<td>4°C</td>
</tr>
<tr>
<td></td>
<td>Other</td>
</tr>
<tr>
<td>Filtered</td>
<td>Unfiltered</td>
</tr>
</tbody>
</table>

**EXAMPLE SAMPLE LABEL**

Figure SOP-9.1
sample collection. The exact sample location and type of sample will be recorded in the field logbook.

All boxes used for archiving or sample storage will be labeled at the end and top with an indelible marking pen. Box labeling will include the following:

- Job Number (e.g., 20239-010)
- Location (e.g., Baxter Springs subsite)
- Site (e.g., 2-5-9)
- Matrix (e.g., soil)
- Depth Interval, If Applicable (e.g., 0-1 foot)
- Date (e.g., August 25, 1990)
- Disposition (e.g., Archived)

Sample Seals

Sample seals are used to detect improper handling of samples following sample collection up to the time of analysis. Items such as gummed paper seals and custody tape will be used for this purpose. An example sample seal is shown in Figure SOP-9.2. Signed and dated seals will be attached so that they must be broken to open either the individual sample containers or shipping containers. Seals will be affixed to containers before the samples leave the custody of the sampling personnel.

Sample Transport

Samples will be packaged and shipped according to U.S. Department of Transportation and EPA regulations. Samples will be delivered to the laboratory so that the requested analyses can be performed within the specified allowable holding time. Samples will be accompanied by the chain-of-custody record and by a sample analysis request form (Figures SOP-9.3 and SOP-9.4, respectively). The request form will list the variables to be analyzed by the
Chain of Custody Seal

Project No. ______________ Sample ID. No. ______________
Sampler (signature) ______________ Date __________ Time ______

EXAMPLE SAMPLE SEAL

Dames & Moore

Figure SOP-9.2
### Chain of Custody Record

**Dames & Moore**

<table>
<thead>
<tr>
<th>SPL NO</th>
<th>DATE</th>
<th>TIME</th>
<th>Sampling Point &amp; Sampling Method Description</th>
<th>Sample Material</th>
<th>Analysis</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tbody>
</table>

- **Relinquished By:** Organization  Date/Time
- **Received By:** Organization  Date/Time  Remarks

---

**Example Chain-of-Custody**

Figure SOP-9.3
### SAMPLE ANALYSIS REQUEST FORM

**Project:**

**Receiving Laboratory:**

**Sampling Contact:**

**Name**

**Phone**

**Attention:**

**Date Shipped:**

**Task Name/Code:**

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Date</th>
<th>Sample Description</th>
<th>Matrix/Preservatives</th>
<th>Requested Analysis</th>
<th>Charge No.*</th>
</tr>
</thead>
<tbody>
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</tr>
</tbody>
</table>

*Lab invoice shall show charges against each charge number.

(1) White - Lab Copy  (2) Canary - D&M Dup.  (3) Pink - D&M Proj. File  (4) Gold - Originator

Figure SOP-9.4
laboratory and total number and type of samples shipped for analysis. Authorized laboratory personnel will acknowledge receipt of shipment by signing and dating the form and returning a copy to the Dames & Moore Project Manager. The laboratory will also sign and date the airbill accompanying the samples and keep it on file with the sample information.

Field Logbook

Information pertinent to the sampling effort will be recorded in a field sampling logbook. The logbook shall be bound, with consecutively numbered pages. All entries will be made in indelible ink and all corrections will follow error correction protocol of one line through the error and initial and date of correction. Field personnel will also record all information on the appropriate sampling forms (Figures SOP-9.5 and SOP-9.6).

At a minimum, entries in a logbook shall include the following:

- Purpose of sampling.
- Location, description, and photographs of the sampling point.
- Details of the sampling site (for example, the elevation of the casing, casing diameter and depth, integrity of the casing, etc.).
- Name and address of field contact.
- Documentation of procedures for preparation of reagents or supplies which become an integral part of the sample (e.g., filters and absorbing reagents).
- Identification of sampling crew members.
SOLID SUBSTANCES SAMPLING FORM

<table>
<thead>
<tr>
<th>Date</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Project | Task No. | Site | Sample ID | Sampling Team Members | Location | Quantity |
<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

SUBSTANCE DESCRIPTION

Sample Type

- [ ] Soils
- [ ] Sediments
- [ ] Other

Sampling Procedures/Methods (describe)

Sample Containers | Analyses
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tr>
</tbody>
</table>

(1) White - D&M Proj. File   (2) Canary - D&M Dup.   (3) Pink - Originator

Figure SOP-9.5
# LIQUID SUBSTANCES SAMPLING FORM

**Project No.** ___________________________  **Date** ___________________________

**Team Member Initials** ___________________________  **Time** ___________________________

**Site** ___________________________  **Sample I.D.** ___________________________

**Site Description** ___________________________

---

**SUBSTANCE DESCRIPTION**

**Sample Type**  
( ) Stream  
( ) Seep  
( ) Pond  
( ) Well  
( ) Other

**Sampling Procedures/Methods (Describe)** ___________________________

**Comments** ___________________________

---

**PHYSICAL PARAMETERS**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td></td>
</tr>
<tr>
<td>Conductance</td>
<td></td>
</tr>
<tr>
<td>Corrected Conductance @ 25°C</td>
<td></td>
</tr>
<tr>
<td>Water Level/Flow Rate</td>
<td></td>
</tr>
<tr>
<td>Dissolved Oxygen</td>
<td></td>
</tr>
</tbody>
</table>

**Sample Containers**  
__________________________  
__________________________  
__________________________  

**Preservatives**  
__________________________  
__________________________  
__________________________  

**Analyses**  
__________________________  
__________________________  
__________________________  

---

*Figure SOP-9.6*
Type of sample (e.g., ground water, surface water, soil, chat, waste rock or tailings).

Suspected waste composition.

Number and volume of sample taken.

Sampling methodology.

Sample preservation.

Date and time of collection.

Collector's sample identification number(s).

Sample distribution and transportation method (e.g., name of the laboratory and cartage agent – Federal Express, United Parcel Service, etc.).

References such as maps of the sampling site.

Field observations.

Any field measurements made (e.g., pH, specific conductance, temperature and water depth).

Signature and date by the personnel responsible for observations.

Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a logbook. However, records shall contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The logbook shall be kept under strict chain-of-custody and stored in a location so as to make it accessible to the project QA manager.

SAMPLE PRESERVATION

All solid samples (soils, chat, waste rock, and tailings) will be collected in plastic bags with no preservative.
All liquid samples (ground water and surface water) will be collected in required sample containers and preserved as indicated in Table SOP-9.1.
### TABLE SOP-9.1
REQUIRED CONTAINERS AND PRESERVATIVES FOR WATER SAMPLES

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>CONTAINER</th>
<th>PRESERVATIVE</th>
<th>MAXIMUM HOLDING TIME (DAYS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Metal Hardness</td>
<td>1-Liter, Poly</td>
<td>HNO₃ to pH&lt;2</td>
<td>180</td>
</tr>
<tr>
<td>Dissolved Metals</td>
<td>1-Liter, Poly</td>
<td>Filter, HNO₃ to pH&lt;2</td>
<td>180</td>
</tr>
<tr>
<td>pH</td>
<td>500ml, Poly</td>
<td>4°C</td>
<td>ASAP</td>
</tr>
<tr>
<td>Specific Conductance</td>
<td></td>
<td></td>
<td>28</td>
</tr>
<tr>
<td>Alkalinity</td>
<td></td>
<td></td>
<td>14</td>
</tr>
<tr>
<td>TDS</td>
<td></td>
<td></td>
<td>7</td>
</tr>
<tr>
<td>TSS</td>
<td></td>
<td></td>
<td>7</td>
</tr>
<tr>
<td>Chloride Sulfate</td>
<td>100ml, Poly</td>
<td>4°C</td>
<td>28</td>
</tr>
<tr>
<td>Phosphate</td>
<td>100ml, Poly</td>
<td>H₂SO₄ to pH&lt;2</td>
<td>28</td>
</tr>
<tr>
<td>Gross Alpha, Radium</td>
<td>2 1-Liter, Poly</td>
<td>HNO₃ to pH&lt;2</td>
<td>180</td>
</tr>
</tbody>
</table>
APPENDIX A

EQUIPMENT MANUFACTURER'S SPECIFICATIONS
STANDARD OPERATION PROCEDURE NO. 10
CONTROL OF MEASURING AND TEST EQUIPMENT

Selection of measuring and test equipment for monitoring activities shall be consistent with the requirements of this plan to assure that such items are of proper type, range, accuracy, and tolerance to accomplish the specified monitoring function.

- Measuring the test equipment shall be calibrated, adjusted, and maintained at prescribed intervals against certified equipment having known valid relationships to nationally recognized standards. If no nationally recognized standards exist, the bases for calibration shall be documented.

- The method and interval of calibration for each item shall be defined, based on the type of equipment stability characteristics, required accuracy, intended use, and other conditions affecting measurement control. When measuring or test equipment are found to be out of calibration, an evaluation shall be made and documented of the validity of previous inspection or test results. Out-of-calibration devices shall be tagged or segregated and not used until they have been recalibrated. If any measuring or test equipment is consistently found to be out of calibration, it shall be repaired or replaced. A calibration shall be performed when the accuracy of the equipment is suspect. Calibration shall be performed in conformance with manufacturer's specification and/or as required by industry standards.

- Calibrated measuring and test equipment shall be identified by a serial number affixed in a suitable permanent manner, and with tags, labels, or stickers identifying the date of calibration and the next due date.

- A measuring and test equipment log shall be maintained to include the equipment/instrument identification, type of equipment instrument, date of last calibration, due date of next calibration, location or equipment/instrument.

- An equipment/instrument identification number shall be recorded on the appropriate documentation, at time of use.
Calibrated equipment/instruments shall be traceable, via identification number, to certificate of calibration.

Calibrated equipment/instruments shall be properly handled and stored to maintain accuracy.

When required, controlled environmental conditions for instruments will be adhered to and documented.
STANDARD OPERATION PLAN NO. 11
SURFACE WATER AND STREAMBED SEDIMENT SAMPLING

IDENTIFICATION OF SAMPLING LOCATIONS

Surface-water monitoring includes water-quality sampling and measurements of stream discharge (see SOP-12). Sampling and discharge measurements will be collected at the same general location in the stream channel. Stream characteristics influencing the choice of measuring/sampling location include:

- A section with a rock riffle or some other channel characteristic providing a section control.
- A relatively uniform cross section with water flowing at both banks.
- A section with a steady velocity distribution of which mixing of the water column is complete.

Sampling/measurement sections may be relocated upstream or downstream of the proposed sites to allow safer wading and/or more accurate streamflow measurements provided there are no obvious seep or tributary inflows that may adversely effect water quality sampling objectives. If field measurements and/or visual observations indicate that lateral or vertical mixing of water within the channel is not complete, the sampling site may be relocated upstream or downstream of the proposed site provided that water quality sampling objectives are not adversely affected.

WATER SAMPLING - METALS AND INORGANICS

Water will be collected using either the grab method or the depth integrated-equal width increment method (U.S.G.S. 1977). For seeps and smaller streams less than approximately 0.5 feet deep and 3-feet wide, a grab sample will be collected. If the channel is
more than 3-feet wide, several grab samples will be collected at equal widths across the channel and composited. The sample will be collected using a hand-held plastic scoop or bottle, depending on the depth of water, and composited into a narrow-mouthed plastic container. For ponded water, either a grab sample will be collected as described above or a weighted bottle sampler will be used, depending on the depth (EPA - SW-846).

For streams deep enough for reasonable depth integration (i.e. >0.5 feet deep), the samples will be collected using the depth integrated-equal width increment method at a minimum of 4 points across the channel. For streams which are wadable, samples will be collected using a one gallon (analytical quality) plastic bottle. The bottle will be hand-held and used in the same manner as prescribed for U.S.G.S. depth integrating sampling equipment (i.e., these will be depth integrated, equal width increment samples).

Samples will be collected during the high-flow (non-wadable) periods by suspending a U.S.G.S. model D-74 suspended sediment sampler from a bridge using a reel and crane. For tributary streams that are not wadable, samples will be collected from the right and left banks (grab) and composited. At sites where no bridge is present a single grab sample will be collected.

The sampler, whether hand held or suspended, will be lowered and raised at a constant rate without overfilling the sample bottle to obtain a representative depth integrated sample (i.e., these will be depth integrated, equal width increment samples). All samples from a section will be composited into a narrow-mouthed plastic container.

EQUIPMENT PROCEDURES

At most sites, both filtered and unfiltered samples will be
collected from the composite sample. Care will be taken to thoroughly agitate the composite container while pouring unfiltered water into the sample bottles for inorganic and metal analysis. Samples will be filtered at the time of collection through a 0.45 micron membrane filter using a geo-filter peristaltic pump and filter. The initial 100 to 200 ml of filtrate will be used to rinse the sampling system and sample bottles and will be discarded prior to actually obtaining a filtered sample.

Equipment decontamination will be performed between each sample. Equipment which comes in contact with potential sources of contamination during sampling will be discarded or decontaminated prior to sampling the next site. The pump hose will be decontaminated between each sample by triple rinsing with deionized water. For composite samples, the subsample and composite bottles will be thoroughly rinsed with the stream water to be sampled prior to obtaining a sample. Equipment such as sample hoses will be replaced when visual staining not removed by triple rinsing is observed. Other equipment will be washed with a mild laboratory detergent and a pH 4 nitric acid solution if necessary to remove staining.

Field measurements at most sites will include pH, dissolved oxygen, temperature, and specific conductance. Specific conductance, temperature, and dissolved oxygen will be measured in situ using YSI portable conductivity and dissolved oxygen meters. Field pH will be measured from the composited water sample using an Orion pH meter. If there is reason to suspect that water in the stream cross section is not thoroughly mixed, specific conductance measurements will be taken within ten feet of both the right and left banks, and the results recorded.

As a minimum, instruments will be calibrated at the beginning of each work day and at any time the calibration range is exceeded during measurements. The pH meter will be calibrated using a dual
point calibration (buffers pH 4 and pH 10). The conductivity meter will use a one point calibration with a certified standard. The dissolved oxygen meter will be calibrated at each site using a calibration chamber. Standard solutions will be labeled with their laboratory measured values and current shelf life. Calibration procedures for the field pH meter, conductivity meter and dissolved oxygen meter are included with the manufacturer’s equipment specifications (Appendix A).

STREAMBED SEDIMENT SAMPLING

An effort will be made to collect fine-grained streambed material found in depositional areas. A hand-held, 1-quart stainless steel scoop will be used to obtain streambed sediments at 3 to 6 points across the sampling section to obtain the required volume. Water will be decanted off the scoop samples, litter and all rocks greater than approximately 1/4-inch diameter will be removed, and the samples composited.

FIELD RECORDS

Information on the site location, sampling method, field measurements and other pertinent information will be recorded on liquid substance sampling form or solid substance sampling for at the time of sampling. This information along with additional sampling information pertaining to the site will also be documented in the field logbook.
STREAMFLOW MEASUREMENTS

Streamflows in the vicinity of the Baxter Springs/Treece Subsites could range from a few gallons per minute in small tributaries and drainage channels to several thousand cubic feet per second in the Spring River. This wide range of flow conditions will require a variety of measurement methodologies in order to quantify flow and constituent mass loadings.

The following five methods may be used to measure surface-water flow, depending on the hydraulic conditions and the accuracy required:

BUCKET AND STOPWATCH METHOD

This method will be used where channel geometry and flow rates allow a hand-held bucket to capture the entire streamflow over a time period of at least 5 seconds (approximately <0.1 cfs). The elapsed time to fill a measured volume is recorded and the flow rate computed. Measurement accuracy is relatively high. The method requires a graduated bucket and stopwatch.

PORTABLE FLUME

If the geometry of the channel is adequate, the flow is less than about 1.0 cubic feet per second and/or the depth is less than about 0.3 feet, a portable cut-throat flume may be installed in the channel to measure the flow. The flume will be leveled and the flow channelized through the throat of the flume. The water level on the flume staff will be recorded and the standard rating supplied with the flume will be used to calculate the discharge. The error at mid-stages on the staff is estimated to be about 10 percent (Baski Instruments, 1986).
SURFACE OR FLOATING STICK METHOD

This method will be used in open channels to approximate streamflow where the bucket and stopwatch, portable flume, and wading methods are not feasible. Under most conditions, the measurement error can be expected to range from about 10 to 25 percent (U.S.G.S. 1977). A representative section of channel with relatively uniform flow is chosen. Water depth is measured at several points across the channel in addition to the channel width. The cross-sectional area is computed and an appropriate length of channel measured. A floating object (stick, float, etc.) is placed in the center of the channel and the elapsed travel time over a measured distance recorded. This should be repeated five times and an average travel time computed. The average velocity (distance/time) is multiplied by a constant of 0.85 to convert surface velocity to a mean velocity in the vertical (U.S.G.S. 1977). Finally, discharge is computed using the equation \( Q = AV \), where \( Q \) = discharge in cubic feet per second, \( A \) = cross-sectional area in square feet, and \( V \) = velocity in feet per second.

WADING METHOD

Discharge measurements will be made by wading across the stream with a current meter and wading rod whenever possible. If the depth is less than about 0.3 feet, one of the aforementioned methods will be utilized to measure flow. For the wading method, the mid section method will be used and standard U.S. Geological Survey procedures will be followed in all note computations (see Buchanan and Somers, 1969). A measuring tape will be extended across the channel and staked perpendicular to the direction of flow. If possible, depth and velocity measurements will be made at a minimum of 20 verticals. However, if the stream width is such that the resulting distance between verticals is less than 0.3 feet for the pygmy-type and 0.5 feet for the AA-type current meters, then the number of verticals for the section will be reduced from
20. Depth will be measured using a top setting wading rod and velocity will be measured using either a AA or pygmy type current meter, depending on velocity and depth. In general, the pygmy-type meter will be used for shallow, lower velocity streams and the AA-type meter for deeper, higher velocity streams. The mean velocity in vertical will be determined using the 6/10 method (velocity measured at 0.6 of the depth from the water surface) for depths less than 2-1/2 feet and the two-point method (velocity measured at 0.2 and 0.8 of the depth from the water surface) for depth greater than 2-1/2 feet. The two point velocity measurements will be averaged to obtain the mean velocity in vertical for the two-point method. Measurements will be recorded on a Discharge Measurement Notes form and in the field logbook.

SUSPENSION METHOD

During high-flow conditions, a AA-type current meter will be suspended from a suitable bridge using a crane or bridge board, reel, and sounding weight. Standard U.S.G.S. methods will be used in all measurement procedures and note computations (see Buchanan and Somers, 1969). The mid-section method will be used and angle coefficients applied where necessary. A measuring tape will be extended across the bridge. Depth and velocity measurements will be taken at a minimum of 20 verticals across the channel, (see Wading Method). The mean velocity in vertical will be determined using the 6/10 method for depths less than 2-1/2 feet and the two-point method for depths greater than 2-1/2 feet (see Wading Method).

For current meter measurements, discharge will be computed by multiplying width times depth to obtain the cross sectional area of each vertical, times the mean velocity in vertical. The discharge of each vertical will be summed to obtain the total discharge for the section. Measurements will be recorded on the Discharge Measurement Notes form. Current meters will be cleaned, oiled, and
spin tested each day to assure precision.

Measurement/sampling cross sections may be relocated upstream or downstream of the proposed sites to allow a safer and/or more accurate measurement provided there are no seeps or tributaries that may adversely effect water-quality sampling objectives.
3.4.3.6 Field Conductivity Measurements

3.4.3.6.1 YSI Conductivity Meter

Item: S-C-T Meter  
Model: 33  
Dames & Moore No.: 124, 12-33-773, 12-33-550, 12-33-340

Operational Procedures

1. Adjust meter zero by turning the bakelite screw on the face to zero.

2. Adjust the meter by turning the switch to red line and adjusting the meter needle with the red line control. If the batteries are low, the machine will not red line.

3. Plug the probe into the probe jack on the side of the instrument and place the probe into the solution to be measured.

4. Set the main switch to temperature and allow a short time for equilibrium to occur between the probe temperature and the solution temperature. The temperature may be read on the bottom scale of the meter in degrees Celsius.

5. For the salinity readings, the temperature from step 4 is transferred to the TEMPERATURE SALINITY knob on the instrument.

6. The instrument is switched to "Salinity" and is read on the red 0-40 percent meter range.

7. Conductivity is read after switching to the CONDUCTANCE position. The range knob is switched to the highest setting, x100 \( \mu \text{mhos/cm} \) and if the reading is below 50 on the 0-500 meter scale, the machine is switched to the next lower range, x10 \( \mu \text{mhos/cm} \). If the reading is still below 50, the instrument must be switched to the lowest scale, x1 \( \mu \text{mhos/cm} \).

8. To check the condition of the probe the "cell test" is performed by switching the meter to x100 or x10 meter range conductance and depressing the cell test button. If deflection is greater than 2 percent of the reading, the probe is fouled.

Calibration

Calibration of the conductivity meter can be checked using a standard solution. The standard solution is made with KCL according to methods in Standard Methods, Sec. 154 (APHA, 1971).
Detection Limit: 1.0 umhos/cm

Booklet YSI Model 33 S-C-T meter should be consulted if additional operational information is needed.

3.4.3.7 Field Dissolved Oxygen (DO) and Temperature Measurements

3.4.3.7.1 YSI DO Meter

Item: Dissolved Oxygen Meter
Model: 51A, 54C
Dames & Moore No.: 4319, 4893, 3286

Operational Procedures

1. Prepare probe as illustrated in probe preparation procedures given in Figure 3-24.
2. Connect the two probe jacks to the instrument.
3. With the instrument turned off, check the mechanical zero of the meter (pointer should indicate zero). Adjust with the screw on the front of meter if necessary. Recheck when the position of the instrument has changed.
4. Switch to the RED LINE position and adjust the meter to red line with the front panel control.
5. Place the probe in the calibration medium (calibration procedures are in the following paragraphs).
6. Switch to the TEMP position and read the temperature when the meter is steady.
7. Switch to the ZERO position and adjust the meter to zero with the ZERO control.
8. Switch to the 0-10 or 0-20 ppm position and calibrate the instrument with the CAL. control.

Calibration:

1. Calibration can be accomplished in the following way. The probe is placed in water of a known dissolved oxygen concentration. Proceed from operational procedures, steps 5 through 8.

The probe must be agitated to prevent local oxygen depletion around the cathode of the probe. Adjust the CAL control to the reading of the oxygen concentration in the sample. The known (calibrated) value for saturated water at the particular air pressure and temperature is found in Table 3-7.
PROPER MEMBRANE MOUNTING

1. WITH EYEDROPPER FILL WITH KCL SOLUTION - ALSO WET "O" RING GROOVE
   A. GRASP THREADED SECTION BETWEEN THUMB AND FOREFINGER
   B. SECURE ONE END OF MEMBRANE UNDER THUMB

2. NOT LIKE THIS!
   WITH RIGHT HAND GRASP FREE END OF MEMBRANE


4. SECURE THE MEMBRANE END UNDER THE LEFT FOREFINGER - INSPECT

5. THE MEMBRANE SHOULD BE TIGHT AND SMOOTH - NO TRAPPED AIR BUBBLES SLIP ON "O" RING

6. - FINAL STEP - TRIM OFF EXCESS MEMBRANE NEAR "O" RING TEMPERATURE SENSOR MUST BE EXPOSED FOR RAPID RESPONSE

Figure 3-24. Probe Preparation Procedures
## Table 3-7. Solubility of Oxygen in Water (Saturated with Air) in PPM at Various Temperatures and Pressures

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The probes described in this instruction sheet are designed for direct use with YSI Models 51B, 54ABP, 54ARC and 57 Dissolved Oxygen Meters. The probes can also be used with discontinued YSI Models 51A, 548P and 54RC Dissolved Oxygen Meters when the YSI 5735 Cable Adaptor is employed (See Accessories).

I. PRINCIPLE OF OPERATION

Each YSI 5700 Series Probe is a complete polarographic system in itself. A thin permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow. The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure under the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

II. SPECIFICATIONS

- Cathode — Gold
- Anode — Silver
- Membrane — .001" FEP Teflon (.0005" FEP Teflon available)
- Electrolyte — Half saturated KCl
- Temperature Compensation — (See instrument specifications)
- Pressure Compensation — effective to 1/2% of reading over a 100 psi range (230 ft. water)
- Response Time — 90% DO value in 10 seconds
- Polarizing Voltage — 0.8 volts nominal
- Probe Current — Air at 30°C = 19 microamps nominal
- Nitrogen at 30°C = .15 microamps or less

ACCESSORIES AND REPLACEMENT PARTS

- YSI 5986 — Diaphragm Kit
- YSI 5775 — Membrane and KCl Kit, Standard — includes 2 each 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle KCl with Kodak Photo Flo.
- YSI 5776 — Membrane and KCl Kit, High Sensitivity — includes 2 each 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle KCl with Kodak Photo Flo.
- YSI 5945 — "O" ring pack — contains replacement "O" rings for all YSI 5700 Series Probes.

Detachable cable:

- YSI 5740-10 = 10' cable
- YSI 5740-25 = 25' cable
- YSI 5740-50 = 50' cable
- YSI 5740-100 = 100' cable
- YSI 5740-150 = 150' cable
- YSI 5740-200 = 200' cable

YSI 5735 — Cable Adaptor to mate YSI 5700 Series Probes with discontinued YSI Models 51A, 548P and 54RC Dissolved Oxygen Meters.

YSI 5486 — Beater Boot Assembly for YSI 5720A Probe.

III. YSI 5739 DISSOLVED OXYGEN PROBE

The YSI 5739 probe, with built-in lead weight, is an improved design that replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes. (See Figure 1.)

The complete probe consists of the YSI 5739 probe body plus a YSI 5740 detachable cable. The detachable cable is a convenience feature that facilitates changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is not designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged, remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Figure 1

PRICE INCLUDING HANDLING $1.00
Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, hand tight.

NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

PRESSURE COMPENSATION

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft. water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum.

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. Large accumulation of salt crystals around the diaphragm plug may be due to a poorly tightened plug or dirt underneath the diaphragm. Cleaning the parts in water and re-tightening may be tried before diaphragm replacement. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm. Flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

PROBE SCHEMATIC

Figure 2

IV. YSI 5720A B.O.D. BOTTLE PROBE

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5720 B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 117VAC (95-135VAC, 50/60 Hz) or 230VAC (190-250VAC, 50/60 Hz) operation. (See Figure 3.)

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. Bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. Bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

Caution: The motor housing on this probe is not waterproof. Do not submerge beyond the part that is inserted into a BOD bottle.

STIRRER BOOT

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 4.)

Boot replacement is as follows:
1. Pull off old assembly and clean shaft.
2. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

V. YSI 5750 B.O.D. BOTTLE PROBE

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 5.)
VI. PROBE PREPARATION AND CARE

1. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.

2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCI solution.

3. Fill the probe with electrolyte as follows: ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS
   A. Grasp the probe in your left hand. (See Figure 6.) When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.
   B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
   C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
   D. Using a continuous motion STRETCH the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe.
   E. Secure the end of the membrane under the forefinger of the hand holding the probe.
   F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
   G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

4. Shake off excess KCl and reinstall the sensor guard.

5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The YSI 5720A and 5750 probes can be stored in a B.O.D. bottle containing about 1" of water.

6. Membranes will last indefinitely if properly installed and treated with care during use. The result of poor membrane application or damage is erratic readings. The cause of erratic behavior can be loose, wrinkled or fouled membranes (by algae for example), or bubbles in the probe from electrolyte loss. If any of these signs occur it is good practice to thoroughly flush the reservoir with new KCI and replace the membrane.

7. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCI and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.

8. The gold cathode should always be bright and untarnished. To clean, wipe with a clean lint-free cloth or hard paper. NEVER USE ANY FORM OF ABRASIVE OR CHEMICAL. Rinse the sensor several times with KCI, refill, and install a new membrane.

9. Some gases contaminate the sensor, evidenced by discoloration of the gold. If the tarnish cannot be removed by vigorous wiping with a soft cloth, lab wipe, or hard paper, return the probe to the factory for service.

10. H, S, SO₃, Halogens, Neon, and CO are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause.

These gases have been tested for response.

- 100% Carbon Monoxide: Less than 1%
- 100% Carbon Dioxide: around 1%
- 100% Hydrogen: Less than 1%
- 100% Chlorine: 2/3 O₂ response
- 100% Helium: none
- 100% Nitrous Oxide: 1/3 O₂ response
- 100% Ethylene: none
- 100% Nitric Oxide: 1/3 O₂ response

11. If the probe has been operated for extended periods with a loose or wrinkled membrane the gold cathode may become plated with silver. In this event return the probe to the factory for refinishing.

VII. WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt serving on all YSI products.

Yellow Springs Instrument Co., Inc.
Service Department
P.O. Box 279
Yellow Springs, Ohio 45387, U.S.A.

Phone: 513-767-7241
The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4½ foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from the probe membrane.

For maximum accuracy, wet the inside of the calibration chamber with fresh water. This creates a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the air-calibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5'/second) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.
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GENERAL INFORMATION

Introduction

The ORION SA 250 pH Meter is an advanced, portable pH meter with many features previously found only on bench-top meters.

Automated functions such as autocalibration, prompting, automatic temperature compensation, and diagnostic operator assistance codes make the SA 250 Meter very easy to use.

With your choice of .1 or .01 pH resolution, you decide if you need speed or better accuracy.

The meter comes with a ROSS Combination pH Electrode and ATC probe for fast accurate results, no matter what the temperature difference between samples and standards.

Created for the outdoor, process, or active lab environment, the meter is lightweight, with a no-slip grip, large LCD display and has a rugged splash and dust-resistant housing. The SA 250 Meter meets or exceeds tests to Department of Transportation and Mil specs for shock, vibration, and moisture.

See Figure 1.

Packing List

Cat. No. 025000 contains:

<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Item</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>917001</td>
<td>ATC probe</td>
<td>1</td>
</tr>
<tr>
<td>815600</td>
<td>Model 81-56 ROSS pH Electrode</td>
<td>1</td>
</tr>
<tr>
<td>020044</td>
<td>Three plastic bottles, one plastic beaker</td>
<td>1</td>
</tr>
<tr>
<td>020043</td>
<td>Support rod and clamp</td>
<td>1</td>
</tr>
<tr>
<td>020042</td>
<td>Carrying case</td>
<td>1</td>
</tr>
<tr>
<td>910002</td>
<td>Electrode holder</td>
<td>1</td>
</tr>
<tr>
<td>9 Volt</td>
<td>battery</td>
<td>1</td>
</tr>
<tr>
<td>0250IM</td>
<td>Instruction manual and training guide</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>Warranty card</td>
<td>1</td>
</tr>
</tbody>
</table>

See Figure 2.

In order to operate the SA 250 Meter without a battery, a line converter will also be needed.

<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Item</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>020120</td>
<td>Line converter, 110V AC</td>
<td>1</td>
</tr>
<tr>
<td>020121</td>
<td>Line converter, 220V AC</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 1
Model SA 250 pH Meter

Figure 2
Model SA 250 pH Meter and Accessories
INSTRUMENT DESCRIPTION

Refer to Figure 3

1. **ON/OFF Switch**: Controls power to the meter. Memory is maintained even when the instrument is turned off.

2. **LCD Display**: SA 250 pH Meter automatically displays data on an easy-to-read 3 1/2 digit LCD.

3. **Mode Switch**: Used to select mV, temp, pH .1, or pH .01 modes.

4. **Keys**: Eight touch keys are used to control the meter. Each key is labelled as to the function performed. The following table summarizes the function of each key:

<table>
<thead>
<tr>
<th>Key</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>sample</td>
<td>Press to display pH of the sample.</td>
</tr>
<tr>
<td>cal</td>
<td>Press to start the calibration sequence.</td>
</tr>
<tr>
<td>iso</td>
<td>Press to display current isopotential point.</td>
</tr>
<tr>
<td>slope</td>
<td>Press to display slope in percent of theoretical.</td>
</tr>
</tbody>
</table>

   The sample, cal, iso, and slope keys function only while the mode control is in either pH .1 or pH .01.

5. **Enter**: Press to enter a value into the meter memory.

   The following keys, X10, ↑, ↓, are used to change the numeric display. This process is called scrolling.

   X10: Increases the displayed value to the next decade — for example: pH 6.14 would increase to 7.00. At the upper end of the scale pressing X10 will cause the display to wrap around — for example: pH 19.00 would go to −2.00.

   X10 works when the mode switch is in either pH .1 or pH .01 and only for slope, iso or cal adjustments.

   ↑ up: Increases the value displayed by increments equal to the least significant digit.

   ↓ down: Decreases the value displayed by increments equal to the least significant digit.

   If the ↑ or ↓ key is kept depressed, the next significant digit will change. The ↑ and ↓ keys work in the temp, pH .1 or pH .01 modes.

5. **Electrode Input**: Accepts BNC connector from combination or sensing electrodes. A separate pin tip input accepts reference electrodes.


7. **Line Converter Jack**: Accepts an AC line converter for use without batteries.

---

Figure 3
SA 250 Meter Controls
INSTRUMENT SET-UP

Support Rod

See Figure 4.

1. Attach support rod base to side of meter carrying case and tighten clamp screw.
2. Insert support rod into base. Tighten rod by turning clockwise.
3. Attach electrode holder to top of support rod.

Power Source

The ORION SA 250 pH Meter operates on one 9 volt nonrechargeable alkaline battery. If the SA 250 pH Meter is left on while using battery power, there will be approximately 30 hours of continuous life. Optional AC line converters are available for both 110 and 220 volt mains. Refer to ACCESSORIES, page 12.

Battery Installation

See Figure 5.

1. Remove access panel on back of meter, by sliding cover towards bottom of meter.
2. Attach battery connector clip to battery terminals, install battery and replace access panel.

Figure 4
Support Rod and Clamp

Figure 5
Rear Access Panel Removed
Meter Check Out Procedure

1. Slide power switch to ON position. Attach BNC Shorting Plug (ORION Cat. No. 090045) to BNC connector on top of meter. Refer to Figure 6.

2. If using optional AC line converter, connect it to meter and appropriate power source. Proceed to step 4.

3. If LO BAT indicator on LCD remains on, the battery must be replaced.

4. Slide mode switch to mV. Display should read 0 ± 0.3.

5. Slide mode switch to temp. Display should read 25.0. If 25.0 is not displayed, scroll, using \ and X10 keys, until 25.0 is displayed and press enter.

6. Slide mode switch to pH .01. Press iso. Display should read the letters ISO then a value of 7.00. If 7.00 is not displayed, scroll until 7.00 is displayed and press enter.

7. Press slope. Display should read the letters SLP then a value of 100.0. If 100.0 is not displayed, scroll until 100.0 is displayed and press enter.

8. Press sample. Observe the letters pH then a steady reading of 7.00 ± 0.02 should be obtained. If not, press cal and scroll until 7.00 is displayed and press enter. Press sample and observe a reading of 7.00.

9. Remove the shorting plug. After a successful completion of steps 1-8 the meter is ready to use with an electrode.

Electrode Connections
Refer to Figure 6.

1. Attach electrodes with BNC connectors to sensor input by sliding connector onto input, pushing down and turning clockwise to lock into position. Connect reference electrodes with pin tip connectors by pushing connector straight into reference input.

NOTE: If using a combination electrode with a BNC connector the reference pin tip jack is not used (4 in Figure 6).
pH Measurements

See Figure 7.

A calibration with one or two buffers should be performed before pH is measured. It is recommended that a calibration with two buffers be performed at the beginning of each day to determine the correct slope of the electrode. This serves the dual purpose of determining if the electrode is working properly and storing the slope value in the meter’s memory. Perform a one buffer calibration every two hours to compensate for electrode drift.

Check the stored value for ISO before calibration. Unless the isopotential point of the electrode is known verify that the display reads 7.00. If not, scroll until 7.00 is displayed and press enter. See Isopotential Point, page 8.

There are two ways of calibrating the SA 250 Meter, autocalibration or manual calibration.

NOTE: It is recommended to select either autocalibration or manual calibration and not use a combination of the two methods. Following is a description and instructions for each method.

Autocalibration

Autocalibration is a feature of the SA 250 Meter that automatically recognizes the 7.00, 4.01 and 10.01 buffers with a range of ± 0.5 pH units. The user waits until the pH display is stable and presses enter. The SA 250 Meter automatically calibrates to the correct buffer value using temperature compensation. Do not scroll when using autocalibration.

While calibrating, the SA 250 Meter compares actual values to theoretical values to determine if the buffer is within range. Buffers greater than ± 0.5 pH units from the correct value will trigger an operator assistance code.

It is recommended that an ATC probe be used for autocalibration. If an ATC probe is not used, all samples and buffers should be at the same temperature or use manual temperature compensation. See Temperature Mode, on page 7.
Autocalibration With Two Buffers

1. Connect electrode(s) to meter. Slide the mode switch to either pH .1 or pH .01. Choose either 4.01 and 7.00, or 7.00 and 10.01 buffers, whichever will bracket your expected sample range.

2. Place electrode(s) into either 4.01, 7.00 or 10.01 buffer.

3. Press cal. The display will alternate between .1 and the pH value of the buffer, indicating this is the first buffer and a value has not been entered. Wait for a stable pH display and press enter. The correct display will freeze for 3 seconds then advance to .2, indicating the meter is ready for the second buffer.

4. Rinse electrode(s) and place into a second buffer. Wait for a stable pH display and press enter.

5. Rinse electrode(s), place into sample. Record pH directly from the meter's display.

Autocalibration With One Buffer

1. Check slope term by pressing slope. If necessary, scroll and enter the correct value. If slope value is unknown, either enter 100.0 or perform a two buffer calibration. A single buffer calibration does not change the slope term.

2. Connect electrode(s) to meter. Slide mode switch to either pH .1 or pH .01.

3. Place electrodes into either 4.01, 7.00 or 10.01 buffer.

4. Press cal. The display will alternate between .1 and the pH value of the buffer, indicating this is the first buffer and a value has not been entered.

5. Wait for a stable pH reading and press enter.

Manually Calibration

To calibrate with buffers other than 4.01, 7.00 or 10.01, use the manual calibration technique. The calibration sequence is the same as autocalibration, except buffer values are scrolled in.

It is recommended that an ATC probe be used. If an ATC probe is not used, all samples and buffers should be the same temperature or use manual temperature compensation. See Temperature Mode.

Please note that even if the correct pH display appears, the user needs to scroll for manual calibration or the meter will assume that autocalibration is to be used.

Manual Calibration With Two Buffers

1. Connect electrode(s) to meter. Slide mode switch to either pH .1 or pH .01. Choose two buffers that will bracket your expected sample range.

2. Place electrode(s) into the first buffer.

3. Press cal. The display will alternate between .1 and the pH value of the buffer, indicating this is the first buffer and a value has not been entered.


5. Rinse electrode(s) and place into the second buffer. Wait for a stable pH display. Scroll in the correct value and press enter.

6. Rinse electrode(s) and place into sample. Read pH directly from the meter's display.
Temperature Mode

Sliding the mode switch to temp will display the temperature in °C. When the temperature is outside of the operating range — 5.0 to 105.0°C, an operator assistance code will be displayed, E-1 for below -5°C, or E+1 for above 105°C.

During a calibration or sample measurement, the mode switch can be changed to temp. When an ATC probe is connected the temperature can be monitored and automatic temperature compensation will take place.

To use manual temperature compensation:

1. Using a thermometer accurate to ± 1°C determine the temperature of the solutions to be measured.

2. Slide mode switch to temp.

3. Scroll, using A, V or X10 keys, until the correct temperature value is displayed.

4. Press enter.

5. Return mode switch to either pH .1 or pH .01.

When an ATC probe is not connected, the last entered value of temperature is displayed. If a temperature value has not been entered since the removal of an ATC probe, a default value of 25°C is displayed.

Manual Calibration With One Buffer

1. Verify slope by pressing slope. If necessary scroll in correct value, using V, V and X10 keys, and press enter. If correct slope is unknown, either enter 100.0 or perform a two buffer calibration.

2. Connect electrode(s) to meter. Slide mode switch to either pH .1 or pH .01.

3. Place electrodes into the buffer.

4. Press cal. The display will alternate between 1. and the pH value of the standard, indicating this is the first buffer and a value has not been entered.

Wait for a stable pH display, scroll until the correct value is displayed and press enter.

The display will freeze for 3 seconds then advance to .2, indicating the meter is ready for the second buffer. By pressing sample the letters PH will be displayed. The meter is now calibrated and automatically advances to sample mode.

5. Rinse electrode(s) and place into sample. Read the pH directly from the display.

NOTE FOR MANUAL CALIBRATION: In the event that scrolling was started but the value was not entered and the mode switch was changed, either a P1 or a P2 will be displayed upon returning to the pH mode. P1 indicates that a value has not been entered for the first buffer while P2 indicates a value has not been entered for the second buffer.

Slope

By pressing the slope key the slope is displayed as a percent of theoretical. A properly functioning electrode will have a 92% to 102% slope. See Troubleshooting, page 10 if the slope is out of range. The slope value is retained in the meter's memory until another two buffer calibration is performed or another value is entered. A one buffer calibration does not change the slope value.

At the beginning of each day and every time a different electrode is used a two buffer calibration should be performed for accurate measurements.

To enter a slope value:

1. Slide the mode switch to either pH .1 or pH .01.

2. Press the slope key.

3. Scroll, using A, V or X10 keys, until the correct value is displayed.

4. Press enter.
Isopotential Point

The isopotential point is the pH at which the potential (mV) of the electrode will not vary with temperature. For the majority of pH electrodes the isopotential point is pH 7.00. There are some exceptions where the operating range used for a particular electrode is primarily at one end of the pH scale.

If your pH electrode has an isopotential point other than 7.00, the correct value may be entered as follows:

1. Slide mode switch to pH .1. or pH .01.
2. Press iso.
3. Scroll, using ∨, ∨, or X10 keys, until correct value is displayed.
4. Press enter.

A two buffer calibration should be performed after an isopotential point value has been changed. It is good practice to verify the isopotential point whenever the meter has been turned on.

Potentiometric Measurements

Potentiometric titrations are performed in mV mode using either pH, ion selective or redox electrodes. Detailed instructions for any ORION Electrode are given in the electrode instruction manual. Titration instructions are included in ORION Redox Electrode (Model 96-78 or 97-75) Instruction Manual, or in standard analytical chemistry texts. Electrodes that have an U.S. Standard Connector need an U.S. Standard to BNC Adapter which are available from Orion (Cat. No. 090033).

Dissolved Oxygen Measurements

Dissolved oxygen measurements are displayed in ppm when ORION Model 97-08 Dissolved Oxygen Electrode is used with ORION SA 250 Meter. Follow these instructions for calibrating the electrode.

1. Connect the Model 970899 to meter and leave electrode mode switch “off”.
2. Do not use an ATC probe.
3. Set the mode switch of the SA 250 Meter to temp and scroll in 25.0°C, press enter.
4. Set the mode switch to pH .1. or pH .01.
5. Press the slope key. Scroll until the value 100.0 appears and press enter.
6. Press the iso key and verify that it is 7.00. If not, scroll in the value 7.00 and press enter.
7. Press the cal key. Scroll in the value 7.00 and press enter.
8. Press sample.
9. Turn the mode switch on the electrode to BT CK. Good battery operation is indicated by a reading of 13.00 or greater on the meter.
10. Turn the mode switch on the electrode to ZERO. Use the zero calibration control to set the meter to read 0.00.
11. Insert the reservoir (funnel) into a BOD sample bottle containing enough water to just cover the bottom. Insert the electrode, making sure that the electrode tip is not immersed in the water and does not have water droplets clinging to the outside of the membrane. Let stand approximately 30 minutes to ensure water saturation of air in BOD bottle. This bottle should be used for electrode storage between measurements.
12. Turn the electrode mode switch to the AIR position. If measurements are being made at sea level, use the AIR calibration control to set the pH meter reading to the prevailing barometric pressure in mm Hg (divided by 100). If the barometric pressure is unknown, if the elevation is above sea level or if the sample has a salinity greater than 2 parts per thousand, consult Table 1 found in the 97-08 Instruction Manual to obtain the correct AIR setting.
13. Turn electrode mode switch to H₂O for sample analysis.
Operator assistance codes are used to inform the user of an out of range value. The following table outlines the operator assistance codes that are available in the SA 250 pH Meter and suggests a remedy. The table is divided according to the modes of the meter.

<table>
<thead>
<tr>
<th>Code</th>
<th>Problem</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>M V MODE</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 1</td>
<td>mV out of range.</td>
<td>1. If occurs when electrodes are out of solution, code will disappear when electrodes are returned to solution.</td>
</tr>
<tr>
<td>E-1</td>
<td></td>
<td>2. Verify electrodes are properly connected and filled.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Dilute standards or samples.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4. Review calibration and operating procedures.</td>
</tr>
<tr>
<td><strong>TEMP MODE</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 1</td>
<td>Temp out of range.</td>
<td>1. Verify ATC probe is properly connected.</td>
</tr>
<tr>
<td>E-1</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>pH .1 or pH .01 MODES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>while in sample function</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 1</td>
<td>mV, temp or pH out of range.</td>
<td>1. Go to mV mode and check. If mV is out of range, perform remedy steps described above for mV mode.</td>
</tr>
<tr>
<td>E-1</td>
<td></td>
<td>2. Go to temp mode and check. If temp is out of range, perform remedy steps described above for temp mode.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Check the sample.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4. Check slope and iso values.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5. Re-calibrate.</td>
</tr>
<tr>
<td>While in cal function:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 1</td>
<td>mV, temp or pH out of range.</td>
<td>1. Go to mV mode and check. If mV is out of range, perform remedy steps described above for mV mode.</td>
</tr>
<tr>
<td>E-1</td>
<td></td>
<td>2. Go to temp mode and check. If temp is out of range, perform remedy steps described above for temp mode.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Check the buffer.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4. Check iso value.</td>
</tr>
<tr>
<td>E21</td>
<td>Slope not in the range 80.0 to 100.0%</td>
<td>1. Press enter to acknowledge code and repeat calibration using fresh buffers.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Clean electrode and refill reference.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Refer to electrode instruction manual for check out.</td>
</tr>
<tr>
<td>E31</td>
<td>First cal point out.</td>
<td>1. Press enter and repeat calibration using fresh buffers.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Check iso, slope, and temp values.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Verify electrodes are properly connected.</td>
</tr>
<tr>
<td>E35</td>
<td>pH Autocalibration error.</td>
<td>1. Press enter and repeat calibration using fresh buffers.</td>
</tr>
<tr>
<td>E36</td>
<td>Electrode voltage being measured is greater than ± 0.5 pH units from nominal value for the pH buffer.</td>
<td>2. Clean electrodes and refill reference. Re-calibrate.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Perform a manual calibration. Certain electrodes may operate out of acceptable range.</td>
</tr>
</tbody>
</table>
# TROUBLESHOOTING GUIDE

<table>
<thead>
<tr>
<th>Malfunction</th>
<th>Possible Cause</th>
<th>Remedy</th>
</tr>
</thead>
</table>
| No Display  | No power to meter | 1. Check that switch is in ON position.  
2. Replace battery.  
3. Check that adapter is receiving power and is plugged in securely. |
| Erratic readings or reading out of range | Electrode failure | 1. Follow instructions in instruction manual.  
2. Follow meter check out procedure. |
| Unable to calibrate | Isopotential error | 1. Verify iso. |
| Unable to calibrate in automatic calibration | Certain electrodes may operate outside the limits of ±0.5 pH units. | 1. Try scrolling value to within range and press enter to cal if E1 or E-1 appearing.  
2. Check temp, slope, and iso and repeat. |

Orion Technical Service Personnel can be consulted for troubleshooting advice by calling 800-225-1480 or 617-864-5400. Outside North America contact your local authorized Orion Representative.
INSTRUMENT WARRANTY

ORION RESEARCH INCORPORATED warrants this instrument will operate for one year from the date of purchase when used under normal laboratory conditions, and in accordance with the operating limitations and maintenance procedures given in the instruction manual. In the event of failure within the warrant period, Orion or its Authorized Dealer, will, at Orion's option, repair or replace the non-conforming instrument at no charge to the customer.

THE WARRANTY DESCRIBED ABOVE IS EXCLUSIVE AND IN LIEU OF ANY OTHER WARRANTY, WHETHER STATUTORY, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO, ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE AND ALL WARRANTIES ARISING FROM COURSE OF DEALING OR USAGE OF TRADE, EXCEPT TITLE. THE BUYER'S SOLE AND EXCLUSIVE REMEDY IS FOR REPAIR, OR REPLACEMENT OF THE DEFECTIVE INSTRUMENT OR PART, OR REFUND OF THE PURCHASE PRICE; BUT IN NO EVENT SHALL ORION (ITS CONTRACTORS AND SUPPLIERS OF ANY TIER) BE LIABLE TO THE BUYER OR ANY PERSON, IN CONTRACT OR IN TORT (INCLUDING NEGLIGENCE) FOR SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES.

Representations and warranties made by any person, including dealers, representatives and employees of Orion, which are inconsistent or in conflict with the terms of this warranty shall not be binding upon Orion unless in writing and signed by one of its officers.

REPAIR AND SERVICE

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

Consult your authorized Orion dealer, or:

ORION RESEARCH INCORPORATED
840 Memorial Drive
Cambridge, Massachusetts 02139
TELEPHONE 617-864-5400
TELEX 921466

In Europe, the Middle East, and Africa contact your authorized Orion dealer, or:

ORION RESEARCH AG
Fähnlibrunnenstrasse 3
CH-8700 Kusnacht, Switzerland
TELEPHONE 01-910-7858
TELEX 57829
### OPTIONAL ACCESSORIES

<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>9678BN</td>
<td>Redox electrode</td>
</tr>
<tr>
<td>970899</td>
<td>Dissolved oxygen electrode</td>
</tr>
<tr>
<td>910004</td>
<td>pH 4 buffer packets, box of 25 packets, each packet making 200 ml of buffer</td>
</tr>
<tr>
<td>910007</td>
<td>pH 7 buffer packets, box of 25 packets, each packet making 200 ml of buffer</td>
</tr>
<tr>
<td>910009</td>
<td>pH 9 buffer packets, box of 25 packets, each packet making 200 ml of buffer</td>
</tr>
<tr>
<td>910104</td>
<td>pH 4.01 buffer, 475 ml bottle</td>
</tr>
<tr>
<td>910107</td>
<td>pH 7.00 buffer, 475 ml bottle</td>
</tr>
<tr>
<td>910110</td>
<td>pH 10.01 buffer, 475 ml bottle</td>
</tr>
<tr>
<td>910002</td>
<td>Electrode holder</td>
</tr>
<tr>
<td>917001</td>
<td>Automatic temperature compensator — epoxy outer body</td>
</tr>
<tr>
<td>917002</td>
<td>Automatic temperature compensator — glass outer body</td>
</tr>
<tr>
<td>09C033</td>
<td>U.S. Standard electrode to BNC meter connector adapter</td>
</tr>
<tr>
<td>09C045</td>
<td>Shorting plug</td>
</tr>
<tr>
<td>020120</td>
<td>110V AC line adapter</td>
</tr>
<tr>
<td>020121</td>
<td>220V AC line adapter</td>
</tr>
<tr>
<td>020041</td>
<td>Neck strap and meter holder for “hands-free” use. See Figure 8.</td>
</tr>
<tr>
<td>020042</td>
<td>Carrying case with foam insert, without meter or accessories</td>
</tr>
<tr>
<td>020044</td>
<td>Accessory pack, includes two 60 ml bottles and one 150 ml beaker</td>
</tr>
<tr>
<td>020045</td>
<td>Rod, electrode holder and stand for “out-of-case” bench top use</td>
</tr>
</tbody>
</table>

### NOTICE OF COMPLIANCE

**WARNING:** This meter may radiate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer’s instructions, may cause interference to radio communications. It has been tested and found to comply with the limits for a Class A computing device pursuant to Subpart J or Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a commercial environment. Operation of the meter in a residential area may cause interference in which case the user at his own expense will be required to take whatever measures may be required to correct the interference.
SPECIFICATIONS

mV
- 999 to 999

Temperature
- 5.0 to 105.0°C

pH
- 2.00 to 19.99

Isopotential
0.01 to 19.99

Slope
80 to 110%

Relative Accuracy
pH 01° ± 01. ± 0.05% whichever is greater
pH 01° ± 01. ± 0.05% whichever is greater
mV ± 1.0

Temperature
± 1.0

Display
3 1/2 digit LCD

Inputs
Single, ATC

Power Requirements
One 9 Volt alkaline battery or line adaptor for either 110
or 220V AC, 50/60 Hz wall outlet

Input Impedance
> 100,000 megohms

Instrument Drift
< 50 microvolts/°C

Input Bias Current
< ± 1 pico amps at 25°C and < ± 4 pico amps over
full operating range

Environmental Requirements
50 to 45°C and 5 to 80% relative humidity, non-
condensing

Meter Dimensions
14 cm X 14 cm X 4 cm

Meter Weight
0.5 kg

Meter Case
Splash-proof, chemical-resistant

Carrying Case
38.1 cm X 27.9 cm X 11.4 cm

Carrying Case Weight
1.8 kg

* when using two buffers calibrated according to the procedure on page 6