ATTACHMENT 1 – SELECT STANDARD OPERATING PROCEDURES (SOPS) AND METHOD GUIDANCE

This attachment includes SOPs from International Paper, Barr Engineering, and Integral, as well as ASTM guidance and User's Guides for sampling and field lab screening work. The items in this attachment are listed below:

General SOPs

SOP - FIELD DOCUMENTATION

SOP - SAMPLE CUSTODY

SOP - TRANSPORTING SAMPLES TO THE LABORATORY

SOP - COLLECTION OF QUALITY CONTROL SAMPLES

SOP - DECONTAMINATION OF SAMPLING EQUIPMENT

SOPs for Soil and Sediment Sampling

SOP - SOIL SAMPLE COLLECTION

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SOP - FIELD CLASSIFICATION OF SOIL

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SOPs for Surface Water Sampling

SOP - COLLECTION OF SURFACE WATER SAMPLES

SOP - MEASUREMENT OF SURFACE WATER AND GROUNDWATER FIELD PARAMETERS

SOP - GROUNDWATER AND SURFACE WATER QC SAMPLING PROCEDURES

ASTM Guidance

ASTM D2488 – STANDARD PRACTICE FOR DESCRIPTION/ID OF SOILS

ASTM D4538 – STANDARD PRACTICE FOR COLLECTION OF FLOOR DUST SAMPLES

User's Guides for Field Screening

XRF USER'S GUIDE

PCP IMMUNOASSAY USER'S GUIDE

PAH RAPID ASSAY USERS GUIDE

SOP - FIELD DOCUMENTATION

All information relevant to field operations must be properly documented to ensure that activities are recorded and actions can be reconstructed from written records. Field documentation should include only a factual description of site-related activities and observations made. Field personnel should not include superfluous comments or speculation regarding the field activities or observations made. Several types of logbooks may be used for this purpose and should be consistently used by field crews (e.g., field logbooks, sample logbooks, field data logbooks). Logbooks will be labeled on the cover with the project name, dates of field work, and the Purchase Order number (or other assigned number). A separate bound logbook with consecutively numbered pages will be used for each field project. Each logbook for a particular project will be numbered (e.g., *Project Name* Sampling Investigation—Field Logbook *Number* 2).

The information recorded in each logbook should be written in indelible ink. All corrections should consist of a single line-out deletion, followed by the author's initials and the date. Field logbooks will be photocopied after each period in the field, and photocopies will be stored in the project files. After field activities are completed, logbooks will be stored in the permanent project file. No bound logbooks should be discarded, even if they are illegible or contain inaccuracies that require a replacement document. When not in use, all logbooks will be stored in the permanent project file.

FIELD LOGBOOKS

The purpose of the field logbook is to document events that occur and record data measured in the field to the extent that someone not present at the site can reconstruct the activity without relying on the memory of the field crew. Each page in the field logbook will be initialed and dated by all persons making entries on that page. The author will sign and date the last page at the end of each day, and a line will be drawn through the remainder of the page. The logbooks, at a minimum, must contain the following information:

1. A purpose and description of the field task,
2. The start time and date of the field work,
3. The location and description of the work area, including sketches, map references, and photograph log, if appropriate,
4. The names and titles of field personnel and anyone present during the field work, including the times they are present,
5. The name, agency, and telephone number of any field contacts,

6. The meteorological conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change,
7. Details of the field work performed, with a description of any deviations from the work plan, sampling and analysis plan, or standard operating procedures,
8. All field measurements made (unless a specific logbook or sampling form [i.e., borehole log or groundwater sampling form] is available for this purpose), including the time of measurement,
9. Any field results not appearing in the field data logbook, including station identification and location, date, and time of measurement,
10. Cross-references of numbers for duplicate samples,
11. References to other logbooks used to record information (e.g., station log, sample log, health and safety log), and
12. Logbooks should include only a factual description of site-related activities. Field personnel should not include superfluous comments, speculation, or other non-factual observations regarding the field activities.

SAMPLE COLLECTION FIELD FORMS

Appropriate sample collection field forms will be used to record the relevant sample information collected/observed during a sampling event. For instructions regarding proper use of sample identifiers, sampling personnel should consult the lead field sampler.

SAMPLE LABELS

Sample labels (tags) are designed to uniquely identify each sample, and must be affixed to each sample container used. The labels should be filled out at the time the samples are collected and should consist of the following information:

1. Sample number,
2. Site name,
3. Date and time sample is collected,
4. Initials of the samplers,
5. Preservatives used, if any, and

6. Types of analyses (e.g., EPA Method 8260B).

PHOTOGRAPHS

In certain instances, photographs of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Photographs should include a measured scale in the picture, when practical. The following items should be recorded in the field logbook for each photograph taken:

1. The photographer's name, the date, the time of the photograph, and the general direction faced (orientation),
2. A brief description of the subject and the field work portrayed in the picture,
3. The sequential number of the photograph and the roll number on which it is contained, and
4. If digital photographs are collected for internal use or presentation purposes, the file name, date, file location, description, orientation, and photograph should be recorded.

The slides, prints, or disks (as appropriate) and associated negatives will be placed in the project files after the film is developed. Any supporting documentation from the field logbooks will be photocopied and placed in the task files to accompany the slides, prints, or disks.

EQUIPMENT CALIBRATION RECORDS

Equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration, should be recorded in the field logbook. At a minimum, equipment used during the investigation should be calibrated daily in accordance with the manufacturers' recommendations.

SOP - SAMPLE CUSTODY

A stringent, established program of sample chain-of-custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used in conjunction with SOP's which cover documentation and sample packaging and shipping. Chain-of-custody record/sample analysis request forms (see Attachment 2 to the FSP) ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession,
2. The sample is in the person's view after being in possession,
3. The sample is in the person's possession and is being transferred to a designated secure area, and
4. The sample has been locked up to prevent tampering after it was in the person's possession.

PROCEDURE

The chain-of-custody record portion of the form is the most critical because it documents sample possession from the time of collection through the final disposition of the sample. The sample analysis request portion of the form provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

The chain-of-custody record/sample analysis request form will be completed after each field collection activity and before the samples are shipped to the laboratory. Sampling personnel are responsible for the care and custody of the samples until they are shipped. When transferring possession of the samples, the individuals relinquishing and receiving the samples must sign the chain-of-custody record/sample analysis request form(s), indicating the time and date that the transfer occurs. Copies of the forms will be made and kept by the sampler, and the originals will be included with the samples in the transfer container. The following guidelines will be followed to ensure consistent shipping procedures and to maintain the integrity of the samples:

1. Each chain-of-custody record/sample analysis request form must be appropriately signed and dated by the sampling personnel. The person who relinquishes custody of the samples must also sign this form.

2. The chain-of-custody record/sample analysis request form should not be signed until the information has been checked for inaccuracies by the lead sampler. All changes should be made by drawing a single line through the incorrect entry and initialing and dating it. Revised entries should be made in the space below the entries. On the handwritten chain-of-custody record/sample analysis request forms, spaces remaining at the bottom of the page after corrections are made should be marked out with single lines. This procedure will preclude any unauthorized additions.

3. At the bottom of each chain-of-custody record/sample analysis request form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date that the transfer occurred. The time that the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as Federal Express or UPS, the name of the carrier and airbill should be recorded on the chain-of-custody record/sample analysis request form. The time of transfer should be as close to the actual drop-off time as possible. After the chain-of-custody record/sample analysis request forms are signed and copied, they should be sealed inside the transfer container.

5. If errors are found after the shipment has left the custody of sampling personnel, a corrected version of the forms must be prepared and sent to all relevant parties. Minor errors can be rectified by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.

6. Samples that are archived internally should be accompanied by a chain-of-custody record/sample analysis request form. While samples remain in the sampler's custody before being shipped, all containers will be kept in sight of sampling personnel or in a secured area to preclude tampering with the samples.

SOP - TRANSPORTING SAMPLES TO THE LABORATORY

PURPOSE

Specific requirements for sample packaging and shipping must be followed to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein.

EQUIPMENT REQUIRED

Specific equipment or supplies necessary to properly pack and ship environmental samples include the following:

- Ice in sealed bags or Blue Ice®
- Sealable airtight bags
- Plastic garbage bags
- Coolers
- Bubble wrap
- Fiber reinforced packing tape
- Scissors
- Chain-of-custody seals
- Airbills for overnight shipment
- Chain-of-custody record/sample analysis request forms.

PROCEDURE

The following steps should be followed to ensure the proper transfer of samples from the field to the laboratories:

1. Appropriately document all samples using the proper logbooks and chain-of-custody record/sample analysis request forms (example provided in Attachment 2 to the FSP).

2. Make sure all applicable laboratory quality control sample designations have been made on the chain-of-custody record/sample analysis request forms. Samples in containers designated exclusively for archiving for future possible analysis should be clearly identified on the chain-of-custody record/sample analysis request form and should also be labeled as "Do Not Analyze: Hold and archive for possible future analysis" as some laboratories interpret "archive" to mean continue holding the residual sample after analysis.

3. Notify the laboratory contact and the project QA/QC coordinator that samples will be shipped and the estimated arrival time. Send copies of all chain-of-custody record/sample analysis request forms to the QA/QC coordinator or project manager, as appropriate.

4. Samples will be placed in secure onsite storage or remain in the possession of the sampling personnel prior to shipment. Any temporary sample storage areas will be locked and secured to maintain sample integrity and chain-of-custody requirements.

5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.

6. Fill out the chain-of-custody/sample analysis request form, and retain the back copy of the form for the project records prior to sealing the cooler. Store the signed chain-of-custody record/sample analysis request forms in a sealable bag and tape them to the inside of the cooler lid. For a shipment containing multiple coolers, indicate on the outside of this cooler "Chain-of-Custody Inside."

7. Check sample containers against the chain-of-custody record/sample analysis request form to ensure all samples intended for shipment are accounted for.

8. Store each sample container in a sealable bag that allows the sample label (example provided in Attachment 2 of the FSP) to be read. Volatile organic analyte (VOA) vials for a single sample must be encased in bubble wrap before being sealed in bags.

9. Choose the appropriate size cooler (or coolers) and line with bubble wrap.

10. Fill the cooler with the samples, separating glass containers with bubble wrap and allowing room for ice to keep the samples cold. Add enough ice or Blue Ice® to keep the samples refrigerated overnight. Ice should be enclosed in sealable plastic bags to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it will insulate the containers from the ice. After all samples and ice have been added to the

cooler, use bubble wrap to fill any empty space to keep the samples from shifting during transport.

11. If possible, consolidate all VOA samples in a single cooler, and ship them with (a) trip blank(s) in accordance with the quality assurance project plan.

12. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it shut with fiber-reinforced packing tape. If the cooler has a drain at the bottom, it should be taped shut in the same manner.

13. As security against unauthorized handling of the samples, apply one or two chain-of-custody seals across the opening of the cooler lid (example provided in Attachment 2 of the FSP). Be sure the seals are properly affixed to the cooler so they are not removed during shipment.

14. Label the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care."

15. If an overnight courier is used, fill out the airbill as required and fasten it to the top of the cooler. The identification number sticker should be taped to the lid, because tracking problems can occur if a sticker is removed during shipment.

SOP - COLLECTION OF QUALITY CONTROL SAMPLES

PURPOSE

To describe the procedures used in the collection of quality control samples (masked duplicate samples, trip blanks, field blanks, and equipment blanks).

APPLICABILITY

This procedure applies to sample handling techniques used by both the technician(s) and the laboratory in regards to quality control.

DEFINITIONS

- **Masked Duplicate Sample**. This is the collection of a sample at the same time the original sample is being collected. Both samples are collected, preserved, and analyzed exactly the same. This is done to check laboratory and sampling precision.

- **Trip Blank**. This is a water blank free of any contaminants, prepared prior to sampling events by the laboratory providing the sampling containers. The purpose of the trip blank is to determine if contamination has occurred from:

    1. Improper sampling container cleaning,

    2. Contaminated blank source water,

    3. Exposure to contaminant during storage and transportation, or

    4. Other environmental conditions during sampling.

- **Field Blank.** This is a sample container prepared onsite consisting of (analyte-free) water (received from the laboratory). These blanks are used to evaluate:

    1. The effects of onsite environmental contaminants,

    2. The purity of reagents used as preservative or additives, and

    3. General sample container filling/collecting techniques.

- **Equipment Blank.** This is a sample collected from the final (analyte-free) rinse water. The water is rinsed on or through sampling equipment. The rinse water is collected for analysis. These blanks are used to determine:

    1. The effectiveness of field cleaning procedures.

    2. Any sources of contamination in a trip blank.

SOP - DECONTAMINATION OF SAMPLING EQUIPMENT

To prevent potential cross-contamination of samples, all reusable soil and water sampling equipment and pumps will be decontaminated. The lead sampler will set up the area used to decontaminate soil and water sampling equipment consisting of three stations, as described below. Where practicable, this area will be located upwind from the specific sampling area and upwind from process areas that could skew sample results. The personnel performing the decontamination procedures will wear protective clothing as specified in the site-specific Site Health and Safety Plan.

This SOP describes procedures for decontaminating sampling equipment contaminated by either inorganic or organic materials. Sampling equipment used for both can combine these procedures, following the order of a tap water rinse, detergent wash, organic solvent, acid rinse, and final water rinse. At stations where both water and soil (or other solid media) will be sampled, separate decontamination areas should be used for each medium where appropriate.

When using a drilling contractor, subsurface soil samplers (i.e., split spoons, Dames & Moore U-type samplers, core barrels, and SPTs) can be decontaminated by using a heated pressure washer (steam cleaner). The decontaminated sampler(s) (if not to be used immediately) will be stored in a plastic bag or wrapped in aluminum foil until ready for use. Storage of sampling equipment must be consistent with the project data quality objectives, and analytical parameters must be considered (e.g., storage in plastic bags is not recommended when analyzing samples for phthalates).

EQUIPMENT REQUIRED TO DECONTAMINATE INORGANIC-CONTAMINATED SAMPLING EQUIPMENT

Equipment:

- 3-gal plastic tubs
- 5-gal plastic containers, tap water
- 5-gal carboy laboratory-grade distilled/deionized (DS/DI) water (organic/analyte-free)
- Properly labeled spray bottles for decontamination solvents
- Alconox® (or equivalent)
- normal nitric acid

- Hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Personal protective equipment as specified in the Health and Safety Plan
- 55-gal drum(s)
- Drum labels.

PROCEDURES USED TO DECONTAMINATE INORGANIC-CONTAMINATED SAMPLING EQUIPMENT

The specific procedures for decontaminating inorganic-contaminated soil sampling equipment include the following:

1. An initial tap water rinse of sampling equipment to remove gross soil or sediment. The rinse water can be containerized and transported to the groundwater treatment facility for treatment.
2. At Station No. 1, first wash the contaminated equipment in a tub containing tap water mixed with a detergent such as Alconox®. Only a small volume (0.5 teaspoon) of Alconox® is necessary, and all Alconox® crystals should be completely dissolved.
3. Move the equipment to the wash tubs at Station No. 2. First, rinse the equipment with potable water, followed by rinsing equipment with 0.1 Normal nitric acid (HNO_3) or similar acid, then rinse with DS/DI water.
4. At Station No. 3, place the clean equipment on plastic sheeting until reuse.

After decontaminating all the sampling equipment, the disposable gloves, and used plastic from Station No. 3 will be placed in garbage bags and disposed of. The wash and rinse water from Station Nos. 1 and 2 will be containerized for proper disposal. At the end of each day, all sampling equipment will be stored in large plastic bags.

EQUIPMENT REQUIRED TO DECONTAMINATE ORGANIC-CONTAMINATED SAMPLING EQUIPMENT

- 55-gallon drum(s) to collect tap water from the initial rinse (for transport to the site groundwater treatment facility)
- 3-gal plastic tubs
- 5-gal plastic containers, tap water

- 5-gal carboy laboratory analyte-free DS/DI water
- Properly labeled spray bottles for decontamination solvents
- Aluminum foil
- Alconox® (or equivalent)
- Hard-bristle brushes
- Pesticide-grade acetone, hexane, and methanol
- Plastic sheeting and garbage bags
- Personal protective equipment as specified in the Health and Safety Plan.

PROCEDURES USED TO DECONTAMINATE ORGANIC-CONTAMINATED SAMPLING EQUIPMENT

The specific procedures for decontaminating the organic-contaminated soil and groundwater sampling equipment include the following:

1. An initial tap water rinse of sampling equipment to remove gross soil or sediment. The rinse water can be containerized and transported to the groundwater treatment facility for treatment.
2. At Station No. 1, first wash the contaminated equipment in a tub containing tap water mixed with a detergent such as Alconox®. Only a small volume (0.5 teaspoon) of Alconox® is necessary, and all Alconox® crystals should be completely dissolved.
3. At Station No. 1, Tub No. 2, double rinse the equipment with site or DS/DI water.
4. At Station No. 2, rinse the equipment with a pesticide-grade organic solvent (e.g., hexane, if appropriate to remove oily contamination) followed by a rinse with acetone or methanol (drying agent). These solvents should be captured in a separate container and allowed to evaporate. Station No. 2 should be placed in a well-ventilated area.
5. At Station No. 3, double rinse the equipment with DS/DI water.
6. At Station No. 4, lay the equipment on clean aluminum foil to air dry.
7. Wrap the equipment in clean aluminum foil until reuse.

The disposable gloves and used foil from Station No. 3 will be placed in garbage bags and disposed of. The initial tap water rinse can be treated through the groundwater treatment system. The Alconox®/water would not contain any gross contamination and would be discharged to the ground with the other rinses.)

The following equipment is used to decontaminate sampling pumps:

- Submersible pumps
- Alconox® (or equivalent)
- Tap water
- Hard-bristle brushes
- Plastic sheeting and garbage bags
- 30-gal plastic trash can or plastic overpack drum
- 55-gal drum(s)
- Hot-water pressure washer (optional).

PROCEDURES USED TO DECONTAMINATE SAMPLING PUMPS

The specific procedures used for decontaminating sampling pumps include the following:

1. It is advisable to begin sampling with the well or surface water stations containing the lowest anticipated analyte concentration. Successive samples should be obtained from wells or stations anticipated to have increasing analyte concentrations. Use of dedicated pump equipment is preferable when feasible.

2. When pumps (e.g., submersible, bladder) are submerged below the water surface to collect water samples, they should be cleaned and flushed between uses. This cleaning process consists of an external detergent wash and rinse, or hot-water pressure washing of pump casing, tubing, and cables, followed by a flush of potable water through the pump. This flushing can be accomplished by pumping approximately 10 gal of an Alconox® solution through the pump and then pumping approximately 10 gal of tap water through the pump. This should be followed by rinsing the external parts of the pump intake hose and cable with a tap-water rinse, and finally with a DI/DS-water rinse. The procedure should be repeated after sampling from each monitoring well location. The pump and hose should

always be placed on clean polyethylene sheeting or in a plastic bag to avoid contact with the ground surface.

3. Surface pumps (e.g., peristaltic) used for well evacuation and surface water sampling need not be cleaned between well locations unless trace metal clean sampling techniques are required. However, a new length of polyethylene and Pharmed® (or equivalent) tubing must be used for each well and discarded after use.

SOP - SOIL SAMPLE COLLECTION

SURFACE SOIL SAMPLING

The following procedures describe collection of surface soil samples. Soil sampling should be sequenced to start with areas of low contaminant-of-concern (COC) concentrations, proceeding to areas of higher COC concentrations. The procedures listed below may be modified in the field by agreement between the lead site sampler and field personnel, based on field and site conditions. In the event that procedures are modifies, appropriate annotations should be been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. Record all pertinent information on the soil sampling Field Data Form (included on attachment 2 of the FSP).

EQUIPMENT

- Appropriate sampling device(s) as determined by site sampling lead (stainless-steel scoop, trowel, plastic disposable sampling tool, split-barrel, piston sampler, backhoe, shovel, etc.)
- Laboratory-supplied sample containers
- Field logbook
- Surface soil field collection form.

PROCEDURES

1. Locate the sampling point as directed in the work plan or SAP. Containers will be labeled with sample tags prior to filling. If analytical testing will be performed for volatile organic compounds (VOCs), the VOC sample will be collected first (with a minimum of disturbance) by placing the sample into the container, with a minimum amount of headspace, and sealed tightly.
2. Expose the soil surface by clearing an approximately 1-ft^2 area at the sampling site of any rocks or organic material greater than approximately 3 inches in size. Note any material removed from the sampling site in the field notebook.

3. Using a decontaminated stainless-steel, disposable plastic sampling tool, or other appropriate sampling tool as specified by the field sampling lead, excavate soil to the depth specified in the work plan or SAP.

4. If required for analysis, first collect VOC samples (prior to any homogenization), placing the samples in the appropriate-size containers.

5. Place additional sample material in a decontaminated plastic or stainless-steel mixing bowl.

6. Thoroughly mix and homogenize the sample using disposable equipment or a decontaminated stainless-steel spoon.

7. Rocks that are greater than 0.5 in. in diameter may be discarded from the homogenized soil after they are positively identified and their percentage contribution to the homogenized soil volume has been determined and noted in the field notebook.

8. If composite samples are being collected, refer to the Soil Sample Compositing SOP; else, remove samples of the homogenized soil from the mixing dish and place in the appropriate size sample container. The sample container should be filled with soil to just below the container lip, and the container should be sealed tightly.

9. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets.

10. Mark the sampling site with a wire flag, wooden stake, metal rebar, or flagging, as appropriate.

11. Decontaminate all sampling equipment (per Decon SOP).

12. Package and ship samples according to procedures in the QAPP and the Sample Packaging and Shipping SOP.

SOP - SOIL SAMPLE COMPOSITING

Both composite and discrete samples can be used for environmental investigations. A composite sample is a single sample created by combining two or more discrete subsamples. Composite samples are valuable for characterizing large areas/volumes of soil.

The purpose of this SOP is to describe the procedures of compositing several discrete samples into one representative sample for analysis. The representative number of subsamples) is defined in the FSP. Additionally, detailed guidance for subsample collection is given from specific programs, (i.e., Minnesota Department of Agriculture Agricultrual Chemical Incidents, MCPA's Leaking Petroeum Storage Tanks). In general, sampling the total investigation area and final numbers of subsamples should be appropriate to meet the data quality objectives for the project.

Discrete soil samples identified for compositing can be collected in several ways. These include a split-barrel sampler, core barrel sampler or by hand excavation. Additional information on soil sample collection can be found in the SOP for soil sample collection. A minimum volume of soil obtained during discrete sampling will be dependent on the final analytical requirements for the composite sample. Analytical samples should not be collected from polyethylene bags sometimes used for field screening purposes. Volatile organic samples should not be composited, due to aeration of the sample during mixing.

The equipment required to composite discrete samples includes: stainless steel spoons or scoops, a large mixing bowl and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

All soil compositing equipment will be carefully cleaned between uses as specified in the FSP. To prevent sample cross-contamination, the sampler will discard the outer pair of sample gloves and put on a new pair between each compositing event.

COMPOSITING DISCRETE SAMPLES

1. After individual samples have been obtained, compositing begins by documenting the discrete sample locations to be included in final composited sample. Appropriate laboratory containers should be labeled with this final sample identifier and the date of collection.

2. Retrieve the samples selected for compositing per the SOP for Soil Sampling.

3. Empty the contents of the sampler into the stainless steel mixing bowl, removing any large debris or rocks. Mix thoroughly.

5. Fill appropriate laboratory sample containers.

6. Complete chain-of-custody documentation.

7. Immediately after samples are composited, they should be placed in a cooler containing ice or ice packs and cooled at 4°C for shipment to the laboratory.

SOP - FIELD CLASSIFICATION OF SOIL

This SOP presents the field classification of soils, adopted from ASTM D-2488-84, which uses the Universal Soil Classification (USC) system for naming soils. Field personnel are encouraged to study these procedures. Soil descriptions should be precise and comprehensive without being verbose. The overall impression of the soil should not be distorted by excessive emphasis on minor constituents. These descriptions will be used to interpret aquifer properties and other potential contaminant transport properties, rather than the exact mineralogy or tectonic environment.

Soil descriptions should be provided in the Soil Description column of the Field Log Form for each sample collected. If there is no difference between consecutive soil samples, subsequent descriptions can be noted as "same as above" or minor changes such as "increasing sand" or "becomes dark brown" can be added.

The format of soil descriptions for each sample or identified stratigraphic layer/soil horizon should be as follows:

- Group symbol—The group symbol should be placed in the Unified Symbol column,
- USC group name—The USC name should be identical to the ASTM D-2488-84 Group Name with the appropriate modifiers,
- Minor components,
- Color,
- Moisture, and
- Additional descriptions.

Examples of soil descriptions are provided in the following table. The minimum elements of the soil descriptions are discussed below.

EXAMPLE OF SOIL SAMPLE DESCRIPTIONS

SM	Silty fine to medium SAND, trace fine gravel and occasional roots, very dark gray, moist to wet.
SW-SM	Fine to coarse SAND with silt, some fine gravel, mottled dark gray and tan, moist. Sand consists of 20 percent biotite flakes, no bedding observed.
ML	Sandy SILT, fine sand, dark gray, moist. Fractures predominantly vertical, at 1–3-in. spacing.
GW	Fine to coarse GRAVEL with 10 percent medium to coarse sand, trace woody debris, gray, moist to wet. Reddish brown staining noted within 2 ft of water table. Gravel is rounded and flat.

DEFINITIONS OF SOIL TYPES

The following table presents the USC system. The USC system is an engineering-properties system that uses grain size to classify soils. The first major distinction is between fine-grained soils (more than 50 percent passing the No. 200 sieve [75 μm/0.029 in.]) and coarse-grained soils (more than 50 percent retained by the No. 200 sieve).

SOIL CLASSIFICATION SYSTEM

Major Divisions			Group Symbol	Group Name
Coarse-Grained Soils More than 50 percent retained on No. 200 sieve	**Gravel** More than 50 percent of coarse fraction retained on No. 4 sieve	Clean gravel	GW	Well-graded gravel, fine to coarse gravel
			GP	Poorly graded gravel
		Gravel with fines	GM	Silty gravel
			GC	Clayey gravel
	Sand More than 50 percent of coarse fraction passes No. 4 sieve	Clean sand	SW	Well-graded sand, fine to coarse sand
			SP	Poorly graded sand
		Sand with fines	SM	Silty sand
			SC	Clayey sand
Fine-Grained Soils More than 50 percent passes No. 200 sieve	**Silt and Clay** Liquid limit <50	Inorganic	ML	Silt
			CL	Clay
		Organic	OL	Organic silt, organic clay
	Silt and Clay Liquid limit ≥50	Inorganic	MH	Silt to high plasticity, elastic silt
			CH	Clay of high plasticity, fat clay
		Organic	OH	Organic clay, organic silt
Highly organic soils			PT	Peat

Note: Field classification is based on visual examination of soil in general accordance with ASTM D-2488-84.

Soil classification using laboratory tests is based on ASTM D-2487-83.

Descriptions of soil density or consistency are based on interpretation of blow count data, visual appearance of soils, and/or test data.

Liquid limit - water content of soil-water where consistency changed from plastic to liquid.

Fine-grained soils are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-84. If these tests are used, the results should be included in the soil description. At least one complete round of field tests should be performed for a site if these fine-grained materials are encountered, preferably at the beginning of the field investigation. The modifiers "fat" and "lean" are used by ASTM to describe soils of high and low plasticity. The soil group symbols (e.g., CL, MH) already indicate plasticity characteristics, and these modifiers are not necessary in the description. Soils with high plasticity can be emphasized by describing them as "silty CLAY with high plasticity." Plasticity is an important descriptor because it is often used to interpret whether an ML soil is acting as

either a leaky or competent aquitard. For example, an ML soil can be dilatent/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.

Coarse-grained soils are classified as either predominantly gravel or sand, with the No. 4 sieve (4.75 mm/0.19 in.) being the division. Modifiers are used to describe the relative amounts of fine-grained soil in a sample, as noted in the table below:

COARSE-GRAINED SOIL DESCRIPTIONS

Description	Percent Fines	Group Symbol
Gravel (sand)	<5 percent	GW, GP (SW, SP)
Gravel (sand) with silt (clay)	5–15 percent	Hyphenated names
Silt (clayey) with gravel (sand)	>15 percent	GM, GC (SM, SC)

The gradation of a coarse-grained soil is included in the specific soil name (i.e., fine to medium SAND with silt). Estimating the percent of size ranges following the group name is encouraged for mixtures of silt sand and gravel. Use of the modifiers "poorly graded" or "well graded" is not necessary because they are indicated by the group symbol.

A borderline symbol is shown with a slash (GM/SM). This symbol should be used when the soil cannot be distinctly placed in either soil group. A borderline symbol should also be used when describing interbedded soils of two or more soil group names when the thicknesses of the beds are approximately equal, such as "interbedded lenses and layers of fine sand and silt." The use of a borderline symbol should not be used indiscriminately. Every effort should be made to place the soil into a single group.

MINOR COMPONENTS

Minor components, such as cobbles, roots, and construction debris, should be preceded by the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). The word "occasional" can be applied to random particles of a larger size than the general soil matrix (i.e., occasional cobbles, occasional brick fragments). The term "with" indicates definite characteristics regarding the percentage of secondary particle size in the soil name. It will not be used to describe minor components. If a non-soil component exceeds 50 percent of an interval, it should be stated in place of the group name.

COLOR

The basic color of a soil, such as brown, gray, or red, must be given. The color term can be modified by adjectives such as light, dark, or mottled. Especially note staining or mottling. This information may be useful to establish water table fluctuations or contamination. The Munsell soil color chart designation is the required color standard. All color designations must

be accompanied by a description of the moisture content of the soil when the color designation was made. It is generally preferable to determine color on moist samples; water may be added to the soil to achieve this moisture content.

MOISTURE CONTENT

The degree of moisture present in the soil should be defined as dry, moist, or wet. Moisture content can be estimated from the criteria listed in Table 3 of ASTM D-2488-84.

ADDITIONAL DESCRIPTIONS

Features such as discontinuities, inclusions, joints, fissures, slickensides, bedding, laminations, root holes, soil animals, and major mineralogical components should be noted if they are observed. Anything unusual should be noted. Additional soil descriptions may be made at the discretion of the project manager or as the field conditions warrant. The Field Borehole Log Form lists some optional descriptions, as does Table 13 of the ASTM standard. The reader is referred to the ASTM standard for procedures of these descriptions.

CONTACTS BETWEEN SOIL TYPES

The contact between two soil types must clearly be marked on the soil borehole log because it is very difficult to interpret borehole logs where soil sample descriptions change over a 5- or 10-ft sample interval if there is no indication of where this change occurred. If the contact is obvious and sharp, draw it in with a straight line. If it is gradational, a slanted line over the interval is appropriate. In the case where it is unclear, a dashed line over the most likely interval is used.

SOP - SEDIMENT SAMPLING

PURPOSE

To describe the procedures and techniques for collecting sediment samples for physical and chemical analysis.

APPLICABILITY

This procedure applies to the collection of sediment sample by the environmental technician.

REFERENCES

Handbook of Techniques for Aquatic Sediment Sampling, Mudroch and MacKnight, CRC Press, 1991.

Coring Devices for Lake Sediments, Wright, Cushing, and Livingstone, Contribution No. 9, Limnological Research Center, University of Minnesota, year unknown.

Quality Assurance Project Plan, Part 3, *Phase II Response Action Plan—USX Duluth Works Site*, USX Corporation, June 1993.

DISCUSSION

Sediment samples are collected to describe the physical nature of the sediments and to analyze the chemical properties of the sediments.

RESPONSIBILITIES

The environmental technical lead is responsible for the collection of sediment samples.

PROCEDURE

A. General

1. Locate the sample site(s)
   - Generally requires the services of a Registered Land Surveyor.
   - Locations can be marked on water using a small buoy, or by using ice stakes.
   - Measure water depth using a depth sounder.

B. Piston-Tube Coring Method

1. Prepare coring device
   - General cleanliness procedures will be followed at all times as outlined in the Quality Assurance Project Plan.
   - Choose appropriate size and material tube.
   - Remove foil from clean tubes.
   - Install rubber stopper with attached cable into tube.
   - Install tube in coring rod.
   - Install appropriate length of extensions for water+sediment depth.

2. Perform coring
   - General cleanliness procedures will be followed at all times as outlined in the Quality Assurance Project Plan.
   - Label the core tube.
   - Lower coring device slowly into the water with second person feeding cable attached to the rubber stopper.
   - When core is at top of sediment, tie cable to fixed point.
   - Push core to desired depth.
   - Remove coring device slowly; cap bottom end of tube as soon as it is at the surface; cut cable at top of tube.
   - Remove core from coring rod and store in upright position.
   - Store on ice if necessary (depends on analysis).
   - Repeat as necessary to obtain appropriate sediment volume.

3. Document cores and bottle samples

For Physical Analysis Only (using clear tube):

- Measure and document stratigraphy, soil type, evidence of organics, etc. with core contained in tube.
- Extrude core as explained below and verify physical description after slicing core open.

For Chemical and Physical Analysis (using clear or opaque tube):

- General cleanliness procedures will be followed at all times as outlined in the Quality Assurance Project Plan.
- Prepare clean surface to hold core; line with foil.
- Remove stopper from core (pull through top).
- Remove bottom cap.
- Insert push rod into bottom of tube; extrude core into holder (two people required).
- Measure and photograph core.

- Slice core down the middle and open halves.
- Measure and document stratigraphy, soil type, evidence of organics, density, etc.
- Label sample bottles.
- Collect appropriate samples according to the soil sample collection standard operating procedure, place in sample bottles and store bottles in sample cooler on ice.
- Clean equipment for next sample.
- Repeat until finished.

SOP - WET SIEVING SEDIMENTS IN THE FIELD

Following sediment sample collection and homogenization, wet sieving will be conducted in the field to obtain an estimate of grain size composition. The method used for particle size analysis through wet sieving comes from the Puget Sound Estuary Program (PESP 1986) as recommended by EPA (2001). Wet sieving separates the sample into size fractions greater than 62.5 um (i.e., sand and gravel) and less than 62.5 um (i.e., silt and clay).

MATERIALS

- 1-L graduated cylinder
- Analytical balance 0.1 mg accuracy
- 62.5-um (4 phi) sieve
- Funnel
- Distilled water
- 50-mL beakers
- Stainless steel spoon
- Water squirt bottle
- Gloves
- Weigh pans

METHODS

- Remove a representative aliquot for wet sieving. The aliquot can range from 20 g for muddy sediments to 100 g for sandy sediments. The critical factor for sample size determination is the weight of fine-grained material that will be used for the pipet analysis. Ideally the total dry weight of fine-grained material in the 1-L graduated cylinder should equal approximately 15 g. However, total weights between 5 and 25 g are considered acceptable. Total weights outside this range are not considered acceptable and it is recommended that aliquot size be modified to bring the amount of fine-grained material into the acceptable range.
- Weigh the wet sample to the nearest 0.01 g.
- Place the 62.5-um (4 phi) sieve in a funnel, with a 1-L graduated cylinder underneath. Moisten the sieve using a light spray of distilled water.

- Place the sample in a beaker, add 20-30 mL of distilled water, and stir to suspend fine-grained material.
- Pour the sample into the sieve and thoroughly rinse the beaker and stirrer with distilled water.
- Wash the sediment on the sieve with distilled water using a water pique or squirt bottle having low water pressure. Aggregates can be gently broken using a rubber rod.
- Continue wet sieving until only clear water passes through the sieve. Try to ensure that the rinsate does not exceed approximately 950 mL. This can generally be accomplished by sieving a sample quantity that is not too large and by efficient use of the rinse water. Both of these techniques may require experimentation before routine wet sieving is started.
- Weigh fractions of both the material that was retained by the sieve and the material that passed through the sieve. Note that the fine material in the graduated cylinder that passed through the sieve should be allowed to settle prior to decanting of the overlying water.

REFERENCES

PESP, 1986. Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound. Particle Size. Puget Sound Estuary Program. March.

EPA, 2001. Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analysis: Technical Manual. EPA-823-B-01-002. U.S. Environmental Protection Agency Office of Water. October.

SOP - COLLECTION OF SURFACE WATER SAMPLES

Information regarding surface water sampling is presented below. Samples can be collected from storm drains, rivers, lakes, or ponds. Record all pertinent information on the surface water sampling Field Data Form (included in Attachment 2 of the FSP).

EQUIPMENT REQUIRED

- Water sample containers
- Vacuum hand pump with disposable filtration units (if applicable).

PROCEDURE

1. Submerse sample bottle in water, mouth pointing upstream and below the water surface. Take care not to collect any streambed solids disturbed by wading.
2. If volatile organic compound (VOC) analysis is required, collect samples for VOCs using a precleaned unpreserved glass sample bottle. Transfer the contents of the sample bottle to 40-mL volatile organic analyte (VOA) vials making absolutely certain that there are no bubbles adhering to the sides or top of the VOA container and that there is no headspace in the container. Be sure to check that the condition of samples is acceptable in the VOA containers before leaving each sampling site. If any air bubbles are present, the VOA sample must be retaken using a fresh sample container.
3. If field filtration for dissolved metals is required, collect samples using a hand pump apparatus and transfer to the appropriate sample bottles.
4. Perform field water quality measurements according to the sampling and analysis plan (SAP).

SOP - MEASUREMENT OF SURFACE WATER AND GROUNDWATER FIELD PARAMETERS

Information and general instructions for field measurement of water quality parameters (pH, Eh, specific conductance, dissolved oxygen, and temperature) are presented below. Due to the variety and complexity of water quality meters available, calibration and measurement procedures should be conducted in accordance with manufacturer's recommendations for specific meters used. The following information describes general procedures for the measurement of water quality parameters. Where possible, sampling should be conducted first in areas least affected by constituents of interest, followed by increasingly affected areas.

GENERAL EQUIPMENT AND MATERIALS

- 250- to 500-mL beakers or flow-through cell for groundwater
- Water quality parameter multimeter or meters specific to parameters of interest (i.e., pH, specific conductance, dissolved oxygen, oxidation-reduction potential, and temperature)
- Calibration solutions and deionized distilled water.

GENERAL PROCEDURES

Calibrate meter(s) in the field at the beginning of each day of field or laboratory work when water quality parameters will be measured. Check meters with calibration standards after every 4 hours of continuous use. If drift is evident, recalibrate.

1. Calibrate meter(s) in accordance with manufacturer's instructions using fresh (unused) calibration buffers and standards.
2. Check slope reading with specifications (in operating manual) to verify slope is within the manufacturer's specified range. Open or slide back electrode vent cap(s) to expose probe vent(s).
3. Thoroughly rinse a 500-mL beaker or 8-ounce jar with sample water. Discard sample water.
4. Rinse electrodes with sample water to acclimate them.
5. Fill beaker with fresh sample water.
6. Immerse electrodes in sample while swirling the sample, if needed, to provide thorough mixing. Turn on meter(s). If a flow-through cell is used,

install probes and connect sample water to bottom port of flow-through cell, directing sample water up through the cell, exiting through the top port. Direct effluent tubing into an appropriate container for storage and handling.

7. When the readings have stabilized, record the measurements displayed on the meter. It is important to determine that the correct units and unit scale are displayed on the meter and recorded for each parameter measured. Record and correct any problems encountered during measurement.

8. If available, field measurement results should be compared to previous measurements for quality control.

SOP - GROUNDWATER AND SURFACE WATER QC SAMPLING PROCEDURES

DISCUSSION

Each time a sampling event occurs, some form of quality control measures must be taken.

RESPONSIBILITIES

The sampling technician(s) are responsible for the accurate collection of quality control samples. The laboratory is responsible for the accurate set up and analysis of quality control samples.

PROCEDURE

Quality Control Samples

A. Masked duplicate sample:

1. Collect samples by rotating sampling containers from original sample to the mask (using the same exact methods for both).

2. Preserve, store, and transport the masked duplicate sample in the same manner as the original sample.

3. Submit the masked duplicate sample to the laboratory for the same analysis as the original sample.

Note: Ten percent of all samples are collected in duplicate (mask).

B. Trip blank:

1. Trip blanks are sealed prior to sampling (prepared by the laboratory doing the analysis).

2. Transport trip blanks to the site in the sample storage cooler.

3. Trip blanks are not to be opened in the field.

4. Transport trip blanks back to the laboratory in the sample storage cooler.

5. The trip blanks should be listed on the chain-of-custody along with the other samples and the analysis required. (Generally, VOCs are the only requirement for trip blanks).

Note: Labeling of all sample blank containers follow the SOP for the collection of groundwater samples.

C. Field blank:

1. Get the appropriate sampling containers and desired amount (analyte-free) water from the laboratory. (Generally, field blanks are taken for each parameter.)

2. Prepare field blanks onsite by filling sample containers with the (analyte-free) water.

3. Seal the field blank sample containers and store with other samples collected (should be handled exactly the same).

Note: One field blank should be prepared per day or at a frequency of 10 percent of the samples per sampling event, whichever is greater.

4. Transport all of the samples to the laboratory for analysis. The analysis on both field blanks and samples should be exactly the same.

D. Equipment Blank:

Bailer blank:

1. Receive (analyte-free) water from the laboratory (enough to fill a bailer).

2. Pour (analyte-free) water into a clean bailer.

3. Pour this water into the appropriate sampling containers.

4. Store and transport the equipment blank with the appropriate samples for laboratory analysis.

Filtered equipment blank:

1. Receive appropriate (analyte-free) water from the laboratory.

2. Pour (analyte-free) water into the groundwater sampling filter.

3. Begin filtering (as described in the standard operating procedure for filtering groundwater samples).

4. After filtering is completed, pour water into the appropriate sampling container.

5. Store and transport the equipment blank with the appropriate samples for laboratory analysis.

Note: The filtered equipment blank is usually conducted for filtered metals samples.

DOCUMENTATION

The quality control samples are documented on the chain-of-custody record and the field log data sheet. The technician(s) are required to document any such quality control samples.

ASTM D2488 – STANDARD PRACTICE FOR DESCRIPTION/IDENTIFICATION OF SOILS

Designation: D 2488 – 84[ε1]

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[1] NOTE—Section 18 was added editorially in January 1989.

1. Scope

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils.

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (See Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 *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.* For specific precautionary statements see Section 8.

1.5 The values stated in inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

3. Definitions

3.1 Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US. standard sieve, the following definitions are suggested:

Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and

Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

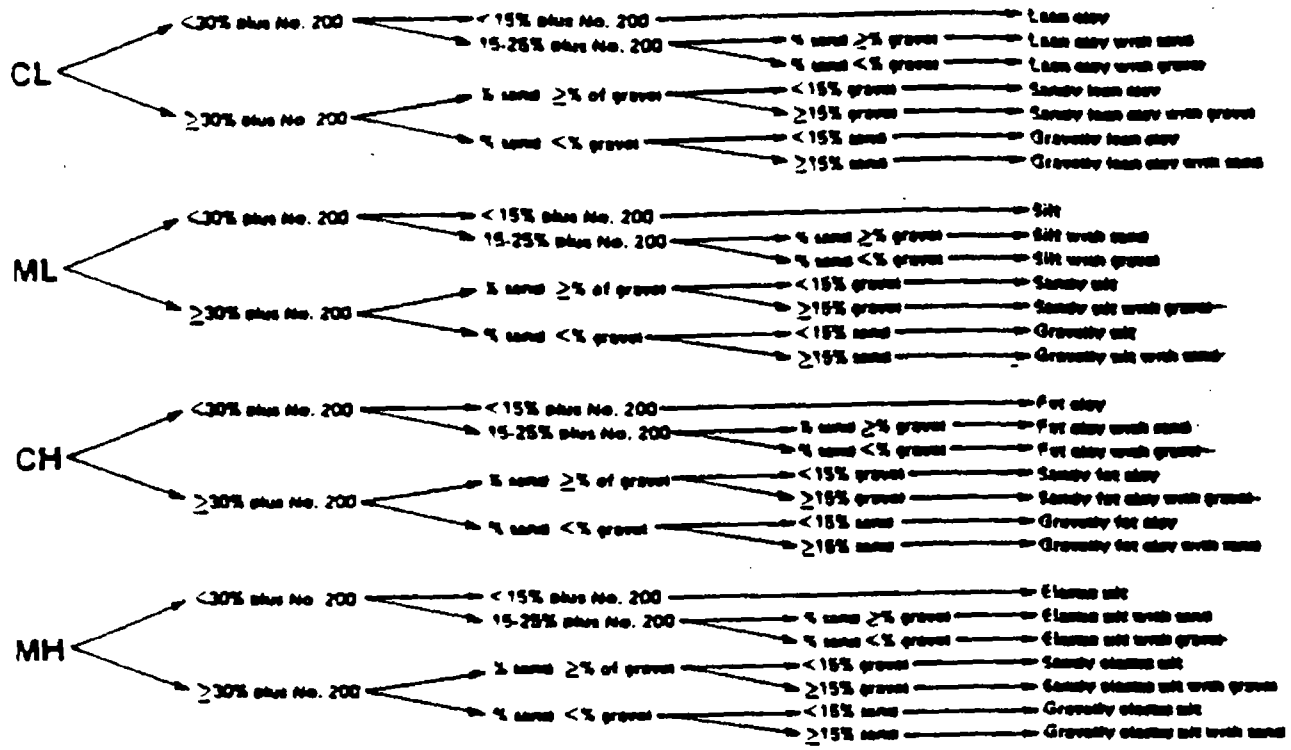
3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved Oct. 3, 1984. Published December 1984. Originally published as D 2488 – 66 T. Last previous edition D 2488 – 69 (1975).

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



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a *Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)*

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-μm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-μm) sieve.

fine—passes a No. 40 (425-μm) sieve and is retained on a No. 200 (75-μm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-μm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

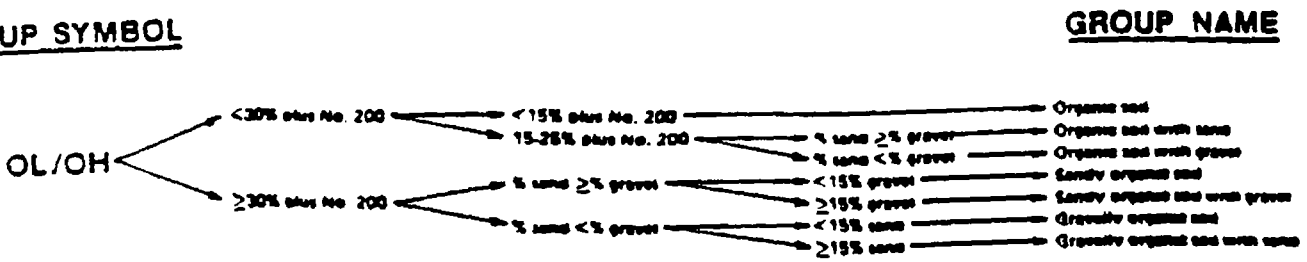
4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Figs. 1a and 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

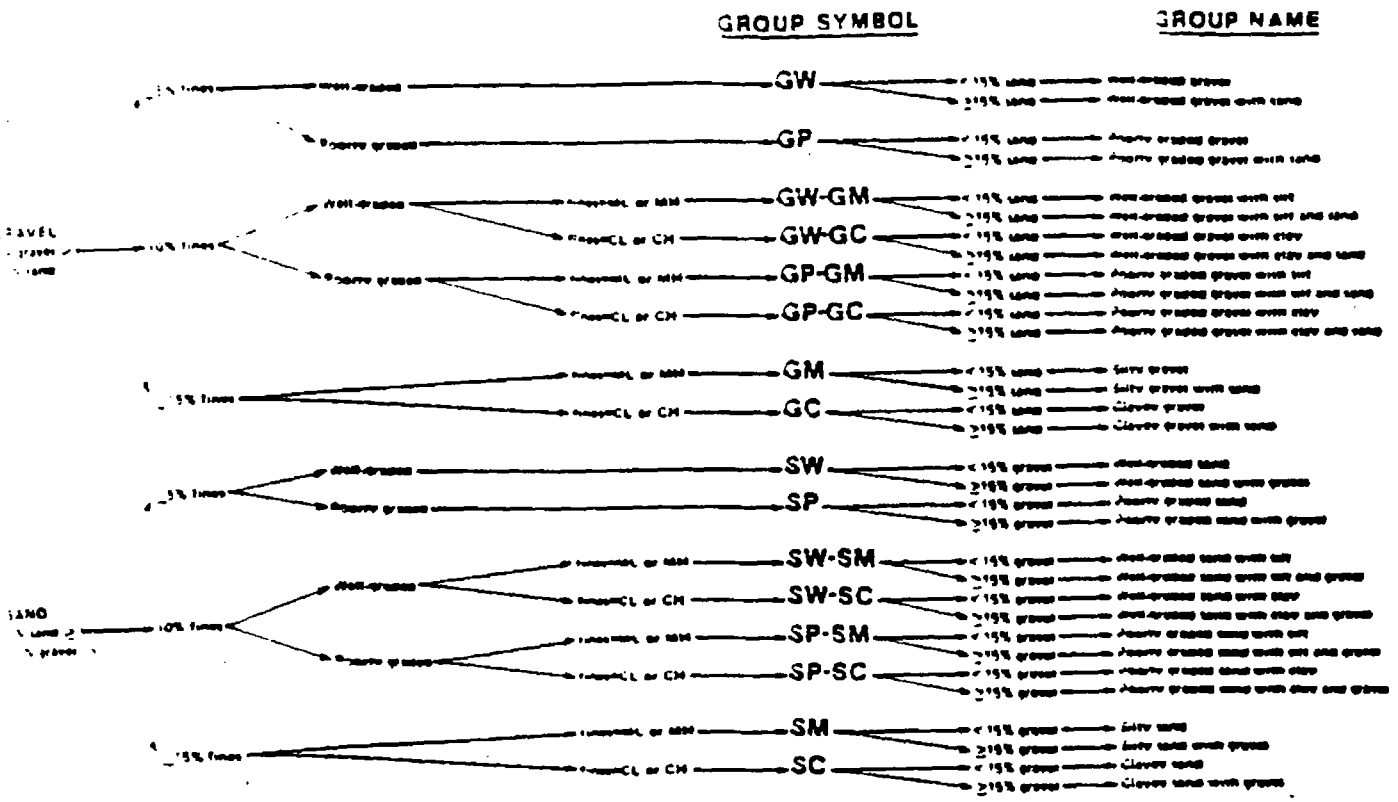
NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1b *Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)*



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

...bols are required when the soil has between 5 and 12 % fines or ...hen the liquid limit and plasticity index values plot in the CL-ML area ...f the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL-ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix A3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5 This practice has particular value in grouping similar ... samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together: one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

6. Apparatus

6.1 *Required Apparatus:*

6.1.1 *Pocket Knife or Small Spatula.*

6.2 *Useful Auxiliary Apparatus:*

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part

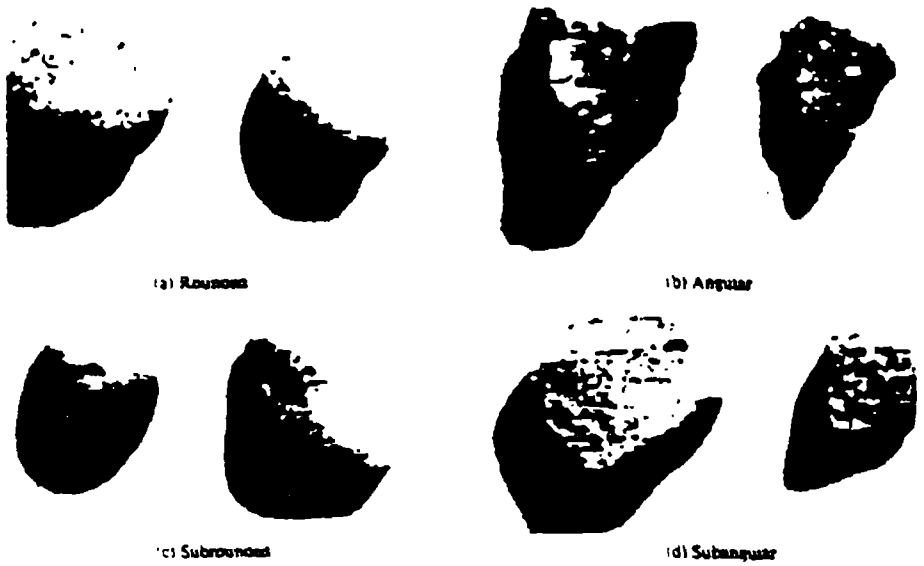


FIG. 3 Typical Angularity of Bulky Grains

concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 Caution—Do not add water to acid.

9. Sampling

9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 5—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 6—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	9.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 7—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively

Flat	Particles with width/thickness ≥ 3
Elongated	Particles with length/width ≥ 3
Flat and elongated	Particles meet criteria for both flat and elongated

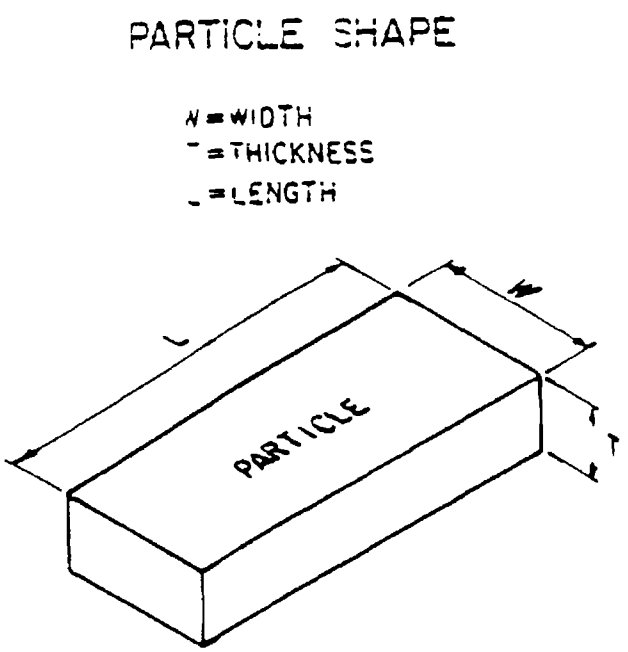


FIG. 4 Criteria for Particle Shape

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the criteria in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

TABLE 4 Criteria for Describing the Reaction with HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼ in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.7. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the particles are hit by a hammer. For example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying materials or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying materials or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

0.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

0.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 8—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 9—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength:*

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 10—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy:*

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally,

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness:*

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils:*

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 11—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as *MH* in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils:*

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 12—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

15. Procedure for Identifying Coarse-Grained Soils (Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words "with clay" or "with silt" to indicate the plasticity characteristics of the fines. For example: "well-graded gravel with clay, GW-GC" or "poorly graded sand with silt, SP-SM" (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "poorly graded gravel with sand, GP" or "clayey sand with gravel, SC" (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words "with cobbles" or "with cobbles and boulders" shall be added to the group name. For example: "silty gravel with cobbles, GM."

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For inter sample:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 13—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

NOTE 14—Other examples of soil descriptions and identifications are given in Appendixes X1 and X2.

NOTE 15—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %

Few—5 to 10 %

Little—15 to 25 %

Some—30 to 45 %

Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Index Terms

18.1 Classification, soil classification, visual classification, soil description, clay, silt, sand, gravel, organic soils.

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incorporated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the

percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL.SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP. SC/GC. GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP. SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML. CH/MH. SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH. MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time: the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

Designation: D 2573 – 72 (Reapproved 1978)

Standard Test Method for Field Vane Shear Test in Cohesive Soil[1]

This standard is issued under the fixed designation D 2573; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the field vane test in soft, saturated, cohesive soils. Knowledge of the nature of the soil in which each vane test is to be made is necessary for assessment of the applicability and interpretation of the test.

2. Summary of Method

2.1 The vane shear test basically consists of placing a four-bladed vane in the undisturbed soil and rotating it from the surface to determine the torsional force required to cause a cylindrical surface to be sheared by the vane; this force is then converted to a unit shearing resistance of the cylindrical surface. It is of basic importance that the friction of the vane rod and instrument be accounted for; otherwise, the friction would be improperly recorded as soil strength. Friction measurements under no-load conditions (such as the use of a blank stem in place of the vanes, or a vane that allows some free rotation of the rod prior to loading) are satisfactory only provided that the torque is applied by a balanced moment that does not result in a side thrust. As torsional forces become greater during a test, a side thrust in the instrument will result in an increase in friction that is not accounted for by initial no-load readings. Instruments involving side thrust are not recommended. The vane rod may be of sufficient rigidity that it does not twist under full load conditions; otherwise a correction must be made for plotting torque-rotation curves.

3. Apparatus

3.1 The vane shall consist of a four-bladed vane as illustrated in Fig. 1. The height of the vane shall be twice the diameter. Vane dimensions shall be as specified in Table 1. Sizes other than those specified in Table 1 shall be used only with the permission of the engineer in charge of the boring program. The ends of the vane may be tapered (see Fig. 1). The penetrating edge of the vane blade shall be sharpened having an included angle of 90°.

3.2 The vane shall be connected to the surface by means of steel torque rods. These rods shall have sufficient diameter such that their elastic limit is not exceeded when the vane is stressed to its capacity (Note 1). They shall be so coupled that the shoulders of the male and female ends shall meet to prevent any possibility of the coupling tightening when the torque is applied during the test. If a vane housing is used, torque rods shall be equipped with well-lubricated bearings where they pass through the housing. These bearings shall be provided with seals to prevent soil from entering them. The torque rods shall be guided so as to prevent friction from developing between the torque rods and the walls of casing or boring.

NOTE 1—If torque versus rotation curves are to be determined, it is essential that the torque rods be calibrated (prior to use in the field). The amount of rod twist (if any) must be established in degrees per foot per unit torque. This correction becomes progressively more important as the depth of the test increases and the calibration must be made at least to the maximum depth of testing anticipated.

3.3 Torque shall be applied to the torque rods, thence to the vane. The accuracy of the torque reading should be such that it will produce a variation not to exceed ±25 lb/ft² (1.20 kPa) shear strength.

3.4 It is preferable to apply torque to the vane with a geared drive. In the absence of a geared drive, it is acceptable to apply the torque directly by hand with a torque wrench or equivalent. The duration of the test should be controlled by the requirements of 4.3.

4. Procedure

4.1 In the case where a vane housing is used, advance the

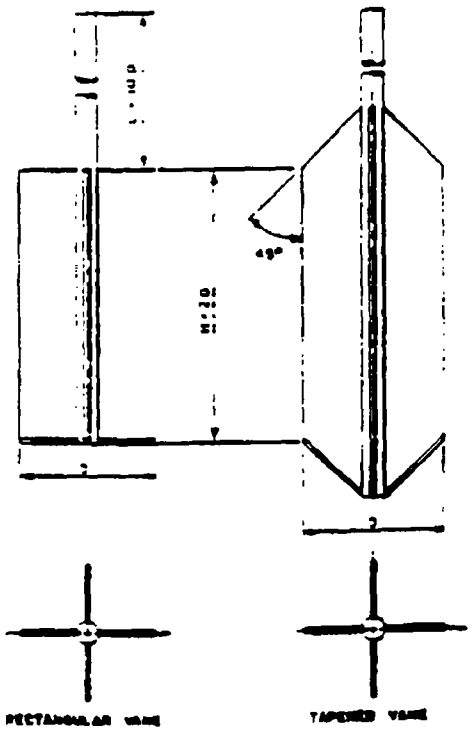


FIG. 1 Geometry of Field Vane

This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

Current edition approved Jan. 17, 1972. Published February 1972. Originally published as D 2573 – 67 T. Last previous edition D 2573 – 67 T.

TABLE 1 Recommended Dimensions of Field Vanes[A]

Casing Size	Diameter, in. (mm)	Height, in. (mm)	Thickness of Blade, in. (mm)	Diameter of Vane Rod, in. (mm)
AX	1½ (38.1)	3 (76.2)	1/16 (1.6)	½ (12.7)
BX	2 (50.8)	4 (101.6)	1/16 (1.6)	½ (12.7)
NX	2½ (63.5)	5 (127.0)	⅛ (3.2)	½ (12.7)
4 in. (101.6 mm)[B]	3⅝ (92.1)	7¼ (184.1)	⅛ (3.2)	½ (12.7)

[A] Selection of the vane size is directly related to the consistency of the soil being tested, that is, the softer the soil the larger the vane diameter.
[B] Casing diameter

housing to a depth which is at least five vane housing diameters less than the desired depth of the vane up. Where no vane housing is used, stop the hole in which the vane is lowered at a depth such that the vane up may penetrate undisturbed soil for a depth of at least five times the diameter of the hole.

4.2 Advance the vane from the bottom of the hole or the vane housing in a single thrust to the depth at which the test is to be conducted. Take precautions to make sure no torque is applied to the torque rods during the thrust.

4.3 With the vane in position, apply the torque to the vane at a rate which should not exceed 0.1°/s. This generally requires a time to failure of from 2 to 5 min, except in very soft clays where the time to failure may be as much as 10 to 15 min. In stiffer materials, which reach failure at small deformations, it may be desirable to reduce the rate of angular displacement so that a reasonable determination of the stress-strain properties can be obtained. During the rotation of the vane, hold it at a fixed elevation. Record the maximum torque. With apparatus with geared drives, it is desirable to record intermediate values of torque at intervals of 15 s or at lesser frequency if conditions require.

4.4 Following the determination of the maximum torque, rotate the vane rapidly through a minimum of 10 revolutions; the determination of the remoulded strength should be started immediately after completion of rapid rotation and in all cases within 1 min after the remoulding process.

4.5 In the case where soil is in contact with the torque rods, determine the friction between the soil and the rod by means of torque tests conducted on similar rods at similar depths with no vane attached. Conduct the rod friction test at least once on each site; this shall consist of a series of torque tests at varying depths.

4.6 In apparatus in which the torque rod is completely isolated from the soil, conduct a friction test with a blank rod (Note 2) at least once on each site to determine the magnitude of the friction of the bearings. In a properly functioning vane apparatus, this friction should be negligible.

NOTE 2—In some cases it is not necessary to remove the vane for the friction test. As long as the vane is not in contact with the soil, that is, where it is retracted into a casing, the friction measurement is not affected.

4.7 Conduct undisturbed and remoulded vane tests at intervals of not less than 2½ ft (0.76 m) throughout the soil profile when conditions will permit vane testing (Note 3). Do not conduct the vane test in any soil that will permit drainage or dilates during the test period, such as sands or silts or in soils where stones or shells are encountered by the vane in such a manner as to influence the results.

NOTE 3—This spacing may be varied only by the engineer in charge of the boring program.

5. Calculation

5.1 Calculate the shear strength of the soil in the following manner: The turning moment required to shear the soil is as follows:

$$T = s \times K$$

where:
T = torque, lbf·ft (or N·m),
s = shear strength of the clay, lbf/ft² (or kPa), and
K = constant, depending on dimensions and shape of the vane, ft³ (or m³).

5.2 Assuming the distribution of the shear strength is uniform across the ends of a cylinder and around the perimeter, calculate the value of K as follows:

Inch-Pound Units:

$$K = (\pi/1728) \times (D^2H/2) \times [1 + (D/3H)]$$

Metric Units:

$$K = (\pi/10^9) \times (D^2H/2) \times [1 + D/3H)]$$

where:
D = measured diameter of the vane, in. (or cm), and
H = measured height of vane, in. (or cm).

It is important that these dimensions are checked periodically to ensure the vane is not distorted or worn.

5.3 As the ratio of length to breadth of the vane is 2:1, the value of K may be simplified in terms of the diameter so that it becomes the following:

Inch-Pound Units:

$$K = 0.0021D^3$$

Metric Units:

$$K = 0.00000366D^3$$

5.4 Since the value of s is required, it is more useful to write the equation as follows:

$$s = T \times k$$

where:
k = $1/K$ and
T, the torque, is measured so that s can be calculated.

5.5 For the tapered vane of Fig. 1, the following modified equation may be used for the vane constant:

Inch-Pound Units:

$$K = 1/1728 [\pi D^3 + 0.37 (2D^3 - d^3)]$$

Metric Units:

$$K = 1/10^9 [\pi D^3 + 0.37 (2D^3 - d^3)]$$

where:
d = rod diameter, in. (cm). For a ½-in. (1.27-cm) rod this reduces to:

Inch-Pound Units:

$$K = 0.00225D^3 - 0.00003$$

Metric Units:

$$K = 0.00000388D^3 - 0.00000076$$

6. Report

6.1 For each vane test record the following observations:

6.1.1 Date of the test.

6.1.2 Boring number.

6.1.3 Size and shape of the vane (tapered or rectangular).

6.1.4 Depth of the vane tip.

6.1.5 Depth of the vane tip below the housing or bottom of the hole.

6.1.6 Maximum torque reading, and intermediate readings if required for the undisturbed test.

6.1.7 Time to failure of the test.

6.1.8 Rate of remolding,

6.1.9 Maximum torque reading for the remolded test, and

6.1.10 Notes on any deviations from standard test procedure.

6.2 In addition, record the following observations for the boring:

6.2.1 Boring number.

6.2.2 Location.

6.2.3 Log of the soil conditions.

6.2.4 Reference elevation.

6.2.5 Method of making the hole.

6.2.6 Description of the vane, that is, housed or not.

6.2.7 Description of the method of applying and measuring the torque.

6.2.8 Notes on the driving resistance.

6.2.9 Name of the drilling foreman, and

6.2.10 Name of the supervising engineer.

ASTM D4538 – STANDARD PRACTICE FOR COLLECTION OF FLOOR DUST SAMPLES

Designation: D 5438 – 00

Standard Practice for Collection of Floor Dust for Chemical Analysis[1]

This standard is issued under the fixed designation D 5438, the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for the collection of a sample of dust from carpets and bare floors that can be analyzed for lead, pesticides, or other chemical compounds and elements.

1.2 This practice is applicable to a variety of carpeted and bare floor surfaces. It has been tested for level loop and plush pile carpets and bare wood floors, specifically.

1.3 This practice is not intended for the collection and evaluation of dust for the presence of asbestos fibers.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 422 Test Method for Particle-Size Analysis of Soils[2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[3]

E 1 Specification for ASTM Thermometers[4]

E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)[3]

F 608 Test Method for Evaluation of Carpet-Embedded Dirt Removal Effectiveness of Household Vacuum Cleaners[5]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D 1356.

3.1.1 *carpet-embedded dust*—soil and other particulate matter, approximately 5-μm equivalent aerodynamic diameter and larger, embedded in carpet pile and normally removable by household vacuum cleaners.

3.1.2 *surface dust*—soil and other particulate matter, approximately 5-μm equivalent aerodynamic diameter and larger, adhering to floor surfaces and normally removable by household vacuum cleaners.

4. Summary of Practice

4.1 The sampling method described in this practice is taken from work published in Roberts, et al,[6,7,8] and Stamper, et al.[9]

4.2 Particulate matter is withdrawn from the carpet or bare floor by means of a flowing air stream passing through a sampling nozzle at a specific velocity and flow rate and separated mechanically by a cyclone. The cyclone collects efficiently particles approximately 5-μm mean aerodynamic diameter and larger. The sampling system allows for height, air flow, and suction adjustments to reproduce systematically a specific air velocity for the removal of particulate matter from carpeted and bare floor surfaces, so that these sampling conditions can be repeated.

NOTE 1—Side-by-side comparison of the HVS3 and a conventional upright vacuum cleaner revealed that both collected particles down to at least 0.2 μm and that the HVS3 was more efficient at collecting particles smaller than 20 μm than conventional vacuum cleaners.[10] If desired, a fine-particle filter may be added downstream of the cyclone to collect 99.9 % of particles above 0.2 μm aerodynamic mean diameter.

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

Current edition approved March 10, 2000. Published June 2000. Originally published as D 5438 – 93. Last previous edition D 5438 – 94.

[2] *Annual Book of ASTM Standards*, Vol 04 08.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 14.03

[5] *Annual Book of ASTM Standards*, Vol 15.07.

[6] Roberts, J. W., Budd, W. T., Ruby, M. G., Stamper, V. R., Camann, D. E., Fortman, R. C., Sheldon, L. S., and Lewis, R. G., "A Small High Volume Surface Sampler HVS3 for Pesticides, and Other Toxic Substances in House Dust," *Paper No 91-150.2*, 84th Annual Meeting, Air & Waste Management Association, Vancouver, British Columbia, June 16–21, 1991.

[7] Roberts, J. W., and Ruby, M. G., "Development of a High Volume Surface Sampler for Pesticides," *U.S Environmental Protection Agency Report No. EPA 600/4-88/036*, Research Triangle Park, NC, January 1989.

[8] Roberts, J. W., Han, W., and Ruby, M. G., "Evaluation of Dust Samplers for Bare Floors and Upholstery," Battelle Subcontract No. 46534(g21733808)-00 03EQ, EPA Prime Contract No. 68-00-0007, U.S. EPA, AREAL, Office of Research and Development, 1993.

[9] Stamper, V. R., Roberts, J. W., and Ruby, M. G., "Development of a High Volume Small Surface Sampler for Pesticide and Toxics in House Dust," *Research Triangle Institute Report No RTI/171-01/02F*, Research Triangle Park, NC, June 1990. Included in supporting data, which are on file at ASTM Headquarters. Request RR:D22-1010

[10] Willis, R. D., "SEM Characterization of House Dusts Collected by Conventional Vacuum and the HVS3 Sampler." Report to the U.S. EPA under Contract 68–D5–0049, Research Triangle Park, NC, ManTech Environmental Technology, Inc., 1995.

4.3 The particulate matter in the air stream is collected in a catch bottle attached to the bottom of the collection cyclone. This catch bottle shall be capped for storage of the sample and transported to the laboratory for analysis.

5. Significance and Use

5.1 This practice may be used to collect dust from carpeted or bare floor surfaces for gravimetric or chemical analysis. The collected sample is substantially unmodified by the sampling procedure.

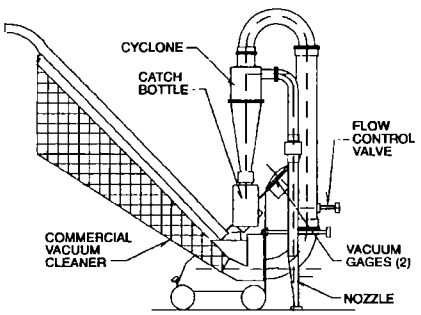


FIG. 1 Floor Dust Sampler Using a Commercial Vacuum Cleaner as the Suction Source

5.2 This practice provides for a reproducible dust removal rate from level loop and plush carpets, as well as bare floors. It has the ability to achieve relatively constant removal efficiency at different loadings of surface dust.

5.3 This practice also provides for the efficient capture of semivolatile organic chemicals associated with the dust. The test system can be fitted with special canisters downstream of the cyclone for the capture of specific semivolatile organic chemicals that may volatilize from the dust particles during collection.

5.4 This practice does not describe procedures for evaluation of the safety of floor surfaces or the potential human exposure to carpet dust. It is the user's responsibility to evaluate the data collected by this practice and make such determinations in the light of other available information.

6. Interferences

6.1 There are no known interferences to the determination of dust loadings covered by this practice.

7. Apparatus

7.1 *Sampling Apparatus*, which may be acquired commercially[11] (as shown in Fig. 1) or constructed as follows:

7.1.1 The dimensions of the sampling apparatus (nozzle size, cyclone diameter, cyclone inlet diameter, etc.) are interdependent. The flow rate must produce a sufficient velocity both at the sampled surface and in the cyclone. The cyclone must have a cut diameter of 5 μm at the same velocity that will provide a horizontal velocity of 40 cm/s at 10 mm from the nozzle in the carpet material, or 5 mm from the nozzle on bare floors. The fundamental principles of this device have been discussed in detail in Roberts, et al.[6,7,8]

7.1.2 *Nozzle*—The edges and corners of the sampling nozzle shall be rounded to prevent catching the carpet material. The nozzle must be constructed to allow for sufficient suction to separate loose particles from the carpet or bare floor and carry them to the cyclone. It must have an adjustment mechanism to establish the nozzle lip parallel to the surface and to achieve the proper suction velocity and pressure drop across the nozzle. A nozzle 12.4 cm long and 1 cm wide, with a 13-mm flange and tapered to the nozzle tubing at no more than 30°, will yield the appropriate velocities when operated as specified in Section 11.

7.1.3 *Gaskets*—Gaskets in joints should be of a material appropriate to avoid sample contamination.

7.1.4 *Cyclone*—The cyclone shall be of a specific size such that a given air flow allows for separation of the particles 5-μm mean aerodynamic diameter and larger. The cyclone must be made of aluminum or stainless steel, and the catch bottle must be made of clear glass or fluorinated ethylene propylene (FEP) to avoid contamination and allow the operator to see the sample.

7.1.5 *Flow Control System*—The flow control system shall allow for substantial volume adjustment. The suction source must be capable of drawing 12 L/s (26.5 CFM) through the system with no restrictions other than the nozzle, cyclone, and flow control system connected. An upright commercial vacuum cleaner with a 7 amp or greater motor capable of pulling a vacuum of 6.5 kPa may be used for this purpose.

7.1.6 *Flow Measuring and Suction Gages*— Two vacuum gages are required — one with a range of 0 to 3.7 kPa (0–15 in. water) is used for setting flow rate and another with a range

[11] The sampling device used in the development and performance evaluation of this test method was manufactured by CS-3, Inc., P.O. Box 1461, Sandpoint, ID 83864, which is the sole source of supply of the sampler known to the committee at this time. If you are aware of alternative suppliers, please provide this information to the Committee on Standards, ASTM Headquarters, 100 Barr Harbor Dr., West Conshohocken, PA 19428. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

of 0 to 2.5 kPa (0–10 in. water) is used to set the pressure drop across the vacuum nozzle.

7.1.7 Optional filter holder assembly with appropriate fine particle filter, such as a 25–cm micro-quartz-fibre, binderless, acid-washed filter.[12]

7.2 *Other Equipment*:

7.2.1 *Stopwatch.*

7.2.2 *Masking Tape and Marking Pen*, for outlining sections for sampling.

7.2.3 *Clean Aluminum Foil and Clean Glass or FEP Jars*, for the collection and storage of samples.

7.2.4 *Thermometer* (see Specification E 1).

7.2.5 *Relative Humidity Meter* (see Test Method E 337).

7.2.6 *Shaker Sieve*, as specified in Test Method D 422, with 100 mesh-screen above the pan to separate the fine dust below 150 μm.

7.2.7 *Analytical Balance*, sensitive to at least 0.1 mg and having a weighing range from 0.1 mg to 1000 g.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[13]

8.2 Methanol is required for sampling train cleaning after sample collection.

9. Sampling Strategy

9.1 The overall sampling strategy should be designed to address the goals of the study. Users should consider factors such as foot traffic volume, types of activities, proximity to potential sources, etc. The sampling strategy should be described in the sampling report so it can be taken into consideration when readers are comparing loadings or concentrations, or both, to those obtained from other studies. The ideal sampling location(s) for the beginning of the test procedure are an area that conforms with the protocol for the user's overall sampling strategy. For example, when sampling in a home for child exposure assessment, protocol may require the selection of a carpeted area for sampling where small children play or are likely to play.

10. Pretest Preparation and Calibration

10.1 *Calibration*—The sampling system described in this practice does not have any calibrated flow devices other than the cyclone and the Magnehelic gages. The cyclone used for the separation of the particles must be designed to give proper separation at varying flow rates throughout the sampling range of the system. The pressure gages and any other devices (that is, temperature gage) used for testing purposes should be calibrated against a primary standard.

10.1.1 *Pressure Gages*—Pressure gages shall be calibrated against an inclined manometer or other primary standard prior to any field test. One means of checking a Magnehelic gage is to set a flow rate through the sampling system with a manometer and then switch to the Magnehelic gage. If the difference in the readings is more than 3 %, the gage is leaking or is in need of repair or calibration. This should be done at two different flow rates when checking the gage.

10.1.2 The cyclone flow measurement is calibrated with a laminar flow element, spirometer, or roots meter. See the appendix for cyclone calibration with a laminar flow element.

10.2 *Pretest Preparation*:

10.2.1 Each catch bottle to be used shall be clean and inspected for any contamination. The bottles should be marked with masking tape and a marking pen for identification of the test site, time, and date.

10.2.2 The sampling train shall be inspected to ensure that it has been cleaned and assembled properly.

10.2.3 The sampling train shall be leak-checked prior to sampling. This can be accomplished by placing a mailing envelope or a piece of cardboard beneath the nozzle and switching on the suction source. The flow Magnehelic gage should read 5 Pa (0.02 in. H_2O) or less to ensure that the system is leak free. If any leakage is detected, the system shall be inspected for the cause and corrected before use.

11. Sampling

11.1 *Sampling a Carpeted Floor*:

11.1.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for a sample data form.)

11.1.2 Select a sampling area according to the established protocol for your sampling campaign. This should be determined prior to testing.

11.1.3 A typical sampling procedure may use measuring tapes placed on the carpet so that they are parallel to each other and on either side of the portion of carpet to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5-m apart and extended as far as practical. They should be taped to the carpet with masking tape every 30 cm.

11.1.4 Place the sampler in one corner of the sampling area and adjust the flow rate and pressure drop according to the type of carpet (see 11.8). The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and distance between the surface and the nozzle flange.

11.1.5 Clean the wheels and nozzle lip with a clean laboratory tissue immediately before sampling. Begin sampling by moving the nozzle between the ends of the two measuring tapes. The sampler is then moved back and forth four times on the first strip, moving the sampler at approximately 0.5 m/s. (The widths of the strips are defined by the width of the sampling nozzle.) Effective nozzle width is 13 cm for the CS_3 sampler. Move in a straight line between the numbers on the measuring tape. Angle over to the second strip on the next pass gradually, and repeat four double passes. After sampling

[12] A filter holder for circular 25–cm particle filters and flow control valve assembly which replaces the normal flow control assembly is available from the manufacturer of the floor vacuum device.

[13] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

SAMPLE DATA SHEET

Operator _____ Date _____ Sample Ident. #: ____

Sampling site _____

Type of Carpet: Plush ___ Level Loop ___ Multilevel ___ Shag ___
Type of Vacuum: Upright ___ Canister ___ Other _____

Last Vacuumed _____ Temp. _____ Humidity _____%

Comments: _____

_____

Location of Area Sampled: _____ Area _____m^2

Sketch of Area Sampled:

Leak Check: Yes__ No__; 20 second cleaning @ end: Yes __ No __

Total Sample Time:__minutes' __seconds Flow ▲P _____ Nozzle ▲P ___

Bottle final Wt: _____g Tare Wt: _____g Net Wt: _____g

Pan & Sample Wt: _____g Pan Tare Wt: _____g Net Wt: _____g

Total Dust: _____ grams/m^2
Fine Dust: _____ grams/m^2

Cyclone Sample #: _____

Lab Sample #: _____

FIG. 2 Sample Data Sheet for Sampling for Floor Dust

approximately 0.5 m^2, determine the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm (0.25 in.) in a 55-mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample an additional 0.5 m^2 next to the area already sampled. Hair, carpet fibers, and other large objects should be excluded from the sample when estimating the quantity collected.

11.1.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch

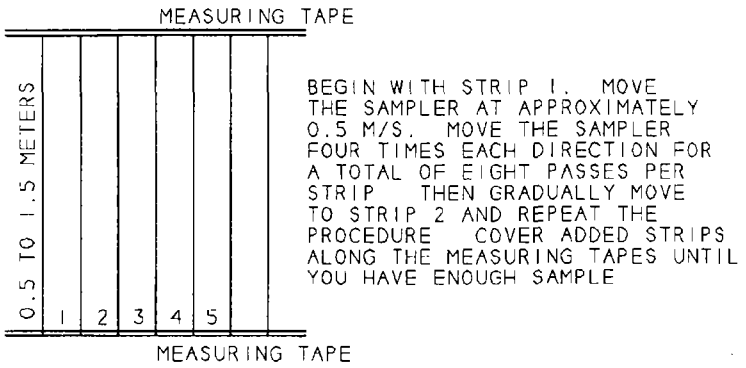


FIG. 3 Example of a Typical Sampling Procedure

bottle can now be removed, labeled, and capped for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.1.7 If the rug area to be sampled is very dirty, or has not been cleaned frequently, care must be taken to avoid filling up the cyclone catch bottle on the first sample area. If it is suspected that this will be the case, start with a 0.25-m^2 sampling area. Then take a second and a third area as before, until the catch bottle is 75 % full.

11.1.8 Adjust the flow rate and nozzle pressure drop to values that approximate those given in Table 1. Use the same flow rate and pressure drop on multilevel and shag carpets as that used for plush carpets.

11.2 *Sampling a Bare Floor*:

11.2.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for sample data form.)

11.2.2 Select a sampling area that is as large as possible and according to the established protocol for your sampling campaign. This should be determined prior to testing. Divide the area into parallel areas 0.5 to 1.5 m apart.

11.2.3 A typical sampling procedure may utilize measuring tapes placed on the floor so that they are parallel to each other and on either side of the portion of floor to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5 m apart and extended as far as practical. They should be taped to the floor every 30 cm with masking tape.

11.2.4 Place the sampler in one corner of the sampling area. Set the height of the nozzle above the floor at approximately 1 mm (a U.S. penny under the nozzle lip will hold it at this height) and adjust the flow rate (see 11.2.7). The two factors that affect the efficiency of the sampling system are the flow rate and the pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and the distance between the surface and nozzle flange.

11.2.5 Clean the wheels and nozzle lip immediately before sampling with a clean laboratory tissue. Begin sampling by moving the nozzle between the ends of the two tapes. The sampler is then moved back and forth two times on the first strip, moving the sampler at approximately 0.5 m/s. (The width of the strips are defined by the width of the sampling nozzle. For the CS_3 sampler, effective nozzle width is 13 cm. Move in a straight line between the numbers on the measuring tape. Gradually angle over to the second strip on the next pass and repeat two double passes. After sampling approximately 10 m^2, check the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm (0.25 in.) in a 55 mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample additional areas as available. It may not be possible to obtain 6 g of dust from a clean or small bare floor.

11.2.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch bottle can now be removed, capped, and labeled for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.2.7 Adjust the flow rate to a flow of 9.5 L/s (20 CFM).

TABLE 1 Approximate Values for Flow Rate and Nozzle Pressure Drop

Carpet Type	Flow Rate	Nozzle Pressure Drop
Plush	9 5 L/s (20 CFM)	2.2 kPa (9 in. H_2O)
Level loop	7.6 L/s (16 CFM)	2.5 kPa (10 in. H_2O)

12. Sample Analysis

12.1 After collection of the sample in the catch bottle, the sample may be left in the same bottle or transferred to another container for transport to the laboratory. The procedure for sample handling is different for metals and organic chemicals. Samples for organic analysis should be maintained at 4°C to the extent possible. (Samples should not be frozen before sieving, as this could alter the particle size distribution.) Storage at ambient temperature is appropriate for samples that will be analyzed only for metals, but cooling the sample is also acceptable.

12.2 If the sample will be analyzed for pesticides or other organic chemicals, transfer the dust from the cyclone catch bottle onto the middle of a piece of aluminum foil that has been cleaned by washing with pesticide-free methanol or hexane. Fold the foil into a small package carefully, keeping the dust in the middle. Place the foil pouch in a clean glass jar. Cover the jar opening with another piece of precleaned foil and secure the lid to the jar. Seal the seam of the lid to the jar with polytetrafluoroethylene tape. Place the sample jar in an ice chest to keep it cool during transport to the laboratory. Label the jar for reference.

12.3 If the sample will be analyzed for metals, it can be transferred from the catch bottle to a new polyethylene "zipper" seal sample bag. Seal the zipper, and tape the seal with any marking tape that will adhere well to the polyethylene bag. Label the sample for reference.

TABLE 2 Sampling Efficiency Using Modified Laboratory Test Method F 608[A]

Parameters	Carpet Type	
	Plush	Level Loop
Flow rate (L/s)	9.4	7 6
Delta P (kPa)[B]	2.3	2.5
Mean % of mass collected in cyclone	69.5	66.8
Standard deviation	1 2	2 8
Number of tests	3	3

[A] Carpet dust loading was 15.9 g/m^2.
[B] Pressure drop at nozzle

12.4 Sieve the samples for 5 min in a shaker in accordance with Method D 422, with a 100-mesh screen above the pan, to determine the weight of fine dust below 150-μm mean diameter.

12.5 Alternative methods for the storage, shipment, and preparation of samples for analysis may be required for some analytes and should be prescribed for specific sampling protocols. The FEP catch-bottle may be used for storage and shipping.

13. Sampler Cleaning

13.1 After the sample bottle is removed, open the flow control valve to maximum flow, tip the sampler back so that the nozzle is approximately 5 cm (2 in.) off the floor, and switch the vacuum on. Place a hand covered by a rubber glove over the bottom of the cyclone and alternate closing and opening the cyclone for 10 s to free any loose material adhering to the walls of the cyclone and tubing. It is not necessary to catch this small amount of dust, as it is usually much less than 1 % of the collected sample.

13.2 Remove the sampler to a well-ventilated cleaning area free of dust. Remove the cyclone and elbow at the top of nozzle tubing from the sampler. Use a 50-cm long by 3-cm diameter (20 by 1.25-in.) brush to clean the nozzle, and clean all related items up to and including the cyclone and catch bottle with reagent grade methanol. This wash can be analyzed at the discretion of the operator. The total amount of dust removed in the air and wet cleaning is usually much less than 1 % of the collected dust. The air and wet cleaning is performed to prevent contamination from passing from one sample to another.

14. Data Analysis

14.1 Weigh the sieved dust sample with an analytical balance accurate to 0.1 mg.

14.2 Calculate the dust weight by subtracting the weight of the pan sample from the final weight according to Method D 422.

14.3 Calculate the loading for dust per square metre (g/m^2) by dividing the final dust weight by the area sampled (expressed in m^2).

14.4 When the analysis results are received from the laboratory, it is possible to calculate the loading of lead, pesticides, or other analytes per square metre of carpet or bare floor area (μg/m ^{2}) in the same way.

14.5 The concentration of any element or chemical associated with the dust may be determined by analysis.

15. Precision and Bias [14]

15.1 Tests for dust collection efficiency have been performed using Test Method F 608 modified by passing it through a 100-mesh sieve.[6,7] The results are given in Table 2.

15.2 Tests performed with a fine particle filter downstream of the cyclone showed that 99 % or more of the collected test dust was retained in the cyclone catch bottle.[6,7]

15.3 Tests performed as in 15.2, but with test dust containing lead, showed that 99 % or more of the lead collected was retained in the cyclone catch bottle.[6,7]

15.4 Tests performed as in 15.2, but with test dust fortified with pesticides, showed that 97 % or more of the pesticides collected were retained in the cyclone catch bottle. The pesticides tested were chlordane, aldrin, chlorpyrifos, heptachlor, and diazinon.

15.5 Tests were conducted on conditioned carpets, as described in Test Method F 608.

16. Keywords

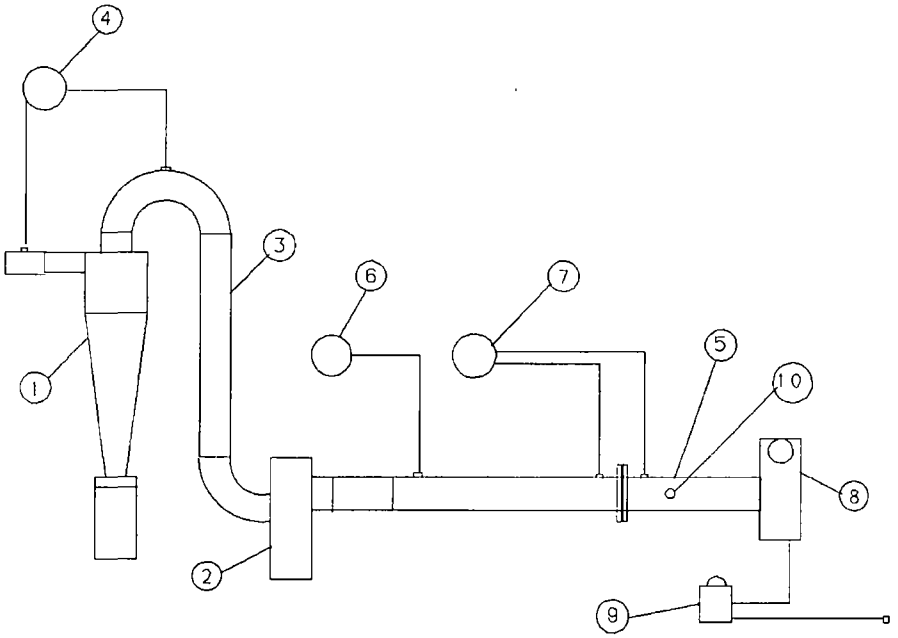
16.1 carpet; cyclone; dust; floors; metals; organic chemicals; particle size; particulate matter; vacuum

[14] Supporting data have been filed at ASTM Headquarters. Request RR:D22-1010.

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF CYCLONE USING A LAMINAR-FLOW ELEMENT



1. Cyclone
2. Suction/blower
3. Flow control valve
4 0 to 2 5 kPa vacuum gage
5. Laminar flow element
6. ±0.25 kPa vacuum gage
7. 0 to 1 kPa vacuum gage
8. Suction/blower
9. Variable autotransformer
10. Temperature gage

FIG. X1.1 Calibration Using a Laminar Flow Element

X1.1 Assemble the necessary components (see Fig. X1.1).

X1.1.1 *Cyclone.*

X1.1.2 *Suction/Blower.*

X1.1.3 *Flow Control Valve*, 1 to 2.5 kPa (0 to 10 in.).

X1.1.4 *Magnehelic Gage*, 1 to 2.5 kPa (0 to 10 in.)

X1.1.5 *Laminar Flow Element* (with manufacturer's certified calibration), with pressure gages and dial thermometer.

X1.1.6 *Suction/Blower*, with power transformer; leak check the system by plugging the inlet to the cyclone and observing the pressure gage.

X1.1.7 Activate Blowers 2 and 8.

X1.1.8 Open the flow control valve on Flow Control Valve 3 so that 2.0 kPa (8.0 in. H_2O) registers on Pressure Gage 4. Then adjust Variable Autotransformer 9 so that 0.0 kPa (0.0 in. H_2O) registers on Pressure Gage 6. Some adjusting of the flow control valve will be necessary.

X1.1.9 Check Pressure Gage 7 for the gas flow reading and record the flow.

X1.1.10 Adjust the flow through the cyclone to 2.5 kPa (10.0 in. H_2O), and repeat the procedure. This action should provide a gas flow rate through the cyclone. This should be between 7.1 and 8.5 L/s (15 to 18 CFM).

XRF USER'S GUIDE

SPECTRACE 9000 FIELD PORTABLE X-RAY FLUORESCENCE OPERATING PROCEDURES

SOP#: 1713
DATE: 01/26/95
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedur e (SOP) is to serve a s a guide to the start-up, check out, operation, calibration, and rout ine use of the Spectrace 9000 field portable x-ray fluorescence instrument for field use in screening hazardous or potentiall y hazardous inorganic materials. It is not intended t o replace or diminish the use of the Spectrace 900 0 Operating Instructions. The Operating Instruction s contain additional information for optimizin g instrument performance and for utilizing differen t applications.

The procedures contained h erein are general operating guidelines which may be changed as required , depending on site conditions, equipment limitations , limitations imposed by Quality Assurance\Qualit y Control (QA\QC) procedure or other protoco l limitations. In all instances, the procedures finall y employed should be documented and included in any or all final reports. Mention of trade names o r commercial produc ts does not constitute endorsement or recommendation for use.

1.1 Principles of Operation

X-ray Fluorescence (XRF) spectroscopy is a no n destructive qualitative and quantitative analytica l technique used to determine the chemical composition of samples. In a source excited XRF analysis , primary X-rays emitted from a sealed radioisotop e source are utilized to irradiate samples. Durin g interaction with samples, source X-rays may eithe r undergo scattering (do minating process) or absorption by sample atoms in a process known as th e photoelectric effect (absorption coefficient). Thi s phenomenon originates when incident radiatio n knocks out an electron from the innermost shell of an atom creating a vacancy. The atom is excited an d releases its surplus energy almost instantly by filling the vacancy with an electron from one of the highe r energy shells. This rearrangement of electrons i s associated with the emission of X-rays characteristic (in terms of energy) of the given atom. This process is referred to as emission of fluorescent X-ray s (fluorescent yield). The overall efficiency of th e fluorescence process is referred to as excitatio n efficiency and is proportional to the product of th e absorption coefficient and the fluorescent yield.

1.1.1 Characteristic X-rays

The Spectrace 9000 utilizes characteristic X-ray lines originating from th e innermost shells of the atoms: K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for an y element and, therefore, are the preferred analytica l lines. The K lines are always accompanied by the L and M lines of the same element. However, wit h energies much lower than those of the K lines, the y can usually be ne glected for those elements for which the K lines are analytically useful. For heavy ele - ments such as cerium (Ce) (atomic number [Z]=58) , to uranium (U, Z=92), the L lines are the preferre d lines for analysis. The L_{α} and L_{β} lines have almos t equal intensities, and the choice of one or the othe r depends on what interfering lines might be present. A source just e nergetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines.

The Spectrace 9000 Operating Instructions contain a table that identifies the X-rays (K or L) and elements measured for each excitation source.

An X-ray source can excite characteristic X-rays from an element only if the source energy is greater tha n the absorption edge energy for the particular lin e group of the element (e.g., K absorption edge, L ab - sorption edge, M absorption edge). The absorptio n edge energy is somewhat greater than the correspon- ding line energy. Actually, the K absorption edg e energy is approximately the sum of the K, L, and M line energies, and the L absorption edge energy i s approximately the sum of the L and M line energies of

the particular element.

Energies of the characteristic fluorescent X-rays are converted (with in the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer (electronic unit) measures the pulse amplitudes, which is the basis of a qualitative X-ray analysis. The number of counts at a given energy is representative of element concentration in a sample and is the basis for quantitative analysis.

1.1.2 Scattered X-rays

The source radiation is scattered from the sample by two physical processes: coherent or elastic scattering (no energy loss), and Compton or inelastic scattering (small energy loss). Thus, source backscatter (background signal) actually consists of two components with X-ray lines close together. The higher energy line is equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. Furthermore, the scattered X-rays have the highest energies in the spectrum and, therefore, contribute most of the total measured intensity signal.

1.2 Sample Types

Solid and liquid samples can be analyzed for elements aluminum (Al) through uranium (U) with proper X-ray source selection and instrument calibration. Typical environmental applications are:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings, etc.), sludges, and liquids (e.g., lead (Pb) in gasoline)
- Light elements in liquids (e.g., phosphorus [P], sulphur [S], and chlorine [Cl] in organic solutions)
- Heavy metals in industrial waste stream effluents
- PCB in transformer oil by Cl analysis
- Heavy metal air particulates collected on membrane filters, either from personnel samplers or from high volume samplers.
- Lead (Pb) in paint

2.0 METHOD SUMMARY

The Spectrace 9000 Portable XRF Analyzer employs three radioactive isotope sources: iron-55 (Fe-55), cadmium-109 (Cd-109), and americium-241 (Am-241) for the production of primary X-rays. Each source emits a specific set of primary X-rays which excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the appropriate source is selected according to its excitation efficiency for the element of interest. See page 1-2 of the Spectrace 9000 Operating Instructions for a chart of source type versus element range.

The sample is positioned in front of the source - detector window and sample measurement is initiated which exposes the sample to primary radiation from the source. Fluorescent and backscattered X-rays from the sample enter through the beryllium (Be) detector window and are counted in the high resolution mercuric iodide (HgI_2) detector.

Elemental concentrations are computed using a Fundamental Parameter (FP) algorithm of the form:

$$\text{Concentration} = R \times S \times (1 + \text{SUM}\{A_n \times C_n\})$$

"R" is the measured analyte X-ray intensity relative to the pure element; "S" is a calculated sensitivity coefficient. The quantity SUM{} is a summation of "n"-element absorption-enhancement terms containing calculated alpha-coefficients and iteratively computed element concentrations. The Spectrace 9000 utilizes FP XRF calibrations derived from theoretical considerations (as opposed to empirical data). The menu-driven software in the Spectrace 9000 supports multiple XRF calibrations called "applications." Each application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of FP calibration coefficients.

The measurement time of each source is user-selectable. The shorter source measurement times (15 - 30s) are generally used for initial screening and hot spot delineation, while longer measurement times (30 - 500s) are typically used for higher precision and accuracy requirements.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This SOP specifically describes equipment operating procedures for the Spectrace 9000; hence, this section is not applicable to this SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user or application related error. Generally, the instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally more significant and will vary with each site and method used. The components of the user or application related error are the following.

4.1 Sample Placement

This is a potential source of error because the X-ray signal decreases as the distance from the radioactive source is increased. However, this error is minimized by maintaining the same distance for each sample.

4.2 Sample Representivity

In order to accurately characterize site conditions, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability. For additional information on representative sampling, refer to the "Removal Program Representative Sampling Guidance, Volume 1 - Soil." [1]

4.3 Reference Analysis

Soil chemical and physical matrix effects may be corrected by using site-specific soil samples which have been analyzed by Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectroscopy as calibration samples. A major source of error can result if these samples are not representative of the site and/or if the analytical error is large. Additionally, when comparing XRF results with reference analyses results, the efficiency of the sample digestion reference analysis should be considered. Some digestion methods may breakdown different sample matrices more efficiently than others.

4.4 Chemical Matrix Effects (Due to the Chemical Composition of the Sample)

Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, iron (Fe) tends to absorb copper (Cu) X-rays, reducing the intensity of Cu measured by the detector. This effect can be corrected mathematically through the use of FP coefficients.

4.5 Physical Matrix Effects (Due to Sample Morphology)

Physical matrix effects are the result of variations in the physical character of the sample. They may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much courser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles; this results in a higher intensity reading for that sample and, consequently, an apparently higher measured concentration for that element.

4.6 Application Error

Generally, the error in the application calibration model is insignificant (relative to the other sources of error) **PROVIDED** the instrument's operating

instructions are followed correctly. However, if th e sample matrix varies significantly from the design of the application, the error may beco me significant (e.g., using the soils application to analyze a 50 percent iron mine tailing sample).

4.7 Moisture Content

Sample moisture content will affect the analytica l accuracy of soils or sludges . The overall error may be secondary when the moisture range is small (5-2 0 percent), or it may be a major source of error whe n measuring the surface of soils that are saturated with water.

4.8 Cases of Severe X-ray Spectrum Overlaps

When present in the sample, certain X-ray lines from different elements can be very close in energy and , therefore, can interfere by producing a severel y overlapped spectrum.

The typical spectral overlaps ar e caused by the K_β line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with t he K_α line of element Z. This is the so-called K_α/K_β interference. Since the K_α :K_β intensity ratio for the given element usually varie s from 5:1 to 7:1, the interfering element, Z-1, must be present in large concentrations in order to disturb the measure ment of analyte Z. The presence of larg e concentrations of vanadium (V) could disturb th e measurement of chromium (Cr). The V K_α and K_β energies are 4.951 and 5.427 Kev, respectively. The Cr K_α energy is 5.41 Kev. The resolution of th e detector is approximately 270 eV. Therefore, larg e amounts of V in a sample will result in spectra l overlap of the V K_β with the Cr K_α peak (see Figure 1, Appendix A) and the measure d X-ray spectrum will include TOTAL counts for Cr plus V lines.

Other interferences arise from K/L, K/M, and L/ M line overlaps. While these are less common, th e following are examples of severe overlap:

$$As\ K_\alpha/Pb\ L_\alpha,\quad S\ K_\alpha/Pb\ M_\alpha$$

In the arsenic (As)/lead case, Pb can be measure d from the Pb L_β line, and a rsenic from either the As K_α or the As K_β line; this way the unwanted interference can be corrected. However, due to the limits o f mathem atical corrections, measurement sensitivity is reduced. Generally, ar senic concentrations can not be efficiently calculated in samples with Pb:As ratios of 10:1 or more. This may result in zero arsenic bein g reported regardless of what the ac tual concentration is.

The Spectrace 9000 us es overlap factors to correct for X-ray spectral overlaps for the elements of interest for a given application.

5.0 EQUIPMENT / APPARATUS

5.1 Description of the Spectrace 9000 System

The analyzer utilizes the meth od of Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometry t o determine the elemental comp osition of soils, sludges, aqueous solutions, oils, and other waste materials.

The Spectrace 9000 analyzer includes three compact, sealed radiation sources contained in a measurin g probe: Fe-55, Cd-109, and Am-241. The analyze r software automatically select s which sources to use as well as measurement time for each source based o n stored information for each application. The probe is equipped with a high resolution HgI_2 detector, which is connected by cable to an environmentally seale d electronic module.

The electronic unit provides internal non volatil e memory for storage of 120 spectra and 300 multi - element analysis reports. An RS-232 serial port i s provided for downloading data and spectra to a peripheral device. The multi-element analysis reports and the 2000-channel spectra can be displayed on the instrument's LCD panel. The replaceable an d rechargeable internal battery provides for field - portable operation.

The Spectrace 9000 is supplied with three factory - installed FP-based applications (calibrations). Th e "Soil Samples" application is for analysis of soil s where the balance of the sample (that portion no t directly measured by the instrument) is silica (SiO_2). The "Thin Film" application is for analysis of thi n films such as air monitoring filters or wipes. Finally, the "PbK in Paint" application is for analyzing Pb in paint films and is reasonably independent of the type of substrate. Spectrace Instruments will also develop calibrations to meet new user appl ication requirements (e.g., adding elements to the present "Soil Samples " application). The PC-based Spectrace 900 0 Application Generater software may also be used t o

develop new applications.

The Spectrace 9000 can be powered from a 115-volt (or 220-volt) wall outlet or from its 4-hour capacity battery. It can be operated in temperatures ranging from 32 to 120° Fahrenheit (F). Furthermore, the probe and electronic unit may be exposed to a light rain. However, additional protection is provided when the system (electronic unit and probe) is contained in the optional water repellant carrying case.

5.2 Equipment and Apparatus List

5.2.1 Spectrace 9000 Analyzer System

The complete Spectrace 9000 Analyzer System includes:

- Analyzer unit for data acquisition, processing, and display
- Hand-held probe including:
  - High-resolution HgI_2 detector
  - Three excitation sources(^{55}Fe, ^{109}Cd, ^{241}Am)
  - Safety cover
- Probe laboratory stand with the following:
  - Base for table top use
  - Safety shield over sample
  - Positioning fixtures for standard 30-mm and 40-mm X-ray sample cups
- Interconnecting cable
- RS-232C Serial I/O Interface cable
- Two blank check samples
- Pure element check samples
- Battery charger
- Battery pack
- System carrying/shipping case
- Spectrace 9000 Operating Instructions, application software, and utilities software. The application software is specific to each unit and cannot be interchanged between different units. The software is identified by the serial number of the unit.

5.2.2 Optional Items

- 31-mm diameter sample cups
- XRF polypropylene film, 0.2 mil thick
- Field carrying case
- Peripheral devices such as a printer and IBM compatible Personal Computer (PC)
- Spare probe window assembly
- Spare battery pack, charger, and charger adaptor (required to charge spare battery outside of electronic unit)

See the Spectrace 9000 Accessories Price List for additional options.

For mobile lab or laboratory X-ray sample preparation accessories (such as drying ovens, grinders, sieves, etc.), consult general laboratory equipment suppliers.

5.2.3 Limits and Precautions

The probes should be handled in accordance with the following radiological control practices.

1. The probe should always be in contact with the surface of the material being analyzed, and that material should completely cover the probe opening (aperture) when the sources are exposed. Do not remove a sample or move the probe while the indicators show **SOURCE ON**.

SOURCE ON indicators are:

- the message on the screen "**SOURCE ON**"
- the flashing light at the base of the probe.

2. When the sources are exposed, under no circumstances should the probe be pointed at the operator or surrounding personnel.

3. Do not place any part of the operator's or co-worker's bodies in line of exposure when the

sources are exposed or partially covered.

4. The probe must be covered with the safet y cover or labo ratory safety shield when not in use.

5. Spectrace Instruments must be notifie d immediately of any condition or concer n relative to the probe's structural integrity , source shielding, so urce switching condition, or operability.

6. The appropriate state agency or the Nuclear Regulatory Commission (NRC) office mus t be notified immediately o f any damage to the radioactive source, or any loss or theft of the device (see factory supplied data o n radiological safety).

7. Labels or instructions on the probe(s) mus t not be altered or removed.

8. The user must not attempt to open the probe.

9. The source(s) in the probe must be leak - tested every 6 months as described in th e Spectrace 9000 Operating Instructions. The leak test certificates m ust be kept on file, and a copy must accompany the instrument at all times.

10. The probe laboratory safety shield assembly must be used when the probe is inverted for measuring samples contained in cups.

11. During operation, the probe must be kept a t least 10 feet from computer monitors and any other source of radio frequency (RF). Some monitors have very poor RF shielding an d will affect measurement results.

12. The Spectrace 9000 shou ld not be dropped or exposed to conditions of excessive shock or vibration.

13. The electronic unit should be left o n whenever the battery charger is connected to it. If the electronic unit is shut off with th e battery charger plugged in, the battery ma y be damaged due to overcharging.

Additional precautions include:

1. The probe cable must never be pulled while unplugging the probe. The p robe plug should be grasped at the ribbed metal connector and squeezed and pulled gently while th e connector is unplugged. The connector must never be forced when plugging in th e connector.

2. The handle of t he electronic unit must not be rotated unless the release buttons on eac h side of the handle are depressed.

3. The Spectrace 9000 should not be stored a t an ambient tempera ture below -4 °F or above 110EF.

4. The battery charging unit should only b e used indoors in dry conditions.

5. Battery packs should be changed only in dry conditions.

5.3 Peripheral Devices

The Spectrace 9000 may be used with a wide range of peripheral devices for electronic data capture or printed readout as long as they are compatible with the RS-232 serial I/O protocol. Such devices include terminals, printers, electronic data loggers, personal computers, etc.

5.3.1 Communication Cable Connection

Plug the 25-pin connector of the RS-232 Serial I/O cable into the Spectrace 9000 25-pin connector (the connection just below the display screen on the electronic unit) and the 9-pin connector of the cable into the serial port of the receiving device.

5.3.2 Communication Port Setup

To communicate with an external device, the Spectrace 9000 **MUST** be set at the same baud rate, word length, and parity as the receiving device. The Spectrace 9000 allows you to select various configurations for these parameters in the communication (Comm.) port setup portion of the More submenu (which can be accessed from the main menu).

The default COM setup for application and utilities software is 9600,N,8,1.

5.3.3 User Software

Refer to your PC software manual for details on additional settings that may be required for proper interfacing between the Spectrace 9000 and your particular software.

5.4 Instrument Maintenance

5.4.1 Probe Window

Should the probe window become damaged or punctured, it should be replaced as soon as possible to prevent dust and moisture from entering the probe. Replacement window assemblies can be ordered from Spectrace Instruments. Note the location of the window aperture; it is closer to one end of the window plate. Simply unscrew the old window plate, press any corner of it, and remove it. Stretch the O-ring for 10 seconds, and lay it back in the groove. The O-ring must lie flat in the groove in order for the new window plate to be installed. Install the new window assembly in the same manner as the old. If the surface of the window plat is not flush with the face of the probe, the O-ring has probably come out of the grove. Remove the assembly, and try the same procedure again.

5.4.2 Further Information and Troubleshooting

Refer to the Spectrace 9000 Operating Instructions for additional detailed operational and /or maintenance and troubleshooting instructions. If no solution is found in the manual, contact Spectrace Instruments for assistance.

An instrument log should be maintained to document specific corrective actions taken to alleviate any instrumental problems, or for recording any service that has been performed.

6.0 REAGENTS

Generally, calibration standards are not necessary for site screening and extent of contamination analyses with the Spectrace 9000. Optionally, an application (only the Soil Sample application will be discussed here) can be optimized or verified to be 1:1 proportional to another analytical (reference) method (see Section 9.3 and 10.1). This can be done by analyzing a suitable set of Site-Specific Calibration Standards (SSCS) or Standard Reference Materials (SRMs) and performing a regression analysis on the reference (dependent) and the Spectrace 9000 results (independent) for each element of concern. SCSS and SRMs must be representative of the sample matrix to be analyzed by XRF, for example, National Institute of Standards and Technology (NIST) SRMs 2709, 2710, and 2711 for the soil application. In an application, any element's calibration can be adjusted by entering the desired slope and offset (intercept) in the Adjust Calibration menu. If any element's calibration has been adjusted in an application, "adj" will appear on the results screen. An adjusted element calibration can always be changed back to the initial slope and offset values of 1 and 0, respectively.

7.0 PROCEDURE

7.1 Prerequisites

If the Spectrace 9 000 will be used in a location where AC power outlets are conveniently accessible, connect the battery charger to the electronic unit and plug the charger cord into the outlet. The probe cable must be connec ted before switching on the power. Pluggin g and unplugging this cable with the power on ca n damage the detector.

To connect the battery, set the electronics unit on its face and use a flat blade sc rewdriver to loosen the two one-quarter turn fasteners on the back. Remove th e battery pack. Inside, find the cord with the red ca p covering the three-prong ed plug. Remove the cap and plug it into the bat tery pack. Put the battery pack into the unit and tighten the fasteners.

Apply power to the Spectrace 9000 by pressing th e <ON> button. The electronic unit may not come o n with the battery charger hooked up if the battery ha s been totally draine d. The drained battery may require a 10 minute charge prior to startup. In a few seconds the display shows the version of software. I f necessary, adjust the contrast knob located on th e underside of the front display. This knob can b e turned so far that the display appears blank.

The initial screen displays for about 10 seconds an d then a prompt will ask if the time and date are se t correctly. The date **MUST** be set correctly otherwise serious errors in source-decay compensation ca n result. Additionally, results tables include the tim e and date of analysis. The main menu appears after the time and date screens.

If a "battery low" message appears, recharge o r change the battery before proceeding, or operate th e unit using line voltage.

Allow the Spectrace 9000 to warm up fo r approximately 30 minutes after it has been turned on before performing analysis.

7.1.1 Gain Control

Automatic gain compens ation is a feature of both Soil and Thin Samples applications, which allow s operation of the instrument over a wide range o f ambient temperatures and from one day to anothe r without standardization. To maintain gain contro l compensation, it is necessary to occasionally operate with a minimum acquisition t ime of 50 seconds on the Cd-109 source. If the automatic gain control fails or is out of range, an error message will appear on th e screen. If t he error message continues to appear after repeat analyses, then the Cd-109 measurement tim e should be checked and/or an energy calibration should be performed. If the problem continues, contac t Spectrace Instruments for help.

7.1.2 Setting Data and Spectr um Store/Send Mode

The Set store/send modes option is located in th e More screen which can be accessed from the mai n menu. Data and/or Spec trum storage must be enabled for automatic on-board storing to occur. Sufficien t memory is available to sto re up to 300 sets of analysis results and up to 120 spectra (40 samples since eac h sample has three spectra). When the availabl e memory is full, the respective spectra or result s storage mode is automatically disabled. The spectra or results memory must be cleared (deleted) and th e respective store mode enabled before results and/o r spectra can be stored again.

7.2 General Keys and Menu Software

This section outli nes the general keys and basic menu softwa re. Flow charts which describe the men u structure in detail are located o n pages 4-13 through 4-17 in the Spectrace 9000 Operating Instructions.

7.2.1 The Keyboard

The row of numeric keys under the LCD scree n performs functions defined by labels (a menu) written to the bottom line of the display by the Spectrace 9000 software. As the operator moves through the various menus, the keys are redefined to provide an efficient user interface.

The keypad to the right of the screen is used fo r numeric ent ry. The <Cont/Pause> key (referred to as the <Cont>) is used:

- to enter information as an <Enter> key
- to begin an analysis
- to pause an analysis in progress

The left arrow < 7> key is used to edit entries before pressing <Cont>.

7.2.2 The Measure (Ready) Screen

This main menu selection displays the application name, revision date, measurement time for each source, and accesses other options (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.3 The Choose an Application Screen

This main menu selection lists the applications currently loaded in the unit. Applications are selected and source measurement times may be modified in this screen (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.4 The Review Stored Results Screen

This main menu selection lists the stored results. *Up* and *Down* scroll are used on many screens. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. Stored results may be reviewed, deleted, or downloaded to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.5 The Review Stored Spectra Screen

This main menu selection lists the stored spectra which may be deleted or transmitted to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions). You cannot display spectra under this screen. Spectra may be displayed in the *Examine Spectrum* portion of the More screen (accessed from the main menu) or in the *Examine Spectrum* selection from the Results screen under the *More Options* menu selection.

7.2.6 The More (Other Functions) Screen

This main menu selection lists the following functions:

- *Set clock/calendar*
- *Comm. port setup*
- *Set store/send modes*
- *Application maintenance*
- *Examine spectrum*

7.2.7 The Results Screen

The Results screen is displayed at the end of the analysis. If the automatic *Store Results* mode is enabled, you will be prompted for sample identification (*ID*) before the Results screen is displayed. *Up* or *Down* scrolls the screen to view more results. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. *Send* transmits results to the COM port. *Store* prompts for an ID and then stores results in memory. *Measr* will immediately begin another analysis cycle. *Opts* displays the first of two screens listing special options under the Results screen (the second screen is located under *More Opts* of the first screen. See flow diagrams in Spectrace 9000 Operating Instructions). The most frequently used functions are the *Examine Spectrum* and *Enable/Disable Display Thresholds* located on the second screen of options.

7.3 Preoperational Checks

7.3.1 Energy Calibration Check

An energy calibration should be performed after an instrument is shipped and periodically (approximately 2 weeks) to ensure proper energy calibration. The *Energy Calibration* function is located in the *Options* section of the Measure Screen. You will be prompted to place the safety shield on the probe and then initiate a 600- second analysis that will update the X-ray energy calibration.

The energy calibration check is performed in the field daily and after an energy calibration to verify proper energy calibration. To perform an energy calibration check, place the safety shield on the probe. Select the *Soil Samples* application and measure the safety shield using a minimum acquisition time of 60 seconds for each source. Save the results and spectra for documentation. Select *Opts*, *More Options*, and then *Examine Spectrum*. Examine the spectrum of each source. Locate and record the centroid KeV (using the x12 horizontal magnification) for each of the following peaks:

Source	Peak	Theoretical (KeV)	Specification (KeV)
Cd-109	Pb L-alpha	10.54	± 0.040
	Pb L-beta	12.61	± 0.040
	Pb L-gamma	14.76	± 0.040
	Source line	22.10	± 0.040
Fe-55	S K-alpha	2.31	± 0.020

	Source line	5.89	± 0.020
Am-241	Pb L-alpha	10.54	± 0.050
	Pb L-beta	12.61	± 0.050
	Source line	59.5	± 0.200

Perform an *Energy calibration* (see Spectrace 900 0 Operating Instructions) and then do another energ y calibration check if any of the peaks fail to mee t specification. The e nergy calibration check should be perform ed once at the beginning of the day, after a n energy calibration, after loading an application, an d whenever the instrument exhibits a persistent drift.

7.3.2 Resolution Check

The resolution check examines the detector's ability to resolve X-ray energies. This should be performe d once at the beginning of the day. Select the *Soil Samples* application, and measure a sample of iro n using a minimum acquisition time of 60 seconds fo r the Cd-109 source. Save the results and spectra fo r documentation. Select *Examine spectrum* under th e More Options section of the Results screen. Examine the Cd-109 spectrum. Locate and record th e maximum peak counts (must be >1000 counts)of the iron K-alpha p eak (6.4 KeV) using the x12 horizontal magnification (see Figure 2 , Appendix A). Divide the maximum peak counts by two. Examine the righ t (high energy) side of the peak and record the count s and KeV of the channel with counts less than or equal to one-half the maximum peak count value (channe l B, Figure 2). Examine the left (low energy) side o f the peak and record the counts and KeV of the channel with counts less than or equal to one-half th e maximum peak count value (channel A, Figure 2) . Subtract the left-side KeV from the right-side Ke V (KeV at B - Kev at A, Figure 2). The differenc e should be less than 0.300 KeV. If the unit fails t o meet this specific ation, call Spectrace Instruments for assistance.

7.3.3 Blank (Zero) Sample Check

The blank (Zero) sample check is performed t o monitor the instrument's zero drift in the selecte d application. The blank sample check and the *Aquire Background Data* operation (discussed below) onl y apply to the application currently selected. Thi s should be done once at the beginning of the day, after an energy calibration, after lo ading an application, and whenever the instrument exhibits a persistent drift on a blank or low-level sample.

Mount the probe in t he laboratory stand and select the *Soil Samples* application. Disable the displa y thresholds. This will permit results less than on e standard deviation (STD) to be displayed (eve n negatives). Measure the quartz blank provided wit h the unit (or a "clean" sand sample) using a minimum acquisition time of 60 seconds for each source . Review the results table. All elemental results fo r target elements with atomic number 24 (Cr) an d higher in the periodic table should be within 3 standard deviations of zero ($0 \pm 3 * |STD|$); all non - target element results should be within 5 standar d deviations ($0 \pm 5 * |STD|$). Repeat the measurement if the unit fails to meet these specifications. If severa l elements continue to be significantly out of thes e specifications, check the probe window and the blank sample for contamination or perform the *Acquire background data* operation located in the Measur e (Ready) screen option. Perform the blank (Zero) sample check again. Save the results and spectra for documentation. Enable the display thresholds prior to sample analysis after the blank sample chec k procedure is completed.

7.3.4 Target Element Response Check

The purpose of the target el ement response check is to ensure that the instrument and the selected application are working properly prior to performing sampl e analysis. This check should be performed at th e beginning of the day. Use low, mid, and hig h samples, or standards with known concentrations for some or all of the target elements to be checked . Select a low sample near the quantitation limit of the target elements. Select a mid sample near the sit e action level and a high sample near the maximu m concentration of the target elements expected on site.

These samples should be measured using the sam e source acquisition times that will be used for sample analysis. Save the sample check results and spectr a for documentation.

7.4 Selecting Source Measuring Time

The source measuring time may be modified under the Measure screen. **Zero (seconds) measuring time should never be selected for any source for any application**. Generally, the element detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time . Although counting statistics impr ove as measurement time increases, the practical limit for typical applications is 600 to 80 0

seconds. The elements are grouped together according to the radioisotope used for their excitation with typical minimum detection limits shown in Sections 7.4.2. and 7.4.3.

Automatic gain compensation is a feature of both the Soil and Thin Samples applications which allows operation of the instrument over a wide range of ambient temperatures and from one day to another without standardization. To maintain this gain control compensation, it is necessary to occasionally operate with a minimum acquisition time of 50 seconds on the Cd-109 source.

The *Real/live* option toggles between real time (true clock time) and live time (total time the instrument is counting). The latter adds time to the analysis to correct for the time the system is busy processing pulses.

7.4.1 Minimum Source Measuring Times

A minimum measuring time (real or live) of 15 seconds for the Fe-55 source, 30 seconds for the Cd-109 source, and 10 seconds for the Am-241 source is recommended when using the Soil Samples application. Measuring times for a source that excites a target element can be increased if lower detection limits are required.

When using the Thin Samples application, the measuring time for any source may be reduced to 10 seconds if the source does not excite a target element since this application does not correct for interelement effects. If a source excites a target element, a minimum measuring time (real or live) of 60 seconds for the Fe-55 source, 60 seconds for the Cd-109 source, and 120 seconds for the Am-241 source is recommended.

A minimum of 60 seconds is recommended for the Cd-109 source when using the PbK in Paint application.

7.4.2 Typical Minimum Detection Limits (MDLs) for the Soil Samples Application

For source measuring times of 60 seconds, typical element MDLs (in milligram per kilogram, mg/kg) for the Soil Samples application are:

Source	Element	MDL (mg/kg)
Fe-55	Potassium (K)	325
	Calcium (Ca)	150
	Titanium (Ti)	110
	Chromium (CrLo)	180
Cd-109	Chromium (CrHi)	525
	Manganese (Mn)	
410		
	Iron (Fe)	225
	Cobalt (Co)	205
	Nickel (Ni)	125
	Copper (Cu)	90
	Zinc (Zn)	70
	Mercury (Hg)	60
	Arsenic (As)	50
	Selenium (Se)	35
	Lead (Pb)	30
	Rubidium (Rb)	10
	Strontium (Sr)	10
	Zirconium (Zr)	10
	Molybdenum (Mo)	10
Am-241	Cadmium (Cd)	
180		
	Tin (Sn)	
100		
	Antimony (Sb)	65
	Barium (Ba)	20

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the matrix of the soil standard used to calculate MDLs, age of sources, moisture content, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Additionally, more elements may be added to the Soil Samples application. Contact Spectrace Instruments for information about modifications to applications.

7.4.3 Typical Minimum Detection Limits (MDLs) for the Thin Samples Application

For source measuring times of 200 seconds for the Fe-55 and Cd-109 sources, and 800 seconds for the Am-241 source, typical element MDLs (in microgram per square centimeter, $\mu g/cm^2$) for the Thin Samples application are:

Source	Element	MDL (µg/cm²)
Fe-55	Potassium (K)	0.40
	Calcium (Ca)	0.20
	Titanium (Ti)	0.15
	Chromium (CrLo)	0.40
Cd-109	Chromium (CrHi)	0.90
	Manganese (Mn)	0.65
	Iron (Fe)	0.65
	Cobalt (Co)	0.50
	Nickel (Ni)	0.30
	Copper (Cu)	0.65
	Zinc (Zn)	0.40
	Mercury (Hg)	0.45
	Arsenic (As)	0.40
	Selenium (Se)	0.15
	Lead (Pb)	0.50
	Rubidium (Rb)	0.10
	Strontium (Sr)	0.10
	Zirconium (Zr)	0.15
	Molybdenum (Mo)	0.10
Am-241	Cadmium (Cd)	2.5
	Tin (Sn)	2.5
	Antimony (Sb)	1.5
	Barium (Ba)	0.70

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the thin sample standard used to calculate MDLs, age of sources, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Use of thick filters or filters with high background or contamination will result in higher MDLs and require a background subtraction. Additionally, more elements may be added to the Thin Samples application. Contact Spectrace Instruments for information about modifications to applications.

7.5 Sample Handling and Presentation

When making XRF measurements, be sure to maintain constant measurement geometry in order to minimize variations in analysis results. Document any anomalies in measurement geometry, sample surface morphology, moisture content, sample grain size, and matrix (see Section 4.0).

7.5.1 Soil Samples

Soil samples may be analyzed either in-situ or in prepared X-ray sample cups. The Soil Samples application assumes the sample to be infinitely thick. For in-situ measurements this is almost always the case. However, for sample cup measurements it is advisable to fill the cup nearly full and tap it on the bench to compact the soil. This ensures that the sample is as uniformly thick as possible from analysis to analysis. The Spectrace 9000 laboratory stand and safety shield should be used when analyzing sample cups.

An area for in-situ analysis should be prepared by removing large rocks and debris. The soil surface should be rendered flat and compact prior to analysis. The Spectrace 9000 probe should be held firmly on the ground to maximize instrument contact with the ground. The probe should not be moved during analysis. Analysis of water saturated soils should be avoided. A thin layer of 0.2-mil polypropylene XRF film may be mounted on the surface probe to minimize contamination. Use of varying thicknesses of plastic (bags) have been shown to interfere in the light element (low atomic number) measurement and may affect the FP calibration of the other element concentrations.[2] Additionally, plastic may contain significant levels of target element contamination.

Course-grained soil conditions or nuggets of contaminated material may preclude a truly representative sample and adversely affect the analysis results (typically by under reporting the target element). Such samples should be prepared before analysis. Preparation consistency is important to minimize variation in analytical results.

This application is specifically designed for soil with the assumption that the balance of the material is silica. If samples with a much lighter (lower atomic number) balance are analyzed, the results will typically be elevated by a factor of two to four. Contact Spectrace Instruments for help in analysis of different matrices.

7.5.2 Thin (Filter) Samples

The Thin Samples application is for analysis of thin samples such as filters or wipes. The detection limits are affected by the thickness of the substrate. Best results are obtained on the thinnest substrates.

Always use the probe safety cover when measuring thin samples. This is not only for user safety, but also ensures a controlled background environment and provides a reference signal for the automatic gain control. Probe safety covers should never be interchanged between instruments.

Filters and wipes should be prescreened before use to establish background and contamination levels. Care should be used to prevent zinc oxide contamination from disposable gloves. Small 37-mm filters can be mounted between two layers of 0.2-mil thick polypropylene XRF film on 40-mm XRF cups for analysis. Larger filters can be placed on the probe with a sheet of 0.2-mil thick polypropylene XRF film between the filter and probe to prevent the window from being contaminated. Then the probe safety cover may be placed over the filter prior to analysis. Filters should be presented loaded side down and wrinkle free.

7.5.3 Lead in Paint

The area selected for analysis should be smooth, representative and free of surface dirt. The Spectrace 9000 probe should be held firmly on the surface to maximize instrument contact. The probe should not be moved during analysis.

When used for specimen application (e.g., on paint chips or nonbacked films) remember to use the probe safety cover. In the PbK Application, you should also position a thick neutral sample, such as the quartz disk (blank), behind the specimen before closing the safety lid. Otherwise, the PbK X-rays excited in the safety cover will be sensed by the detector. In this application, do not perform the *Acquire background data* option from the list of options under the Ready screen.

8.0 CALCULATIONS

The Spectrace 9000 is a direct readout instrument that does not require any calculations.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

9.1 Precision

The precision of the method is monitored by reading a low- or mid-target element concentration sample (or SSCS or SRMs selected as described in Sections 6.0) at the start and end of sample analysis and after approximately every tenth sample. (A daily total of seven measurements is recommended.) Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action. Therefore, selection of a sample with a target element concentration at or near the site action level or level of concern is recommended. The sample is analyzed by the instrument for the normal field analysis time, and the results are recorded. The standard deviation for each target element is calculated. The relative standard deviation (RSD) of the sample mean can be used to calculate precision. The RSD should be within ± 20 percent for the data to be considered adequately precise.[3]

9.1.1 The Method Detection Limit (MDL) and Method Quantitation Limit (MQL)

The MDL and MQL may be calculated from the measurement of either a low or blank sample, (or a SSCS or SRMs selected as described in section 6.0), at the start and end of sample analysis, and after approximately every tenth sample (a daily total of seven measurements is recommended). Alternatively, the quartz blank or "clean" sand may be used if a blank soil or sediment sample is unavailable.

Disable the display thresholds. This will permit results less then one standard deviation (STD) to be displayed (even negatives). Measure the sample using the same application and measuring time used for the samples. Enable the display thresholds prior to analyzing the next sample.

The sample standard deviation of the mean for each target element is calculated. If the standard deviation has a fractional component, round up to the next whole number prior to calculating the MDL and MQL.

The definition of the MDL is three times the calculated standard deviation value.

The definition of the MQL is 10 times the calculated standard deviation value.

9.2 Reporting Results

All raw XRF data should be reported including th e individual result s of multiple analyses of samples and sampling points. The average an d concentration range of each multiple analysis should also be reported.

A "repo rted" value for each analysis or average o f multiple analyses should be process ed in the following manner.

1. Round the value to the same degree o f significance contained in the SSCS or SRM sample assay values (usually two) if th e element's calibration has been adjusted (se e Section 6.0). Round to 2 significant figures for sample results. DO NOT round result s for standards used to determine MDL o r RSD values (use raw data).

2. Report all values less than the MDL as no t detected (ND).

3. Flag and note all values greater than or equal to the MDL and less than the MQL (usually with a "J" next to the reported value).

4. Report all values equal to or greater than the MQL and within the linear calibration range (if the element's calibration has been adjusted [see section 6.0]).

5. Flag and note all values above the linea r calibration range (greater than the highes t SSCS used in the calibration adjustmen t procedure) if SSCS were used and th e calibration was adjusted.

9.3 Accuracy

Accuracy, relative to a specific digestion method and elemental analysis procedure, is determined b y submitting an XRF analyzed sample (prepared sample cups may be submitted) for AA or ICP analysis at a laboratory.

The on-site analysis of soils by XRF instrumentation should be considered a screening effort only (QA 1 data). Data derived from the instrument should b e used with discretion. Confirmatory analyses on a subset of the screening samples (minimum 10 percent) can be used to determine if the XRF data meets QA2 data objectives. The confirmation samples shoul d ideally be selected randomly from the sample set and include a number of samples at or near the critica l level. The results of the metal analysis (dependent) and the XRF analysis (independent) are evaluate d with a regression analys is. The correlation factor (R^2) should be 0.7 or greater. [3]

XRF results may be multiplied by the slope prior t o substitution for metal analysis results in contouring , kriging programs, or removal volume estimates . Correcting the XRF results based on confirmator y analyses should only be undertaken after carefu l consideration. It must be understood that th e confirmatory analysis (AA or ICP) is an estimate o f the concentration of metal contamination and i s dependent upon the specific instrumentation an d sampling methodology used. Since XRF is a tota l elemental technique, any comparison with refere e results must account for the possibility of variabl e extraction, dependent upon the digestion method used and its ability to disso lve the waste or mineral form in question.

9.3.1 Matrix Considerations

Other types of QA/QC verification should includ e verification that the instrument calibration i s appropriate for the specific site to be assessed. This includes verification of potential multiple soil matrix types that may exist at a site. Matrix difference s which affect the XRF measurement include larg e variations in calcium content, which may b e encountered when going from siliceous to calcareous soils, as well as large variations in iron content.

10.0 DATA VALIDATION

10.1 Confirmation Samples

Confirmation samples are recommended at a minimum rate of 10 percent and are required if QA2 data objectives have been established for site activities.[3] Ideally, the sample cup that was analyzed by XRF should be the same sample that is submitted for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a 6-inch by 6-inch area for both an XRF measurement and confirmation analysis.

The XRF and metals results are analyzed with a regression analysis using a statistical program such as SAS® or Statgraphics® with the intercept calculated in the regression. The correlation factor between XRF and AA/ICP data must be 0.7 or greater for QA2 data objectives.[3]

10.2 Recording Results

Record all results and monitoring activities in a laboratory or field notebook. Alternatively, record results electronically on a hard drive or floppy disk.

10.3 Downloading Stored Results and Spectra

Results (analytical reports) and spectra which have been stored in the Spectrace 9000 internal memory should be downloaded and captured in disk files on a PC (see section 5). Spectrace Instruments provides software for this purpose. Additionally, they provide software to prepare results or spectra for importing into a spreadsheet. Refer to the instructions provided with the programs for details on their operation.

Alternatively, other software with terminal data logging capabilities may be used to capture results and spectra to disk files.

After capturing results to a file, print a copy and save both the disk files and the printout for future reference and documentation purposes.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, corporate and/or any other applicable health and safety practices.

12.0 REFERENCES

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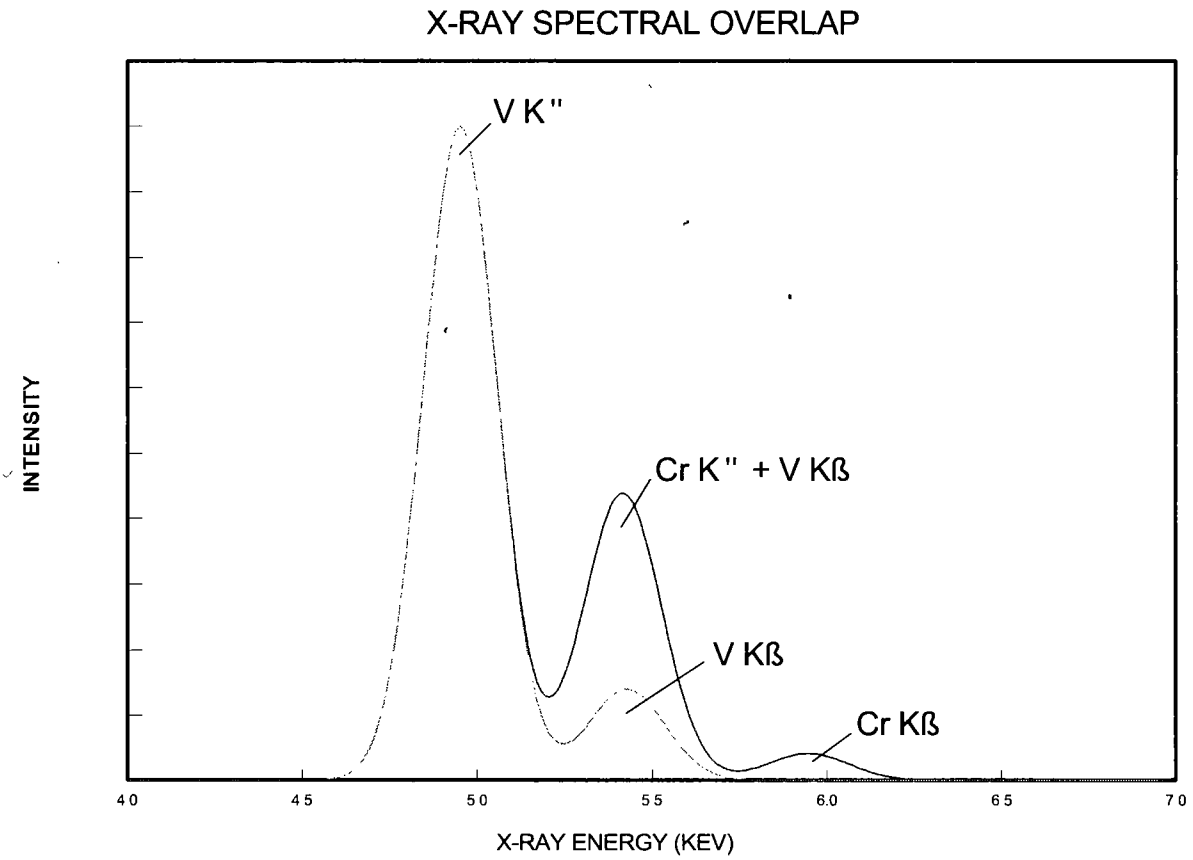
APPENDIX A

Figures

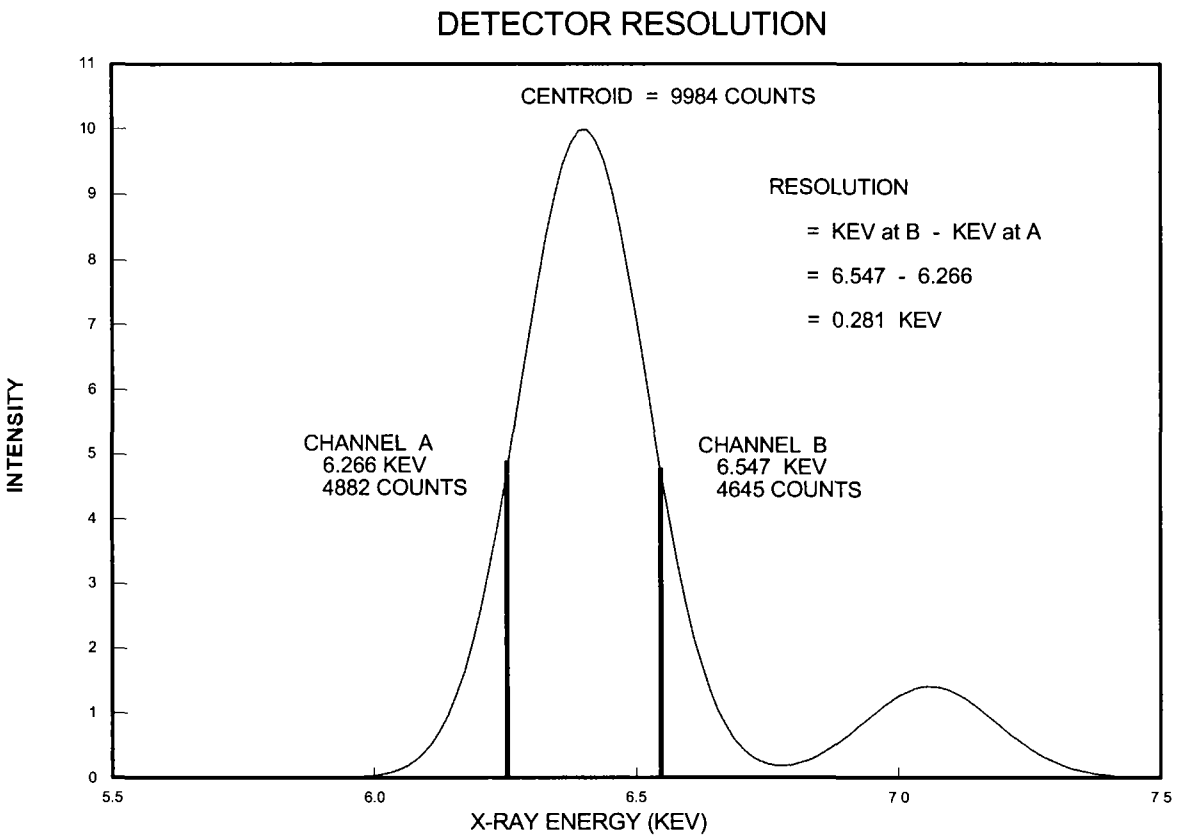
FIGURE 1. X-Ray Spectral Plot Showing Overlap of Vanadium K $_{\beta}$ X-Rays in the Chromium K $_{\alpha}$ Measurement Region.



APPENDIX A (CON'T)

Figures

FIGURE 2. Iron X-Ray Spectrum Illusrating Detector Resolution Measurement



PCP IMMUNOASSAY USER'S GUIDE

Pentachlorophenol

• Intended Use

For detection of pentachlorophenol in water (groundwater, surface water, well water). For soil, crop, and food use refer to specific application bulletins.

For use as SW-846 Method 4010 "Screening for Pentachlorophenol by Immunoassay" see Strategic Diagnostics Inc. Technical Bulletin #T00094.

• Principle

The Pentachlorophenol RaPID Assay® applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of pentachlorophenol. The sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles with antibodies specific to pentachlorophenol attached. Both the pentachlorophenol (which may be in the sample) and the enzyme labeled pentachlorophenol (the enzyme conjugate) compete for antibody binding sites on the magnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (with pentachlorophenol and labeled pentachlorophenol analog bound to the antibodies on the particles, in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with Washing Buffer.

The presence of pentachlorophenol is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5'-tetramethylbenzidine). The enzyme-labeled pentachlorophenol analog bound to the pentachlorophenol antibody catalyzes the conversion of the substrate/ chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Since the labeled pentachlorophenol (conjugate) was in competition with the unlabeled pentachlorophenol (sample) for the antibody sites, **the color developed is inversely proportional to the concentration of pentachlorophenol in the sample**.

• Reagents

1 Pentachlorophenol Antibody Coupled Paramagnetic Particles
The pentachlorophenol antibody (rabbit anti-pentachlorophenol) is covalently bound to paramagnetic particles, which are suspended in buffered saline with preservative and stabilizers.
30 test kit: one 20 mL vial
100 test kit: one 65 mL vial

2. Pentachlorophenol Enzyme Conjugate
The horseradish peroxidase (HRP) labeled pentachlorophenol analog is diluted in buffered saline with preservative and stabilizers.
30 test kit: one 10 mL vial
100 test kit: one 35 mL vial

3 Pentachlorophenol Standards
Three concentrations (0.1, 2.0, 10.0 ppb) of pentachlorophenol standards in buffered saline with preservative and stabilizers are supplied. Each vial contains 2.0 mL.

4 Control
A concentration (approximately 1 ppb) of pentachlorophenol in buffered saline with preservative and stabilizers. A 2.0 mL volume is supplied in one vial.

5 Diluent/Zero Standard
Buffered saline containing preservative and stabilizers without any detectable pentachlorophenol.
30 test kit: one 10 mL vial
100 test kit: one 35 mL vial

6. Color Solution
A solution of hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine in an organic base.
30 test kit: one 20 mL vial
100 test kit: one 65 mL vial

7. Stopping Solution
A solution of sulfuric acid (0.5%).
30 test kit: one 20 mL vial
100 test kit: one 60 mL vial

8. Washing Buffer
Buffered saline containing preservatives and stabilizers.
30 test kit: one 70 mL vial
100 test kit. one 250 mL vial

9. Test Tubes
Polystyrene tubes (36) are packaged in a box.
30 test kit: one 36 tube box
100 test kit· three 36 tube boxes

• Reagent Storage and Stability

Store all reagents at 2-8°C. Do not freeze. Reagents may be used until the expiration date on the box. *The test tubes require no special storage condition and may be stored separately from the reagents to conserve refrigerator space*

Consult state, local and federal regulations for proper disposal of all reagents.

• Materials Required but Not Provided

In addition to the reagents provided, the following items are essential for the performance of the test:

Pipets* Precision pipets capable of delivering 200, 250 and 500 uL and a 1.0 mL repeating pipet.

Vortex Mixer* Thermolyne Maxi Mix, Scientific Industries Vortex Genie, or equivalent

Magnetic Separation Rack*

RPA-I™ RaPID Analyzer* or equivalent photometer capable of readings at 450 nm

* These items are available from Strategic Diagnostics Inc.

• Sample Information

This procedure is recommended for use with water samples. Other samples may require modifications to the procedure and should be thoroughly validated.

Samples containing gross particulate matter should be filtered (e.g 0 2 um Anotop™ 25 Plus, Whatman, Inc.) to remove particles.

Samples which have been preserved with monochloroacetic acid or other acids, should be neutralized with strong base e.g. 6N NaOH, prior to assay.

If the pentachlorophenol concentration of a sample exceeds 10 ppb, the sample is subject to repeat testing using a diluted sample. A ten-fold or greater dilution of the sample is recommended with an appropriate amount of Diluent/Zero Standard or Sampel Diluent. For example, in a separate test tube make a ten-fold dilution by adding 100 uL of the sample to 900 uL of Diluent/Zero Standard Mix thoroughly before assaying Perform the assay according to the Assay Procedure and obtain final results by multiplying the value obtain by the dilution factor e.g. 10.

The presence of the following substances up to 250 ppm were found to have no significant effect on Pentachlorophenol RaPID Assay results: calcium, copper, manganese, magnesium, mercury, nickel, nitrate, phosphate, sulfite, thiosulfate and zinc. In addition, sodium chloride up to 0 65 M, sulfate to 10,000 ppm, iron to 50 ppm and humic acid to 10 ppm, showed no significant effect on results.

• Reagent Preparation

All reagents must be allowed to come to room temperature and the antibody coupled paramagnetic particles should be mixed thoroughly before use.

• Procedural Notes and Precautions

As with all immunoassays, a consistent technique is the key to optimal performance. To obtain the greatest precision, be sure to treat each tube in an identical manner.

Add reagents directly to the bottom of the tube while **avoiding contact between the reagents and the pipet tip.** This will help assure consistent quantities of reagent in the test mixture.

Avoid cross-contaminations and carryover of reagents by using clean pipets for each sample addition and by avoiding contact between reagent droplets on the tubes and pipet tips.

Avoid foam formation during vortexing.

The magnetic separation rack consists of two parts: an upper rack which will securely hold the test tubes and a lower separator which contains the magnets used to attract the antibody coupled paramagnetic particles. During incubations the upper rack is removed from the lower separator so that the paramagnetic particles remain suspended during the incubation. **For separation steps, the rack and the separator are combined to pull the paramagnetic particles to the sides of the tubes.**

To obtain optimum assay precision, it is important to perform the separation steps carefully and consistently. Decant the rack by slowly inverting away from the operator using a smooth turning action so the liquid flows consistently along only one side of the test tube. While still inverted, place the rack on an absorbent pad and allow to drain. Lifting the rack and replacing gently onto the pad several times will ensure complete removal of the liquid from the rim of the tube (technique is demonstrated on training video, available from Strategic Diagnostics Inc.).

Mix the antibody coupled paramagnetic particles just prior to pipetting.

Do not use any reagents beyond their stated shelf life.

Avoid contact of Stopping Solution (sulfuric acid) with skin and mucous membranes. If this reagent comes in contact with skin, wash with water.

• Limitations

The Pentachlorophenol RaPID Assay will detect pentachlorophenol and related compounds to different degrees. Refer to specificity table for data on several of the organochlorines. The Pentachlorophenol RaPID Assay kit provides screening results. As with any analytical technique (GC, HPLC, etc...) positive results requiring some action should be confirmed by an alternative method.

The total time required for pipetting the magnetic particles should be kept to two (2) minutes or less, therefore the total number of tubes that can be assayed in a run should be adjusted accordingly.

• Quality Control

A control solution at approximately 1 ppb of pentachlorophenol is provided with the Pentachlorophenol RaPID Assay kit It is recommended that it be included in every run and treated in the same manner as unknown samples. Acceptable limits should be established by each laboratory.

• Assay Procedure

Read Reagent Preparation, Procedural Notes and Precautions before proceeding.

1. Label test tubes for standards, control, and samples.

Tube Number	Contents of Tube
1,2	Diluent/Zero Standard, 0 ppb
3,4	Standard 1, 0.1 ppb
5,6	Standard 2, 2.0 ppb
7,8	Standard 3, 10 0 ppb
9	Control
10	Sample 1
11	Sample 2
12	Sample 3

2. Add 200 uL of the appropriate standard, control, or sample.
3. Add 250 uL of Pentachlorophenol Enzyme Conjugate to each tube.
4. Mix the Pentachlorophenol Antibody Coupled Paramagnetic Particles thoroughly and add 500 uL to each tube.
5 Vortex for 1 to 2 seconds minimizing foaming.
6. Incubate for 30 minutes at room temperature.
7 Separate in the Magnetic Separation Rack for **two (2) minutes**
8. Decant and **gently** blot all tubes briefly in a consistent manner.
9. Add 1 mL of Washing Buffer to each tube and allow them to remain in the magnetic separation unit for **two (2) minutes**.
10. Decant and **gently** blot all tubes briefly in a consistent manner.
11 Repeat Steps 9 and 10 an additional time.
12. Remove the rack from the separator and add 500 uL of Color Solution to each tube.
13. Vortex for 1 to 2 seconds minimizing foaming.
14. Incubate for 20 minutes at room temperature.
15. Add 500 uL of Stopping Solution to each tube.
16. Add 1 mL Washing Buffer to a clean test tube. Use as blank in Step 17.
17. Read results at 450 nm within 15 minutes after adding the Stopping Solution.

• Results

Manual Calculations

1. Calculate the mean absorbance value for each of the standards.
2. Calculate the %B/Bo for each standard by dividing the mean absorbance value for the standard by the mean absorbance value for the Diluent/Zero Standard.
3. Construct a standard curve by plotting the %B/Bo for each standard on vertical logit (Y) axis versus the corresponding pentachlorophenol concentration on horizontal logarithmic (X) axis on the graph paper provided.
4. %B/Bo for controls and samples will then yield levels in ppb of pentachlorophenol by interpolation using the standard curve.

(Contact SDI for detailed application information on specific photometers)

RPA-I RaPID Analyzer

Using the RPA-I RaPID Analyzer, calibration curves can be automatically calculated and stored. Refer to the RPA-I operating manual for detailed instructions. To obtain results from the Pentachlorophenol RaPID Assay on the RPA-I the following parameter settings are recommended:

Data Reduct	:	Lin. Regression
Xformation	:	Ln/LogitB
Read Mode	:	Absorbance
Wavelength	:	450 nm
Units	:	PPB
# Rgt Blk	:	0
Calibrators:		
# of Cals	:	4
# of Reps	:	2

Concentrations:

#1:	0 00	PPB
#2:	0.10	PPB
#3:	2.00	PPB
#4:	10.00	PPB

Range	:	0.06 - 10.00
Correlation	:	0.990
Rep. %CV	:	10%

• Expected Results

No interferences were observed in a study conducted on approxdimately 400 water samples from locations across the U.S. using the Pentachlorophenol RaPID Assay. The Pentachlorophenol RaPID Assay was shown to correlate well against EPA Method 625 (GC/MS) in a study with 20 water samples (r = 0 980)

• Performance Data

Precision

The following results were obtained:

Control	1	2	3	4
Replicates	5	5	5	5
Days	5	5	5	5
n	25	25	25	25
Mean (ppb)	0.51	1 67	3.16	8 63
% CV (within assay)	12.5	8.8	7.7	6.7
% CV (between assay)	11 4	8.6	1 8	3.2

Sensitivity

The Pentachlorophenol RaPID Assay has an estimated minimum detectable concentration, based on a 90% B/Bo of 60 ppt.

Recovery

Four (4) samples, including a municipal water source, drinking water from a local well, and samples from a local pond and a small creek were spiked with various levels of pentachlorophenol and then assayed using the Pentachlorophenol RaPID Assay. The following results were obtained:

Amount of Pentachlorophenol Added (ppb)	Recovery Mean (ppb)	S D. (ppb)	%
0.50	0.49	0.09	98
1.50	1.63	0.17	108
3 00	3 34	0.29	111
8 00	8 43	0 71	105
Average			105

Specificity

The cross-reactivity of the Pentachlorophenol RaPID Assay for various organochlorine compounds can be expressed as the least detectable dose (LDD) which is estimated at 90% B/Bo, or as the dose required to displace 50% (50% B/Bo).

Compound	LDD (ppb)	50% B/Bo (ppb)
Pentachlorophenol	0.06	2.20
2,3,5,6-Tetrachlorophenol	0 21	4 06
2,3,4,6-Tetrachlorophenol	0.91	14.6
2,3,5-Trichlorophenol	1 52	119
2,3,6-Trichlorophenol	2.44	62 9
Tetrachlorohydroquinone	8.70	148
2,4,6-Trichlorophenol	15.1	463
2,4,5-Trichlorophenol	21.5	574
2,3,4-Trichlorophenol	53.2	1730
2,5-Dichlorophenol	62.9	7830
2,6-Dichlorophenol	266	5990
2,3-Dichlorophenol	611	>10,000
2,4-Dichlorophenol	887	>10,000
3,5-Dichlorophenol	1670	>10,000
Hexachlorobenzene	1560	>10,000
Hexachlorocyclohexane	5790	>10,000

The following compounds demonstrated no reactivity in the Pentachlorophenol RaPID Assay at concentrations up to 10 ppm: alachlor, aldicarb, benomyl, butachlor, butylate, captan, carbaryl, carbendazim, carbofuran, 4-chlorophenol, 3,4-dichlorophenol, chlorothalonil, 2,4-D, 1,3-dichloropropene, dinoseb, MCPA, metalaxyl, metolachlor, metribuzin, pentachlorobenzene, pentachloronitrobenzene, picloram, propachlor, terbufos, thiabendazole, and thiophanate-methyl.

• Assistance

For ordering or technical assistance contact:

Strategic Diagnostics Inc.
111 Pencader Drive
Newark, Delaware 19702-3322 USA
Phone(800)544-8881 Fax(302)456-6782
www.sdix.com
techservice@sdix com

• Availability

Strategic Diagnostics Inc.
- Pentachlorophenol RaPID Assay
  - 30 Test Kit
  - 100 Test Kit
- Pentachlorophenol Proficiency Samples
- Pentachlorophenol Sample Diluent
- Soil Collection Kit
- PCP Sample Extraction Kit

Z00134 R020498

PAH RAPID ASSAY USERS GUIDE

STRATEGIC DIAGNOSTICS INC.

RaPID Assay® PAH Test Kit
A00156/A00157

Intended Use

The RaPID Assay® PAH (polyaromatic hydrocarbons) Test Kit can be used as a quantitative, semi-quantitative or qualitative enzyme immunoassay (EIA) for the analysis of PAH in water (groundwater, surface water, well water). For soil application please refer to specific procedure. For applications in other matrices please contact our Technical Service department or refer to the soil application procedure provided. The RaPID Assay® PAH Test Kit allows reliable and rapid screening for PAH (measured and reported as phenanthrene) and related compounds, with quantitation between 2.66 and 66.5 (as phenanthrene). The minimum detection level of the kit is 0.93 (as phenanthrene) in water.

Test Principles

The PAH RaPID Assay® kit applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of PAH and related compounds. The sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles with antibodies specific to PAH attached. Both the PAH (which may be in the sample) and the enzyme labeled PAH (the enzyme conjugate) compete for antibody binding sites on the magnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (with PAH and labeled PAH analog bound to the antibodies on the particles, in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with Washing Solution.

The presence of PAH is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5' – tetramethylbenzidine). The enzyme labeled PAH analog bound to the PAH antibody catalyzes the conversion of the substrate/chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Since the labeled PAH (conjugate) was in competition with the unlabeled PAH (sample) for the antibody sites, the color developed is inversely proportional to the concentration of PAH in the sample.

NOTE: Color development is inversely proportional to the PAH concentration.

Darker color = lower concentration
Lighter color = higher concentration

The determination of the PAH level in an unknown sample is interpreted relative to the standard curve generated from kit standards after reading with a spectrophotometer.

Performance Characteristics

The PAH RaPID Assay® will detect PAH and related compounds to different degrees. Refer to the table below for data on several of these compounds. The PAH RaPID Assay® kit provides screening results. As with any analytical technique (GC, HPLC, etc.) positive results requiring some action should be confirmed by an alternative method.

The PAH RaPID Assay® immunoassay test does not differentiate between PAH and other related compounds. The table below shows compounds at the method detection limit (MDL) which is the lowest concentration of the compound that can be picked up in the assay in a water matrix. The limit of quantitation (LOQ) is an approximate concentration in water required to yield a positive result at the lowest standard. **This is the lowest concentration of the compound that can be quantified in the assay in water**. The IC50 is the concentration required to inhibit one half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

Compound	MDL (ppb)	LOQ (ppb)	IC50 (ppb)
Phenanthrene	0.93	2.66	21.9
Fluoranthene	0.43	0.76	6.25

Benzo(a)pyrene	0.67	1.12	9.18
Pyrene	0.27	1.24	10.24
Chrysene	0.53	1.26	10.4
Anthracene	0.72	1.77	14.6
Indeno(1,2,3-c,d)pyrene	1.04	4.4	36.2
Benzo(a)anthracene	1.02	4.6	37.8
Fluorene	2.19	5.7	46.8
Benzo(b) fluoranthene	1.21	8.77	72.1
Acenaphthylene	13.3	72.3	595
Benzo(k) fluoranthene	1.02	84.7	697
Acenaphthalene	17.2	111.1	915
Benzo(g,h,i) perylene	19.6	>162	>1330
Naphthalene	86.5	>162	>1330
Dibenzo(a,h) anthracene	34.2	>162	>1330
Heating Fuel	17.02	47.2	388.4
JP-5	452.2	1011.3	8326
JP-4	811.3	>1615	>13300
Gasoline	1330	>1615	>13300
Kerosene	1662.5	>1615	>13300
Jet A Fuel	>13300	>13300	>13300

The presence of the following substances up to 250 ppm were found to have no significant effect on PAH RaPID Assay® results: calcium, copper, iron, manganese, magnesium, mercury, nickel, nitrate, phosphate and zinc. In addition, sodium chloride up to 1.0M, sulfate to 10,000 ppm, sulfite and thiosulfate to 100 ppm, showed no significant effect on results.

The Total PAH (sum of 16 PAH compounds) of the indicated contaminant types in soil samples are expressed below, at each of the three kit calibrator (standards) levels, in units comparable to results from GC Method 8270 or HPLC Method 8310.

PAH RaPID Assay®
Total PAH in Water (in ppb)

Contaminant	S1 Equivalent	S2 Equivalent	S3 Equivalent
Creosote	0.532	4	13.3
Coal Tar Oil	0.532	2.66	13.3
Diesel	0.133	1.33	5.32
Turbine Jet Fuel	2	10.64	53.2
Fuel Oil #1	2	10.64	53.2
Fuel Oil #2	0.133	1.33	6.65
Fuel Oil #4	0.133	0.665	3.33
Fuel Oil #5,6	0.133	0.4	1.33

Precautions

- Training is strongly recommended prior to using the RaPID Assay® test system. Contact Strategic Diagnostics for additional information.
- Treat PAH, solutions that contain PAH, and potentially contaminated samples as hazardous materials.
- Use gloves, proper protective clothing, and methods to contain and handle hazardous material where appropriate.
- Reagents must be added in a consistent manner to the entire rack. A consistent technique is the key to optimal performance. Be sure to treat each tube in an identical manner.
- Water samples should be at a neutral pH prior to analysis. Samples containing gross particulate should be filtered (e.g. 0.2 um Anotop™ 25 Plus, Whatman, Inc.) to remove particles.
- Store all test kit components at 2°C to 8°C (36°F to 46°F). Storage at ambient temperature (18°C to 27°C or 64°F to 81°F) on the day of use is acceptable. *Test tubes require no special storage and may be stored separately to conserve refrigerator space.*
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test. This typically requires at least 1 hour to warm from recommended storage conditions.
- Do not freeze test kit components or expose them to temperatures above 100°F (39°C).
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Do not mix reagents from kits of different lot numbers.
- Use approved methodologies to confirm any positive results.

- Do not under any circumstances attempt to disassemble the base of the magnetic rack. Magnets will be violently attracted to each other.
- Adequate sample number and distribution are the responsibility of the analyst.
- The photometer provided in the accessory kit requires electricity and comes with a 110V adapter. Adapters for 220V are available. Do not attempt to operate with a car adaptor.
- Do not expose color solution to direct sunlight.
- Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.
- Tightly recap the standard vials when not in use to prevent evaporative loss.

Materials Provided

- Antibody Coupled Paramagnetic Particles in buffered saline containing preservative and stabilizers.

  30 test kit: one 20 mL vial
  100 test kit: one 65 mL vial

- Lyophilized Enzyme Conjugate

  1 vial

- Enzyme Conjugate Diluent

  30 test kit: one 10 mL vial (minimum)
  100 test kit: one 35 mL vial (minimum)

- Standards

  Three concentrations (2.0, 10.0, 50.0 ppb) of Phenanthrene standards (as phenanthrene analog) in buffered saline containing preservative and stabilizers are supplied. Each vial contains 4 mL.

- Control

  A concentration (approximately 25 ppb) of Phenanthrene (as phenanthrene analog) in buffered saline containing preservative and stabilizers. A 4 mL volume is supplied in one vial.

- Diluent/Zero Standard

  Buffered saline containing preservative and stabilizers without any detectable PAH.

  30 test kit: one 10 mL vial
  100 test kit: one 35 mL vial

- Color Solution containing hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine in an organic base.

  30 test kit: one 20 mL vial
  100 test kit: one 65 mL vial

- Stop Solution containing a solution of 2M sulfuric acid.

  30 test kit: one 20 mL vial
  100 test kit: one 60 mL vial

- Washing Solution containing preserved deionized water with detergent.

  30 test kit: one 70 mL vial
  100 test kit: one 250 mL vial

- Polystyrene test tubes

  30 test kit: one 36 tube box
  100 test kit: three 36 tube boxes

- User's Guide

Materials Required and Ordered Separately

See "Ordering Information" for the appropriate catalogue numbers.

Rapid Assay® Accessory Kit

Accessory equipment may be rented or purchased from Strategic Diagnostics. See "Ordering Information" for the appropriate catalogue numbers.

The accessory kit contains the following items:

- Adjustable Volume Pipet
- Eppendorf™ Repeater® Pipettor
- Electronic timer
- Portable balance capable of weighing 10 g (for soil samples)
- Vortex mixer
- Magnetic separation rack

- RPA-I RaPID Analyzer (or equivalent spectrophotometer capable of reading 450 nm in a 1 mL sample size).

Other Items

- 12.5 mL Combitips® for the Repeater pipettor - for 0.25 mL to 1.25 mL dispensing volumes (5)
- Pipet tips for adjustable volume pipet (100-1000 uL)

NOTE: Order replacement Combitips® and pipet tips separately. See the "Ordering Information" section.

Materials Required but Not Provided

- Methanol (HPLC grade or equivalent) for water samples
- Protective clothing (e.g., latex gloves)
- Absorbent paper for blotting test tubes
- Liquid and solid waste containers
- Marking pen
- Instructional video (optional)

Suggestions for Pipettor Use

- Practice using both pipettes (adjustable volume and Repeater pipettor) with water and extra tips before you analyze your samples.
- Use a new tip each time you use the Repeater pipettor to pipette a different reagent to avoid reagent cross-contamination. Tips can be rinsed thoroughly, dried completely and reused. By using the same tip to dispense the same reagent each time you can avoid cross contamination.

NOTE: Repeator tips should be changed periodically (after ~10 uses) since precision deteriorates with use.

- Draw the desired reagent volume into the Repeater pipettor and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
- To add reagents using the Repeater pipettor, pipette down the side of the test tube just below the rim.
- When adding samples and standard using the positive displacement pipettor, always pipette into the bottom of the tube without touching the sides or bottom of the tube.
- Use a new adjustable volume pipet tip each time you pipette a new unknown.

Assay Procedure

Prior to performing your first Rapid Assay®, please take time to read the package inserts in their entirety and review the videotape if available. **On site training is strongly recommended for new users of this test system.** Please contact your account manager for further information. This procedure is designed for quantitative analysis. For running the kit semi-quantitatively or qualitatively, please contact Technical Support.

Reagent Preparation

The PAH Enzyme Conjugate is provided as a lyophilized preparation that must be reconstituted prior to use.

1. Prepare the conjugate by adding approximately 3 mL of the conjugate diluent to the lyophilized conjugate vial using the disposable transfer pipet.
2. Swirl gently to dissolve the conjugate.
3. Accurately transfer the vial contents to the diluent bottle.
4. Repeat this procedure twice more with 3 mL aliquots of conjugate diluent.
5. Invert diluent bottle several times to mix completely and let stand approximately 5 minutes before use.
6. Enter the date of reconstitution on the side label of the PAH Conjugate Diluent bottle. Also, enter the expiration date of the PAH Enzyme Conjugate solution which is **21 days** from the date of reconstitution.
7. If the conjugate cannot be used up within 21 days of reconstitution, aliquots should be prepared and frozen. *Frozen aliquots of reconstituted conjugate may be used until the expiration date found on the kit box label.*

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

1. Water samples should be collected in glass vessels with teflon cap liners. **Immediately upon collection, water samples should be diluted with (HPLC grade) methanol (1:3 or 1 part water sample to 3 parts of methanol) to prevent adsorptive losses to the glass containers.** This is a 1.33x dilution, which must be accounted for when interpreting results. See "Results Interpretation", Section 3a for further details. Use this diluted sample as "sample" in "Perform the Test".

NOTE: This 1.33x dilution is not required for soil samples.

2. Samples should be collected in appropriately sized and labeled containers.

3. If testing soil samples, follow the SDI Sample Extraction Kit User's Guide or the appropriate technical bulletin to properly collect and store your sample.

4. Samples should be tested as soon as possible after collection. If this is not possible, storage at 4°C (39°F) is recommended to minimize evaporative losses.

Set Up

1. Remove kits from refrigerator. All reagents must be allowed to come to room temperature prior to analysis. Remove reagents from packaging and place at room temperature at least 1 hour prior to testing.

2. Turn on the RPA-1 or other spectrophotometer. The RPA-1 should be warmed up for at least 30 minutes prior to the run.

3. Label five 12.5 mL Combitips "Conjugate", "Particles", "Wash", "Color" and "Stop". In addition, add the name of the compound you are testing for to each Combitip.

4. Remove nine clean blank test tubes for standards and control and one test tube for each sample (if testing in singlicate). Label the test tubes according to contents as follows.

Tube #	Contents
1	Negative control (replicate 1)
2	Negative control (replicate 2)
3	Standard 1 (replicate 1)
4	Standard 1 (replicate 2)
5	Standard 2 (replicate 1)
6	Standard 2 (replicate 2)
7	Standard 3 (replicate 1)
8	Standard 3 (replicate 2)
9	Control
10	Sample 1
11	Etc.

***Label at top of tubes to avoid interference with reading of tubes in photometer**

Sample Extraction and Dilution

Filtration may be necessary to remove gross particulate from the water sample. If testing at levels higher than standard kit levels is desired, contact SDI for special instructions. Please follow the instructions from the SDI Sample Extraction Kit to prepare and dilute the soil extract prior to running the assay. Dilute water samples as described in "Collect/Store the Sample."

Perform the Test

1. Separate the upper rack from the magnetic base. Place labeled test tubes into the rack.

2. Add **250 uL** of standards, control or samples to the appropriate tubes using the adjustable volume pipet with the dial set on **0250**. The negative control, standards and control must be run with each batch of samples.

NOTE: Sample should be added to the bottom of the tube by inserting the pipet tip into the tube without touching the sides or the bottom of the tube. Take care not to contact sample with pipette tip once dispensed into bottom of the tube.

3. Using the Repeater Pipettor with the "Conjugate" tip attached and the dial set on **"1"**, add **250 uL** of Enzyme conjugate down the **inside wall** of each tube. (Aim the pipet tip ¼" to ½" below the tube rim or tube wall; deliver liquid gently to avoid splashback.)

4. Thoroughly mix the magnetic particles by swirling (avoid vigorous shaking) and attach the "Particles" tip

to the Repeater Pipettor. With the dial set on **"2"** add **500 uL** of magnetic particles to each tube, aiming down the side of the tube as described above. Vortex, mixing each tube 1 to 2 seconds at low speed to minimize foaming. Pipetting of magnetic particles should be kept to 2 minutes or less.

5. Incubate 30 minutes at room temperature.

6. After the incubation, combine the upper rack with the magnetic base and press all tubes into the base; allow 2 minutes for the particles to separate.

7. With the upper rack and magnetic base combined, use a smooth motion to invert the combined rack assembly over a sink and pour out the tube contents.

NOTE: If the rack assembly inadvertently comes apart when lifting to pour out tube contents, re-combine and wait an additional 2 minutes to allow particles to separate.

8. **Keep the rack inverted** and gently blot the test tube rims on several layers of paper towels. It is important to remove as much liquid as possible but **do not bang** the rack or you may dislodge the magnetic particles and affect the results.

9. Set the Repeater Pipettor dial to **"4"** and put on the tip labeled "Wash". Add **1 mL** of Washing Solution down the inside wall of each tube by using the technique described earlier. Vortex tubes for 1-2 seconds. **Wait 2 minutes** and pour out the tube contents as described previously. **Repeat this step one more time.**

NOTE: The number of washes and wash volume are important in ensuring accurate results.

10. Remove the upper rack (with its tubes) from the magnetic base. With the "Color" tip attached to the Repeater Pipet and the dial set to **"2"** add **500 uL** of Color Reagent down the inside wall of each tube as described previously. Vortex 1 to 2 seconds (at low speed).

11. Incubate 20 minutes at room temperature. During this period, add approximately 1 mL of Washing solution to a clean tube for use as an instrument blank for "Results Interpretation".

12. After the incubation, position the Repeater pipettor at Setting **"2"** and use the "Stop" tip to add **500 uL** of Stop solution to all test tubes.

13. Proceed with results interpretation.

WARNING: Stop solution contains 2M sulfuric acid. Handle carefully.

Results Interpretation

1. After addition of Stop Solution to the test tubes, results should be read within 15 minutes.

2. Wipe the outside of all antibody coated tubes prior to photometric analysis to remove fingerprints and smudges.

Photometric Interpretation Using the RPA-I

1. The RPA-I photometer (provided in the Rapid Assay® Accessory kit) can be used to calculate and store calibration curves. It is preprogrammed with various RaPID Assay® protocols. To obtain results from the PAHs Rapid Assay® test kit parameters are as follows:

Data Reduct:	Lin. Regression	
Xformation :		Ln/LogitB
Read Mode	:	Absorbance
Wavelength :	:	450 nm
Units	:	PPB
# Rgt Blk	:	0

Calibrators:

# of Cals	:	4
# of Reps	:	2

Concentrations:

#1:	0.00	ppb
#2:	2.00	ppb
#3:	10.0	ppb
#4:	50.0	ppb
Range	:	0.7 – 50.0

Correlation	:	0.990
Rep. %CV	:	10%

NOTE: Prior to analysis the RPA-I User's Manual should be thoroughly reviewed for more detailed operation instructions.

2. Follow the instrument prompts to read the absorbance of all tubes:

Instrument Display	Operator Response
SELECT COMMAND RUN PROTOCOL	Press RUN Scroll using the YES [] or NO [] keys until the desired protocol appears. Then press ENTER
SPL. REPLICATES (1-5)	Press 1 (for analysis of samples in singlicate.) Press ENTER
BLANK TUBE, INSERT TUBE, EVALUATING TUBE, REMOVE TUBE (Beep)	Insert blank tube containing 1mL wash solution. Remove tube
CAL #1, REP. #1, INSERT TUBE, EVALUATING TUBE, REMOVE TUBE (Beep)	Insert Tube #1 Remove tube

Follow prompts to read tubes.

NOTE: Tube order is important. The RPA-I expects to see the standards in ascending order, in duplicate, starting with the negative control.

Following evaluation of all standards, the instrument will display:

PRINTING DATA,	Data will print
PRINTING CURVE	Curve will print only if programmed to print (See RPA1 User's Manual).
CTRL #1 REP #1, INSERT TUBE, EVALUATING TUBE, REMOVE TUBE (Beep)	Insert Control Tube Remove Tube
EDIT CALIBRATORS YES/NO	Press NO (if editing is necessary press YES and refer to the RPA1 User's Manual).
SPL #1 REP#1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert first sample tube Remove tube

Continue to follow prompts. After all samples have been read, press STOP.

Expected Results:

- **%CV (coefficient of variation) between standard duplicates of 10% or less.**
- **Absorbance reading for the 0 ppb standard should be between 0.8 and 2.0 for all assays.**
- **Correlation (r) of 0.990 or greater for all assays.**
- **Kit control within range specified on vial.**
- **Absorbance of negative control and standards should be as follows:**

  Negative Control>Std. 1>Std. 2>Std. 3.

3. Concentrations will be indicated for all samples on the RPA-I printout.

   a) The concentration, as indicated on the printout, is multiplied by the appropriate dilution factor (if applicable) introduced in the procedure. The quantitation range of the kit is also multiplied by this factor.

   EXAMPLE: Water samples were diluted 1.33 with methanol upon collection (see "Collect/Store the Sample" in the User's Guide). As a result, the concentrations listed on the printout should be multiplied by 1.33 to determine the sample concentration. The standard concentrations are also multiplied by 1.33 to give a quantitation range in water 2.66 to 66.5 ppb.

   b) Samples with an "nd" and no concentration listed have an absorbance greater than the negative control; therefore, no concentration

can be computed for these samples. Results must be reported as <2.66 ppb (or Standard 1 multiplied by the dilution factor).

c) Samples with an "nd" next to a listed concentration have an estimated concentration below the minimum detection level of the test kit. Results must be reported as <2.66 ppb (or Standard 1 multiplied by the dilution factor).

NOTE: Any samples with concentrations determined to be lower than Standard 1 (the limit of quantitation) must be reported as <2.66 ppb (or Standard 1 multiplied by the dilution factor). Quantitation is not possible below this standard as this is outside the linear range of the assay.

d) Similarly, samples with a "hi" next to a listed concentration have an estimated concentration higher than Standard 3 and must be reported as >66.5 ppb (or Standard 3 multiplied by the dilution factor).

NOTE: In order to determine the concentration of samples with concentrations greater than Standard 3, they must be subjected to repeat testing using a diluted sample. A ten-fold or greater dilution of the sample is recommended with an appropriate amount of PAH diluent. This additional dilution must then be taken into account when calculating the concentration. Please contact Technical Support for assistance in performing dilutions.

Photometric Interpretation Using Other Photometers

Other photometers may also be used to interpret results obtained from the RPA-I photometer. It is important that the photometer be able to read absorbance at 450nm and that the instrument can read at a 1 mL fill volume. Absorbances obtained from other spectrophotometers (reading at 450 nm) may be used to manually calculate sample concentrations as outlined below.

1. Calculate the mean absorbance for each of the three standards and the negative control.

2. Determine the standard deviation and %CV (coefficient of variation) of each standard and ensure %CV is less than 10% for each.

3. Calculate the %B/Bo for each standard by dividing the mean absorbance value for the standard by the mean absorbance value for the negative control and multiplying the results by 100.

4. Construct a standard curve by plotting the %B/Bo for each standard on the vertical logit (y) axis versus the corresponding analyte concentration on the horizontal logarithmic (x) axis on the graph paper provided in the test kit. **Graph papers are specific for each method. Use only the graph paper supplied with each kit.**

5. Draw the best straight line through all points. Using the %B/Bo of the sample, the concentration can be interpolated from the standard curve.

6. Multiply results by the appropriate dilution factor (if applicable) introduced in the procedure. For example, if the sample was diluted 10-fold to increase the detection levels of the kit then the results must be multiplied by 10. This dilution also changes the range of the assay (standards) by the same factor.

Limitations of the Procedure

The Rapid Assay® PAH Test Kit is a screening test **only**. Sampling error may significantly affect testing reliability. Adequate sample number and distribution are the responsibility of the analyst.

Ordering Information

Description	Catalogue Number
Rapid Assay® PAH 30 Tube Kit	A00156
Rapid Assay® PAH 100 Tube Kit	A00157
Rapid Assay® Accessory Kit**	6050100
Adjustable Volume Pipet Tips (100-1000 uL)	A00013
12.5 mL Combitip for Repeating Pipette (1 each)	A00009
PAH Diluent	A00159
PAH Soil Proficiency Sample	A00158
Rapid Assay® Rental Accessory Kit	6997010
**** To obtain part numbers and pricing for individual items in the Accessory Kit contact SDI at the number below.**	

Ordering/Technical Assistance

Should you have any questions regarding this procedure prior to analysis contact Technical Service to avoid costly mistakes.

To Place an Order or Receive Technical Assistance, please call Strategic Diagnostics Inc. at:

Call toll-free **800-544-8881**`

Or 302-456-6789 Phone
302-456-6782 Fax
Web site: www.sdix.com
E-mail: techservice@sdix.com

General Limited Warranty

SDI's products are manufactured under strict quality control guidelines and are warranted to be free from defects in materials and workmanship. New instruments and related non-expendable items are warranted for one year from date of shipment against defective materials or workmanship under normal use and service.
Warranty obligation is limited to repair or replacement of the defective product or to refund of the purchase price, at the discretion of SDI. Other warranties, express or implied, are disclaimed. SDI's liability under any warranty claim shall not exceed the refund of the purchase price paid by the customer. Under no circumstances shall SDI be liable for special, indirect or consequential damages.

Safety

To receive an MSDS for this product, visit our web site at www.sdix.com.

Z00304.1, Rev 4/6/00

Operation of the Repeater Pipet

To Set or Adjust Volume

To determine the pipetting volume, the dial setting (1-5) is multiplied by the minimum pipetting volume of the tip (indicated on the side of the Combitip, e.g. 1≃100 uL.)

To Assemble Pipet Tip

Slide filling lever down until it stops. Then raise the locking clamp and insert the tip until it clicks into position. Be sure the tip plunger is fully inserted into the barrel before lowering the locking clamp to affix the tip in place.

To Fill Tip

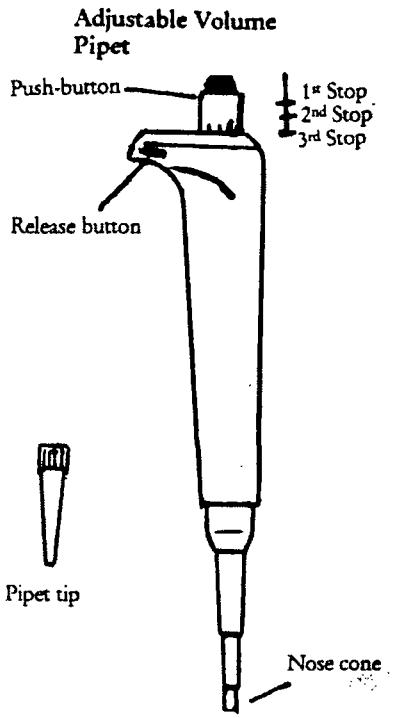
With tip mounted in position on pipet, immerse end of tip into solution. Slide filling lever upward slowly. Combitip will fill with liquid.

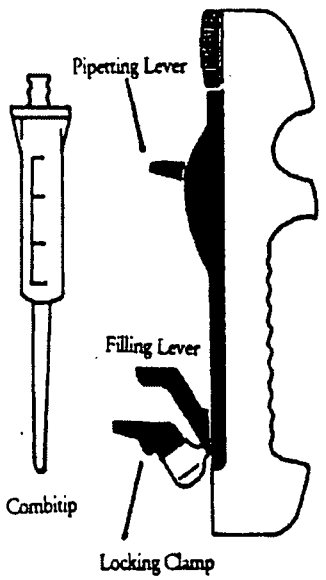
To Dispense Sample

Check the volume selection dial to ensure pipetting volume. Place tip inside test tube so that tip touches the inner wall of tube. Completely depress the pipetting lever to deliver sample. NOTE: Dispense one portion of reagent back into the container to engage the ratchet mechanism and ensure accuracy.

To Eject Tip

Empty tip of any remaining solution into appropriate container by pushing filling lever down. Raise locking clamp upward, and remove the Combitip.





Operation of the Adjustable Volume Pipet

To Set or Adjust Volume

Press release button on side of pipette and turn the push-button to adjust volume up or down. Volume setting is displayed on top of pipet. See kit instructions for appropriate setting. Pipet will accurately dispense volumes between 100 and 1000 uL.

To Assemble Pipet Tip

Gently push nose cone of pipet firmly into a pipet tip contained in the pipet tip rack.

To Withdraw Sample

Keep pipet almost vertical. With tip mounted in position on pipet, press push-button to 1st stop and hold it. Place tip at bottom of liquid sample and slowly release push-button to withdraw measured sample. Ensure that no air bubbles exist in the pipette tip. If bubbles exist, dispense sample and re-withdraw. Slide tip out along the inside of the vessel.

To Dispense Sample

Wipe any liquid from outside of tip taking care not to touch orifice. Place tip into tube, almost to the bottom, and slowly press push-button to 2nd stop. Hold push-button at 2nd stop when removing tip from tube.

To Eject Tip

Press push-button to 3rd stop. Tip is ejected.

ATTACHMENT 2 – SAMPLE FIELD FORMS

This attachment provides the following forms for survey information collection and field sampling documentation:

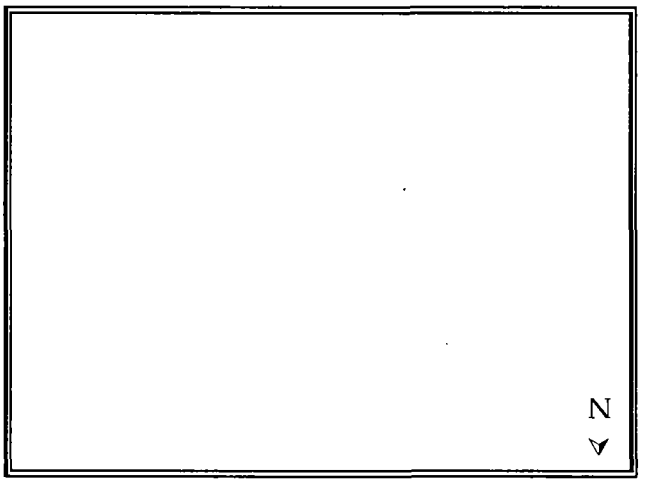
- **PRODUCE SAMPLE DATA SHEET**
- **HOUSE DUST STUDY PRE-SAMPLING SURVEY**
- **HOUSE DUST SAMPLE DATA SHEET**
- **CHAIN OF CUSTODY / SAMPLE ANALYSIS REQUEST FORM**
- **SOIL SAMPLE CLASSIFICATION FORM**
- **SAMPLE LABEL**
- **SURFACE WATER SAMPLING FIELD DATA FORM**
- **SOIL SAMPLING FIELD DATA FORM**

PRODUCE SAMPLE DATA SHEET

Date: _____ Time Begin: _____ Time End: _____
Sample Crew: _____
Sample Nos: (AG)_____ (BG) _____
Sample Location: _____
Temperature: _____ Humidity: _____%
Approximate Garden Dimensions: _____ ft x _____ ft
Above-ground Produce Sample: _____ Approx. Quantity: _____
Below-ground Produce Sample: _____ Approx. Quantity: _____
Does resident peel produce prior to consumption? Yes _____ No _____
Equipment Decontamination Check: _____ (initial)
List Produce and Approximate Quantity Currently Growing in Garden:

PRODUCE TYPE	APPROX. QUANTITY	AG/BG

Sketch of Residence, Location of Garden:


N

HOUSE DUST STUDY PRE-SAMPLING SURVEY

1. Today's date: _____

2. Name and Address of Household: _____

_____

_____

_____

_____

3. Telephone Number of Household: _____

4. How long has the family lived at the present address? _____ Years

5. How many people live in household? Adults_____ Children _____
   What are children's ages? _____

6. How old is the house? (Circle one)
1 = 0-9 years old
2 = 10-19 years old
3 = 20-29 years old
4 = 30-39 years old
5 = 40-49 years old
6 = 50 or more years old

7. How old is the carpet? _____

8. What type of carpet is in the house? Plush _____ Level Loop ____ Multilevel ____ Shag _____

9. What type of vacuum is used: Upright ____ Canister _____ Other _____
   How often is vacuum used? _____ times per month

10. Where do family members spend most of their time when at home?
   Adults:
   Children:

11. Does anyone smoke in the home? Yes _____ No _____
If yes, how many people? _____
With what frequency? _____

12. Are there any bare soil areas in the yard (including flower or vegetable gardens)? What are they used for (e.g., garden child's play area)?

Area Type Use

_____

_____

_____

_____

Approx. % of grass cover in yard: _____ %

13. Is there outdoor play equipment in the yard? Yes _____ No _____

14. What % of days are windows and/or doors left open? _____%
Are there screens on windows and/or doors? Yes _____ No _____

15. Are there pets at this home? How many? Where do they spend their time?

Pet Name	Pet Type	Exclusively Indoors	Exclusively Outdoors	Indoors and Outdoors

16. Are shoes worn inside the home? Yes _____ No _____

17. What is primary entry/exit for residents (front door, back door, garage, etc)?

_____

18. Does the home have a fireplace? Yes _____ No _____
If yes, % of time used: _____ %
If yes, do you ever burn trash, newspapers, or painted wood in the fireplace?
Yes_____ No _____
How often? _____

19. What is the home's heating source (e.g., wood, electric, oil, etc)? _____
_____

20. Are there any cars, motorcycles, lawn mowers, garden equipment, or other machinery that use diesel or gasoline at the house? Yes _____ No _____

21. Is there a garage at this house? Yes _____ No _____
If yes, is it attached to the house? Yes _____ No _____

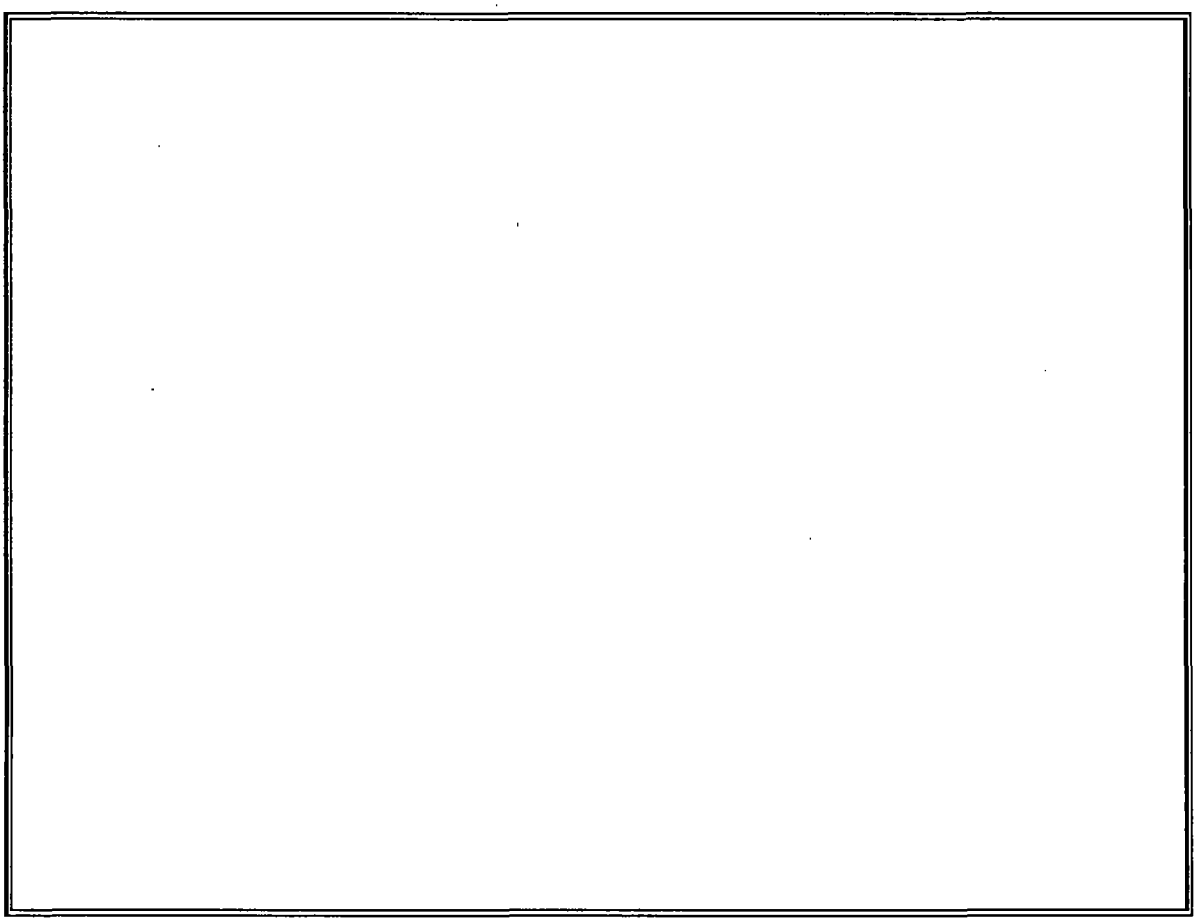
HOUSE DUST SAMPLE DATA SHEET

Date: _____ Time Begin: _____ Time End: _____
Sample Crew: _____
Sample No: _____
Sample Location: _____
Temperature: _____ Humidity: _____%

Type of Carpet: Plush _____ Level Loop _____ Multilevel _____ Shag _____
Type of Vacuum: Upright _____ Canister _____ Other _____
Date Carpet Last Vacuumed: _____ Approx. Age of Carpet: _____

Comments: _____
_____

Location of Area Sampled: _____ Dimensions: _____
Leak Check: Yes _____ No ____; 20 second cleaning @ end of sampling: Yes_____ No___
Total Sample Time: _____
Sketch of Areas Sampled:



CHAIN OF CUSTODY RECORD/SAMPLE ANALYSIS REQUEST FORM

Facility Number/Project Name:

Lab Contact: _____ Office: ____

Ship to: _____

_____

_____

Lab Contact Phone: _____

Samplers:

Company Name:

Analyses Requested

Sample No.	Date	Time	Matrix							Extra Container	Archive	Remarks

INTERNATIONAL PAPER

Matrix Code: GW - Groundwater SL - Soil SD - Sediment SW - Surface water

OTHER - Please identify codes _____

Priority: ☐ Normal ☐ Rush Rush time period _____

Shipped via: ☐ FedEx/UPS ☐ Courier Airbill _____

Condition of Samples Upon Receipt: _____

Custody Seal Intact: ☐ Yes ☐ No ☐ None

Relinquished by: _____ (Signature) Date/Time: _____

Company: _____

Relinquished by: _____ (Signature) Date/Time: _____

Company: _____

Received by: _____ (Signature) Date/Time: _____

Company: _____

Received by: _____ (Signature) Date/Time: _____

Company: _____

Distribution: White and Yellow Copies - Accompany Shipment; Pink Copy - Project File

INTERNATIONAL PAPER

FACILITY NUMBER	PROJECT NAME
SITE / LOCATION	BORING No.

LOCATION SKETCH

NORTH

SAMPLING CONTACT/COMPANY:

DRILLING METHOD: BOREHOLE DIAMETER:

SAMPLING METHOD:

DRILLING CONTRACTOR:

STAFF:

SHEET OF

WATER LEVEL INFORMATION					
	DATE				
	TIME				
	DEPTH TO WATER				
	CASING / AUGER DEPTH				

START DATE / TIME:

FINISH DATE / TIME:

GROUND SURFACE ELEVATION:_____

SAMPLER TYPE	SAMPLE NUMBER	TIME	BLOW COUNT	RECOVERY	OVM (ppm) HS / BZ	INTERVAL DEPTH	UNIFIED SYMBOL	Surface Conditions **Description** - USC group name, minor components color, moisture (particle angularity, shape, plasticity, odor, consistency, density, cementation, geologic interpretation, drilling conditions)	Comments

TYPES OF SOIL SAMPLERS:
SS - Split Spoon
D&M - Damas and Moore U-Type
SPT - Standard Penetration Test

ABBREVIATIONS:
HS - HEAD SPACE
BZ - BREATHING ZONE

KEYS TO SAMPLES:
▨ INDICATES SAMPLE INTERVAL
▩ INDICATES ANALYTICAL SAMPLE COLLECTED

Sample Label

INTERNATIONAL PAPER

SAMPLE NO.

SITE NAME

DATE | TIME

SAMPLER | PRESERVATIVE

Chain of Custody Seal

INTERNATIONAL PAPER OFFICIAL SAMPLE SEAL	SAMPLE NO.	DATE
	SIGNATURE	
	PRINT NAME AND TITLE	

INTERNATIONAL PAPER

Surface Water Sampling Field Data Form

STATION IDENTIFICATION	PROJECT NAME	DATE
FACILITY NUMBER	LOCATION	TIME
SAMPLING CONTACT/COMPANY	SAMPLER'S PROJECT NUMBER	

FIELD CONDITIONS/ WEATHER _____

FIELD TEAM (signature/company): _____

_____

FIELD MEASUREMENTS

Gauge Height: _____ **Substrate Level on Gauge:** _____ **pH:** _____

Dissolved Oxygen (ppm): _____ **Water Temperature (°C):** _____

SC @ Field Temperature (microhos/cm): _____ **Estimated Streamflow (cfs):** _____

Turbidity: _____

_____

_____

WATER QUALITY SAMPLES

	SAMPLE No.	DATE / TIME	STATION	CONTAINER TYPE	FILTERED	PRESERVATIVE	ANALYSIS
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							

SITE SKETCH

INTERNATIONAL PAPER

Soil Sampling Field Data Form

STATION IDENTIFICATION	PROJECT NAME	DATE
FACILITY NUMBER	LOCATION	TIME
SAMPLING CONTACT/COMPANY	SAMPLER'S PROJECT NUMBER	

FIELD CONDITIONS/ WEATHER _____

FIELD TEAM (signature/company): _____

_____

SOIL SAMPLES

	SAMPLE NUMBER	DATE / TIME	STATION	DEPTH INTERVAL	SOIL TYPE / LAYER	CONTAINER TYPE	ANALYSIS CODE
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							

PROFILE / SITE SKETCH

Attachment 3 – Field Sampling Equipment Lists

This attachment presents equipment lists for field sampling. These lists should be evaluated by the lead field sampler and field crews in conjunction with the pre-evolution review of methods and health and safety. Additional supplies may be necessary based on site-specific needs and Field Sampling Plan specifications. The following lists are included in this attachment:

- **Field equipment for soil and soil invertebrate sampling,**
- **Field equipment for surface water sampling,**
- **Field equipment for sediment and benthic invertebrate sampling,**
- **Field equipment for garden produce/wild rice/forage plant sampling, and**
- **Field equipment for house dust sampling.**

Field equipment for soil and soil invertebrate sampling

General Equipment
Field vehicle
Clipboard
Camera or digital camera with time/date patch
Color slide film or disks
First-aid kit
Field sampling plan
Sampling and analysis plan - Figure 3
Site-specific health and safety plan (HSP)
Health and safety equipment listed in HSP
Sample labels and Sharpie pens
Logbook, indelible ink pens
Chain-of-custody/sample analysis request forms
Relevant field forms
Shipping forms and labels
Chain-of-custody seals
Cellular phone
Rubber boots
Tool kit
GPS unit

Decontamination Equipment
Plastic tubs or coolers
Ten 10-gallon garbage bags
Silver Shield gloves
Beaker brush
Bristle brush
Water carboys
Organic-free deionized water from analytical laboratory
Tap water
Pesticide-grade acetone
Pesticide-grade hexane
Liquinox
Plastic squirt bottles
Aluminum foil or untreated butcher paper

Miscellaneous Equipment
Sample packaging materials/bubble wrap
Analytical balance (0.1mg accuracy)
Cellophane tape to cover sample tags
Strapping tape
Duct tape (2 rolls)
Rain gear
Utility knife
10-ft metal tape measure
Plastic garbage bags
Paper towels

Sampling Equipment
Sample bottles
Ice chest (s)
Ice or freezable cold packs
Nitrile gloves
1-in. ID stainless-steel hand auger
Stainless-steel spoons
Stainless-steel bowls
Stainless steel knife
Shovel
Stainless steel ruler
Plastic tarp
Potting soil
Earth Colors soil color chart
100-ft cloth measuring tape
Compass

Field equipment for surface water sampling

General Equipment
Boat (if needed)
Large-scale site map
Sampling and analysis plan - Figure 5
Camera with date/time patch, color print film
Site and profile description logs
Topographic map
First-aid kit
Field sampling plan, quality assurance project plan, health and safety plan, and related documents
Chain-of-custody/sample analytical request forms
Daily activity forms
Sample container labels and custody seals
Logbook, indelible pens, pencils
Shipping paperwork
Waders
Personal flotation devices (if needed)
500-ft anchor line and three anchors (if boat used)
GPS unit

Sampling Equipment
Sample bottles
Beakers
Brunton compass
Tape measure (10-, 50-, and 100-ft)
Duct tape
Resealable plastic bags
Trash bags
Ice chests, refreezable cold packs
Large stainless-steel bucket or churn splitter for composite samples
Dissolved oxygen meter
C-Flex® and Teflon® tubing
Stainless-steel strainer
Vacuum hand pump (with filters if applicable)
Sample tubing
Multimeter with calibration solutions

Decontamination Equipment
Brushes
Plastic tubs
Kimwipe® towels
Alconox® detergent
Protective clothing
Nitrile gloves
Polyethylene sheeting
Distilled/deionized water
Decontamination solvents (generally hydrochloric or nitric acid, acetone, and hexane)

Miscellaneous Equipment
Sample packaging materials/bubble wrap
Cellophane tape to cover sample labels
Strapping tape
Fragile labels

Field equipment for sediment and benthic invertebrate sampling

General Equipment
Boat
Large-scale site map
Sampling and analysis plan - Figures 4 and 5
Camera with date/time patch, color print film
Site and profile description logs
Topographic map
First-aid kit
Respirators with combination organic vapor/HEPA cartridges (if required by HASP)
Field sampling plan, quality assurance project plan, health and safety plan, and related documents
Chain-of-custody/sample analytical request forms
Daily activity forms
Sample container labels and custody seals
Logbook, indelible pens, pencils
Shipping paperwork
Waders
Personal flotation devices
500-ft anchor line and three anchors
GPS unit

Sampling Equipment
Sample bottles
Brunton compass
Tape measure (10-, 50-, and 100-ft)
Duct tape
Resealable plastic bags
Trash bags
Flagging and stakes
Ice chests, refreezable cold packs
Stainless-steel scoops, spatulas, knives
Munsell color chart
Stainless-steel mixing bowls, spoons
Ponar grab, Ekman dredge, Piston-tube core setup, or mini van Veen grab sampler, and line

Wet Sieving Equipment
1-L graduate cylinder
Analytical balance (0.1mg accuracy)
Funnel
Distilled water
50 mL beakers
Stainless steel spoon
Water squirt bottle
Nitrile gloves
Weigh pans

Decontamination Equipment
Brushes
Plastic tubs
Kimwipe® towels
Alconox® detergent
Protective clothing
Nitrile gloves
Polyethylene sheeting
Distilled/deionized water
Decontamination solvents (generally hydrochloric or nitric acid, acetone, and hexane)

Miscellaneous Equipment
Sample packaging materials/bubble wrap
Cellophane tape to cover sample labels
Strapping tape
Fragile labels

Field equipment for garden produce/wild rice/forage plant sampling

General Equipment	**Miscellaneous Equipment**
Field vehicle	Analytical balance (0.1mg accuracy)
Clipboard	Sample packaging materials
Camera or digital camera with time/date patch	Cellophane tape to cover sample tags
Color slide film or disks	Strapping tape
First-aid kit	Duct tape (2 rolls)
Field sampling plan	Rain gear
Sampling and analysis plan	Utility knife
Site-specific health and safety plan (HSP)	Garden shears
Health and safety equipment listed in HSP	10-ft metal tape measure
Sample labels and Sharpie pens	Plastic garbage bags
Logbook, indelible ink pens	Paper towels
Chain-of-custody/sample analysis request forms	**Sampling Equipment**
Shipping forms and labels	Sample bottles
Chain-of-custody seals	Large Ziplock-type bags
Cellular phone	Ice chest (s)
Rubber boots	Ice or freezable cold packs
Waders	Nitrile gloves
Tool kit	Garden shears
GPS unit	Aluminum foil
Decontamination Equipment	Hand digging tools
Plastic tubs or coolers	Stainless-steel bowls
Ten 10-gallon garbage bags	Shovel
Silver Shield gloves	Plastic tarp
Beaker brush	100-ft cloth measuring tape
Bristle brush	Compass
Water carboys	
Organic-free deionized water from analytical laboratory	
Tap water	
Pesticide-grade acetone	
Pesticide-grade hexane	
Liquinox	
Plastic squirt bottles	
Aluminum foil or untreated butcher paper	

Field equipment for house dust sampling

General Equipment
Field vehicle
Clipboard
Camera or digital camera with time/date patch
Color slide film or disks
First-aid kit
Field sampling plan
Sampling and analysis plan
House Dust Sample Data Sheets
Site-specific health and safety plan (HSP)
Health and safety equipment listed in HSP
Sample labels and Sharpie pens
Logbook, indelible ink pens
Chain-of-custody/sample analysis request forms
Shipping forms and labels
Chain-of-custody seals
Cellular phone
Tool kit

Miscellaneous Equipment
Sample packaging materials
Cellophane tape to cover sample tags
Strapping tape
Duct tape (2 rolls)
Utility knife
100-ft cloth tape measure

Sampling Equipment
Analytical balance (0.1mg accuracy)
Approved vacuum cleaner and fitted bottles
Decon equipment for vacuum cleaner (if specified in manufacturer's instructions)
Sample bottles
Masking tape
Large Ziplock-type bags
Ice chest (s)
Ice or freezable cold packs
Nitrile gloves
Compass