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Appendix A

OU6 Rail Maintenance SAP Project-Specific Procedures and Libby Asbestos Site Standard Operating Procedures

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Project-Specific Procedures - Rail Maintenance Activity-Based Sampling and Analysis Plan

Operable Unit 6
Libby Asbestos Site
Libby, Montana
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Acronyms

AS	Analytical Sensitivity
ASTM	American Society of Testing and Materials
BNSF	Burlington Northern Santa Fe
CARB	California Air Resource Board
CFR	Code of Federal Regulations
COC	Chain-of-Custody
cm	Centimeter
DB	Drying Blanks
EDD	Electronic Data Deliverable
FSDS	Field Sample Data Sheet
FTL	Field Team Leader
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
ID	Identification
IDW	Investigation Derived Waste
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
LA	Libby amphibole
LB	Laboratory Blank
MB	Method Blank
mm	Millimeter
ND	non-detect
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NOAA	National Oceanic and Atmospheric Administration
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PCM	Phase Contrast Microscopy
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RD	Recount Different
ROW	Right-of-Way
RS	Recount Same
SAP	Sampling and Analysis Plan

Site	Libby Asbestos Site
SOP	Standard Operating Procedure
TEM	Transmission Electron Microscopy
Tr	Trace
ug	Microgram
USEPA	U.S. Environmental Protection Agency
USGS	United States Geological Survey
VE	Visual Estimation

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<u>Project-Specific Procedures</u>

Project-Specific Procedure - 1 Sample Custody

Project-Specific Procedure - 2 Packaging and Shipping Environmental Samples

Project-Specific Procedure - 3 Guide to Handling Investigation-Derived Waste (IDW)

Project-Specific Procedure - 4 Field Logbook Content and Control

Project-Specific Procedure - 5 Photographic Documentation of Field Activities

Project-Specific Procedure - 6 Control of Measurement and Test Equipment

<u>Site-Specific Standard Operating Procedures (SOPs)</u>

(CDM-LIBBY-05, Revision 2) Site-Specific SOP for Soil Sample Collection

(CDM-LIBBY-06, Revision 1) with modifications, Site-Specific SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil

(SRC-LIBBY-01, Revision 2) Site-Specific SOP for Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy

(SRC-LIBBY-03, Revision 1) Site-Specific SOP for Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy

(CDM-LIBBY-09, Revision 0) Site-Specific SOP for GPS Coordinate Collection and Handling

(USEPA Emergency Response Team [ERT] #2084) with modifications, Activity-Based Air Sampling for Asbestos

(EPA-LIBBY-09 Rev1) Site-Specific SOP for TEM Data Review and Data Entry Verification

(EPA-LIBBY-08) Site-Specific SOP for Indirect Preparation of Air and Dust Samples for TEM Analysis

(ISSI-LIBBY-01) Site-Specific SOP

(LB-000016) Site-Specific SOP

(LB-000019) Site-Specific SOP

(LB-000024) Site-Specific SOP

(LB-000024a) Site-Specific SOP

(LB-000028) Site-Specific SOP

(LB-000029a) Site-Specific SOP

(LB-000029b) Site-Specific SOP

(LB-000030) Site-Specific SOP

(LB-000031) Site-Specific SOP

(LB-000045) Site-Specific SOP

(LB-000053) Site-Specific SOP

(LB-000066c) Site-Specific SOP

(LB-000084) Site-Specific SOP

(LB-000085 rev) Site-Specific SOP

(LB-000086) Site-Specific SOP

This document provides the project-specific procedures for all activities (field and laboratory based) to support the investigation described in the Rail Maintenance Activity-Based Sampling and analysis Plan for Operable Unit 6 (OU6) at the Libby Asbestos Site (Site), which is defined as the Railroad Transportation Corridors or Right-of-Way (ROW). This document is meant to provide the basic elements of a quality assurance project plan (QAPP); however, the QAPP for OU6 will be formally developed as part of the RI/FS Work Plan once an Administrative Order for OU6 is finalized. The following sections describe various sampling activities to be conducted as part of upcoming rail maintenance activities scheduled to occur in September 2008.

The information presented in this document is essentially a compilation of existing approved Libby Site procedures and were obtained either from the Libby project website [http://www.epa.gov/region8/superfund/libby/] or from USEPA.

This project is being conducted for Burlington Northern Santa Fe Railway Company (BNSF) by ENSR and EMR Incorporated, who are contractors to BNSF.

1.0 Field Planning Meetings

Prior to the start of any new field programs and investigation activities, a field planning meeting will be conducted by ERM and will be attended by all field staff conducting the work, in addition to CDM staff who will provide oversight for the sampling activities. The United States Environmental Protection Agency (USEPA) and CDM will be notified of the meeting's date and time. The meeting will briefly discuss and clarify:

- Objectives and scope of the fieldwork;
- Equipment and training needs;
- Field operating procedures, schedule of events, and individual assignments;
- Required quality control (QC) measures;
- Health and safety requirements;
- Documents governing fieldwork that must be on site; and
- Any changes in the field planning documents.

A written agenda will be distributed and an attendance list will be circulated for signature. Copies of these documents will be maintained in EMR's project files. Additional meetings will be held when the documents governing fieldwork require it or when the scope of the assignment changes significantly.

The field team personnel will perform the following activities before and during field activities, as applicable:

- Review and understand applicable governing documents
- Record appropriate levels of documentation regarding activities conducted
- Ensure that all sample analyses are scheduled through the laboratory
- Obtain required sample containers and other supplies
- Obtain and check field sampling equipment
- Obtain and maintain personal protective equipment (PPE)

2.0 Field Team Training Requirements

Prior to starting work at the Site, any new team member must complete the following, at a minimum:

- Read the health and safety plan (HASP; see Appendix B) – documented on plan signature sheet and investigation-specific Required Reading Report;
- Attend an orientation session with the Site health and safety office – documented on orientation session attendance sheet; and
- Read and understand all relevant governing documents – documented on an investigation-specific Required Reading Report.

Depending on the work to be conducted, additional training may apply. These requirements may include the following:

- Occupational Safety and Health Administration (OSHA) 40 hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8 hour refreshers – documented by training certificates;
- Respiratory protection as required by 29 Code of Federal Regulations (CFR) 1910.134 – documented by training certificate;
- Asbestos awareness as required by 29 CFR 1910.1001 – documented by training certificate
- Sample collection techniques – documented by field logbook entries or on training session attendance sheets; and
- Identification of vermiculite and Libby mine related materials – documented by field logbook entries or on training session attendance sheets.

All documentations of trainings are stored in EMR's Libby project files.

3.0 Equipment Decontamination

Soil Sampling Equipment used to collect, handle, or measure soil samples will be decontaminated in accordance the project-specific procedure, with project-specific modifications, as described in CDM-LIBBY-05, Site-Specific Standard Operating Procedure (SOP) for Soil Sample Collection (CDM 2007, Section 1).

When air sampling equipment is used in an environment where asbestos structures may be present (i.e., activity-based sampling), the equipment will be wiped down using commercially available wet-wipes or paper towels moistened with locally available distilled water in general conformance with the project-specific procedure presented in Section 4.2 of the HASP. Materials used in the decontamination process will be disposed of as investigation derived waste (IDW) which is described in Section 10.0.

4.0 Field Logbooks

Field logbooks will be maintained in general conformance with the Project-Specific Procedure – 4: Field Logbook Content and Control with project-specific modifications. The field logbook is an accounting of activities at the Site and will duly note problems or deviations from the governing plans and observations related to investigation-specific sampling and analysis plan (SAPs).

As described in the Project-Specific Procedure – 4: Field Logbook Content and Control, field logbook modifications will be completed with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry. Separate field logbooks will be kept for each investigation program and the cover of each field logbook will clearly indicate the name of the investigation program and its sequence number.

Field logbooks will be completed daily for each investigation activity prior to leaving a property. Field logbooks will be checked for completeness and adherence on a daily basis for the first week of each new activity. When incorrect field logbook completion procedures are discovered during these checks, the errors will be discussed with the author of the entry and corrected. Field logbook checks will be extended to once per month as activities continue, and any errors noticed during the checks will be discussed with the author.

The field administrative staff will manage the field logbooks by assigning unique identification numbers to each field logbook, tracking to who and the date each field logbook was assigned, the investigation activities recorded in each field logbook, and the date when the field logbook was returned. As field logbooks are completed, originals will be maintained by EMR and a copy of the logbooks will be sent to USEPA.

5.0 Field Sample Data Sheets (FSDSs)

Media-specific field sample data sheet (FSDSs) will be used to record detailed sample notes for each field and QC samples. FSDSs are area-specific – up to 3 individual samples from the same area can be recorded on an FSDS. If columns are left incomplete due to fewer than three samples being recorded on a sheet, the blank columns will be crossed out dated and signed by the staff member completing the sheet. Erroneous information recorded on a sheet will be corrected with a single line strikeout initial and date. The correct information will be entered in close proximity to the erroneous entry. The version of each FSDS in use at the Site for each type of media to be sampled are included as Appendix D of the OU6 Rail Maintenance SAP.

FSDSs will be completed in the field before field personnel leave the sampling location. To ensure that all applicable data are accurately entered and all fields are complete, a different field team member will check each FSDS. The team member completing the form and the team member checking the form will initial the FSDS in the proper fields. In addition, investigation-specific FTL also complete periodic checks of FSDSs prior to relinquishment to the sample coordinator. Once FSDSs and samples are relinquished to the sample coordination staff, the sheets are again checked for accuracy and completeness. The member of the sample coordination staff who completed the check and data entry of required information will initial the FSDS in the proper field.

If during any of these checks a revision is required to the FSDS, it will be returned to the team member initially responsible for its completion. The error will be explained to the team member and the sheet corrected. If the team member is no longer onsite, revisions will be made by sample coordination staff.

Each media-specific FSDS is assigned a unique sequential ID number. This number will be referenced in field logbook entries related to samples recorded on individual sheets. Field administrative staff will manage the FSDSs and send a copy of the completed sheets to USEPA by media and individual sheet number.

6.0 Photographic Documentation

Photographic documentation will be collected with a digital camera as requirements are identified in investigation-specific governing documents, and at any other place the field sampling personnel determine necessary. Photographic documentation will be performed in general conformance the Project-Specific Procedure – 5: Photographic Documentation of Field Activities.

Digital photographs will be archived by EMR, until project closeout, at which time project management will determine a long-term electronic file storage system. File names will be in a format prescribed as detailed in investigation-specific SAPs.

Field logbook entries of digital photographs will be reviewed for content and correct completion during field logbook reviews. If photographic logs are found to be incorrectly completed, the reviewer will discuss the error with the author of the field logbook and necessary corrections will be made by the author.

7.0 Global Positioning System (GPS) Point Collection

Global positioning system (GPS) points are collected in accordance with Site GPS data collection guidelines. General procedures used for GPS point collection are discussed below:

- For each grab soil sample, a GPS point is collected directly above the sample location.
- For composite soil samples, a GPS point is collected at the approximate center of each sample area. In the case of an irregularly shaped sample area or sample area that is non-continuous, a GPS point is collected at the center of the largest continuous sample area.
- A single GPS point is collected for each soil sample taken at different depths from the same X, Y coordinates. Each sample collected is assigned the same location ID number.
- For outdoor air samples, a GPS point is collected at each unique sample location unless otherwise stated in the investigation-specific SAP. All subsequent air samples taken at that location will be assigned the sample location ID number and X, Y coordinate.

GPS data are not collected for the following types of samples:

- **Soil Duplicates.** The same location ID number is used for the parent and the field duplicate samples, resulting in the same X, Y coordinates.
- **Collocated Ar.** The same location ID number is used for the original and the co-located sample resulting in the same X, Y coordinates.
- **Personal Air Samples.** The locations for these samples are the same coordinates assigned to the property or building where the samples were collected.

To ensure proper collection of GPS data the following criteria have been established at the Site for data with accuracy to plus or minus (±) 1 meter:

- The operator of the GPS unit must be standing at the sample location before the data collection begins.
- Once the unit begins collection of location data, the operator must remain standing at the sample location until the minimum required data points have been collected.
- A minimum of number of data points must be collected at each X, Y coordinate.

The conversion of GPS points to usable Geographic Information System (GIS) formatted X, Y coordinates is described in this section and CDM-LIBBY-09 (attached).

After the conversion from GPS points to GIS files, 100% of the data is checked to ensure data entry errors, such as too many spaces, into the data dictionary are rectified prior to loading in the Geodatabase. In addition 10% of all GPS points collected per each investigation will be randomly chosen for additional field verification of accuracy. If a GPS point is found to be inaccurate by more than 6 feet a new GPS point will be collected and submitted to replace the original point. If this first tier of verification finds 5% or more of the points to be inaccurate, 10% of all points collected on the same day will also be verified and corrected if necessary. If the second tier of verification finds 50% or more points require correction all points collected on the same day will be verified and corrected.

8.0 Equipment Calibration

Prior to sample collection each air sampling pump is calibrated to a primary or secondary calibration standard to the desired flow rate. The primary calibration standard used at the Site is a Bios DryCal® DC-Lite (see operation manual).

Calibration of Rotameter to Primary Standard

Rotameters, the secondary calibration standard used at the Site for day-to-day calibrations, are calibrated to the DryCal® on a quarterly basis using the following procedures:

1. Obtain the actual temperature and pressure in Libby, MT from the local National Oceanic and Atmospheric Administration (NOAA) weather station identified as LBBM8. Record actual temperature and pressure in the fields provided on the Precision Rotameter Calibration Data Sheet.

2. Set up the calibration train with the sampling pump, rotameter, and primary flow meter.

3. Assemble the base of the flow meter with the screw provided, tighten in place, and mounted within 6° of vertical.

4. Turn the DryCal® DC-Lite and sampling pump on.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Calibrate rotameter to desired ball reading, as read from the middle of the flow ball, with a sampling pump and sample cassette in-line. Cassette must be the same type and from the same manufacture's lot of sample cassettes that will be used for sampling. Record value in the Ball Reading column on the rotameter calibration data sheet.

7. Check adjusted flow rate of sample pump to the DryCal® DC-Lite flow calibrator primary flow standard. Ten repetitive flow measurements will be averaged and that result recorded in the flow rate column for the selected interval.

8. Repeat this process at 10 intervals over the range of the precision rotameter.

9. Input data into rotameter calculation sheet to generate the corrected flow rate.

Calibration of Sampling Pump with a Rotameter

Prior to sample collection, each sampling pump will be calibrated with a rotameter that has been calibrated to the primary flow standard, DryCal® DC-Lite. The procedures used for sampling pump calibration with a rotameter are as follows:

1. Set up the calibration train using a rotameter, sampling pump, and a representative sample cassette. The sample cassette to be used for sampling is installed between the pump and the calibrator.

2. To set up the calibration train, attach one end of tubing to the sample cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the sample cassette cap to the rotameter.

3. Assemble the base of the flow meter with the screw provided and tighten in place and mounted within 6° of vertical.

4. Turn the sampling pump on.

5. Turn the flow adjust screw or knob until the middle of the float ball on the rotameter is lined up with the pre-calibrated flow rate value.

To prevent potential cross-contamination, each rotameter used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag. The cap used at the end of the rotameter tubing will be replaced each morning after it is used.

Flow Rate Verifications

If the duration of a sampling event is longer than 4 hours, the flow rate will be checked at least at the midpoint of the sample collection period. Flow rate verifications can also be implemented on a more frequent basis as needed.

At each flow rate verification, flow rates will be adjusted back to the target rate by adjustment of the flow adjust screw or knob. Adjustment of flow rates during flow rate verifications will be performed as described below and as the last action before leaving a sampling location anytime the sampling pump is moved. Should the flow rate change by more than 10 percent, the following procedure will be used to make the adjustment:

1. Set up the calibration train using a rotameter, sampling pump and a representative sample cassette. The sample cassette to be used for sampling is installed between the pump and the calibrator.

2. To set up the calibration train, attach one end of tubing to the sample cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the sample cassette cap to the rotameter.

3. Assemble the base of the flow meter with the screw provided and tighten in place and mounted within 6° of vertical.

4. Record the observed flow rate and time of observation.

5. Adjust the flow rate, if necessary, to the target flow.

6. Record the new flow rate and the time flow adjustment was completed as the same minute recorded in step 2.

Should the flow rate change by more than 10 percent during the sampling period, the average of the pre- and post- flow rates will be used to calculate the sample volume during each check period. The individual volumes collected between these check periods will be added together to calculate the total sample volume collected.

If at any time the measurement indicates that the flow-rate has decreased by more than 30 percent or increased by more than 50 percent, the sampling shall be terminated, and further action will be taken as detailed in the OU6 Rail Maintenance SAP.

9.0 Field Equipment Maintenance

All field equipment is maintained in accordance with manufacturer's specifications and the Project-Specific Procedure - 6: Control of Measurement and Test Equipment. Equipment required to make field acceptable repairs will be available. This will ensure timely repair of any out-of-order equipment. When a piece of equipment is found to be operating incorrectly, the piece of equipment will be labeled out-of-order and placed in a separate area from the rest of the sampling equipment. The person who identified the equipment as out-of-order will notify the EMR staff member overseeing the investigation activities. It is the responsibility of

that person to facilitate repair of the out-of-order equipment. This may include having appropriately trained field team members complete the repair or shipment to the manufacture.

10.0 Handling Investigation Derived Waste (IDW)

Any disposable equipment or other IDW will be handled in general conformance with the Project-Specific Procedure – 3: Guide to Handling of Investigation-Derived Waste.

During periodic reviews, IDW handling procedures will be evaluated. If field teams are observed not to be following the handling procedures, the field teams will be re-instructed on correct handling procedures.

11.0 Field Sample Custody and Documentation

Chain-of-custody (COC) procedures will follow the requirements as stated in the Project-Specific Procedure - 1: Sample Custody. The COC is used as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A complete COC record is required to accompany each shipment of samples.

At the end of each day, all samples will be relinquished to the sample coordinator by the sampling team following COC procedures, and an entry will be made into the field logbook indicating the time samples were relinquished and the sample coordinator who received the samples. The sample coordinator will follow COC procedures to ensure proper sample custody between acceptance of the sample from the field teams to delivery or shipment to the laboratory.

12.0 Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with the Project-Specific Procedure - 2 Packaging and Shipping of Environmental Samples. For dust and air samples, a custody seal will be placed so that both end caps of the sampling cassette are covered by the seal but will not obstruct the sample index identification (ID). For soil samples, custody seals are not typically required for individual (unless otherwise stated in the OU6 SAP); rather seals will be placed over at least two sides of the shipping cooler and then secured by tape if samples are released to a non-sampler. The sample coordinator is responsible for performing a final check of the contents of a shipment with the COC before custody seals are placed on the shipping container.

The sample coordinator will be responsible for shipment of samples. For soil samples, samples will be shipped by a delivery service to CDM prior to the designated laboratory (EMSL). Vermiculite, shredded paper, or expanded polystyrene are not acceptable as packing material on the Libby Project. Plastic bubble wrap is an acceptable packing material.

13.0 Modification Forms

All deviations from investigation-specific guidance documents will be recorded on the Libby Asbestos Project Record of Modification Form, as presented in Appendix E of the OU6 Rail Maintenance SAP. The Record of Modification Form will be used to document all permanent and temporary changes to procedures contained in guidance documents governing investigation work. In addition, the Record of Modification Form will be used to document any information of interest. As modifications to governing documents are implemented, EMR field team leader (FTL) will communicate the changes to the field teams conducting activities associated with the

modification. If the USEPA oversight team (CDM) determines the need, revised governing documents may be issued to incorporate modifications.

Record of Modification Forms are completed by the FTL overseeing the investigation/activity, or by technical staff as assigned. Once a form is prepared a technical review is completed by EMR, and then reviewed and approved by the CDM project leader or designate.

A modification tracking log is maintained by the EMR data manager. The log briefly describes the modification being documented, as well as form author, the EMR and CDM reviewers, and date of approval. Each completed Record of Modifications Form is assigned a unique for number and is maintained by EMR's data manager.

14.0 Laboratory Analysis and Requirements - Related QA/QC Procedures

This section summarizes the Quality Assurance and Quality Control (QA/QC) objectives and procedures used in the laboratory testing program. The following section discusses the more common types of analytical methods used for investigation samples, the project-specific SOPs and method deviations to commercial testing standards, and the QA/QC procedures associated with each project method. This is followed by discussion on general laboratory QA practices used and audited by the project management team.

14.1 Analytical Methods – QA/QC Requirements

The typical analytical methods used to analyze samples collected that support investigation activities at the Libby Site are discussed in this section. The specific analytical methods used for each investigation activity will be listed in investigation specific SAPs.

The following sections outline the methods and any established project-specific QA/QC requirements. The project-specific methods should be consulted for detailed descriptions of method required QA/QC measures.

14.2 TEM Methods

Transmission electron microscopy (TEM) methods are more complex than phase contrast microscopy (PCM) and polarized light microscopy (PLM) and require the use of a more sophisticated analytical instrument that can distinguish between asbestos and non-asbestos fibers and asbestos types. TEM methods can be used on dust, air, and solid media.

14.2.1 TEM Project-Specific SOPs and Method Deviations

TEM methods used on the Libby Project have been updated by the following project specific SOP:

- USEPA-LIBBY-08 – *Indirect Preparation for TEM Analysis* (CDM 2005). This SOP provides a standardized procedure for the indirect preparation of a sample to minimize the loss of analytical sensitivity.
- USEPA-LIBBY-09 – *SOP for TEM Data Review and Data Entry Verification* (CDM 2005). This SOP describes the steps for selection of TEM analysis for review and verification, review of the laboratory bench sheets, and verification of the transfer of results from the bench sheets into the project database (Libby2). As processes used for TEM data review and verification are revised, the SOP will be updated and reissued.

TEM methods have been further modified and clarified by the following permanent project-specific laboratory modifications (CDM 2005).

- LB-000019 – Clarifies the bench sheet recording format for grid openings in which no countable structures are recorded.
- LB-000028 – Clarifies the action on more complete TEM reanalysis data such as when some of the originally read grid openings in a sample selected for reanalysis become unreadable.
- LB-000029 / LB-000029a/ LB-000029b – Standardizes the frequency of analysis and procedures for interpretation of the results for laboratory-based QC samples for TEM analysis.
- LB-000030 – Documents a requirement to the laboratory analyst to include sketches of all asbestos structures observed up to a maximum of 50 structures per sample. Sketches should be sufficiently detailed to include an indication of structure appearance, morphology, and orientation relative to any nearby landmarks, if present.
- LB-000031/LB-000031a – Clarify and expand the TEM structure measurement and counting as expressed in the American Society of Testing and Materials (ASTM) 5755 methodology.
- LB-000045 – Cross references the investigation-specific TEM counting rules.
- LB-000053 – Provides a reference table by sample prefix to indicate what analysis method(s) should be used.

As new TEM method modifications and clarifications are needed, a new Laboratory Modification document number will be assigned and all subcontracted laboratories will be involved in the development and review process of the final document before it becomes available for signature and subsequent distribution to all assigned document holders of the *Modifications to Laboratory Activities* (CDM, 2003c). The laboratories will be advised of the effective date of the laboratory modification so that the change(s) can begin immediately after its final draft has been approved by USEPA and Volpe. Electronic and hard copy distribution of the approved laboratory modification to each document holder will complete the modification cycle.

14.2.1.1 QA/QC Procedures

QA/QC at the laboratory level for TEM samples is maintained by following the requirements specified in the method. Laboratory-based QC for TEM is based on satisfactory performance covered by the requirements in NIST's National Voluntary Laboratory Accreditation Program (NVLAP). This laboratory accreditation signifies the competency of a laboratory to provide testing services. The third-party accreditation complies with the standards published by International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC), specifically ISO/IEC 17025.

The NVLAP program reviews management and technical requirements pertaining to quality systems, personnel, facilities, test and calibration methods, equipment, measurement traceability, sampling, handling of test and calibration items, and reporting (NIST, 2006). In addition, laboratories are required to participate in one proficiency testing activity per accreditation on a minimum frequency interval of every 4 years. Unsatisfactory performance due to non-participation in regularly scheduled proficiency test rounds or unresolved technical nonconformities can subject a laboratory to denial or suspension of their accreditation and subsequent suspension on the Libby project. Current copies of NVLAP certifications from the contracted asbestos testing laboratories are submitted to the CDM Laboratory Coordinator for the contract files.

Environmental monitoring is performed on a monthly basis and consists of the TEM analysis of air and dust samples. Copies of these results are submitted directly to BNSF and USEPA (or their representative).

Laboratories may also conduct annual internal QC audits that are performed by a senior staff member, QA director, or representative from their corporate office. This audit may cover such mechanical aspects as microscope alignment, resolution, and a field area determination. QC aspects such as the successful reading

of a known reference slide and re-analysis of samples are also covered. The audit typically extends to document control, report generation and review, and data QA. Copies of these audits are maintained at the laboratory and at corporate headquarters, as applicable.

Additional project specific QA/QC requirements for this TEM method are discussed below.

14.2.1.2 Project Requirements

Laboratory modification forms LB-000029, LB-000029a, and LB-000029b provide guidelines to standardize the frequency of QC sample for all TEM analyses and are summarized below:

QC Sample Type	Frequency (percent
Laboratory Blank	4
Recount Same	1
Recount Different	2.5
Verified Analysis	1
Repreparation	1
Interlaboratory	0.5
Total	10

When indirect preparation methods are used, 1 method blank per COC will be added to the above QC sample list. In addition, 1 drying blank per COC may be added, as appropriate (LB-000055).

Lab Blanks (LB) – This is a TEM grid that is prepared from a new, unused filter at the laboratory and is analyzed using the same procedure as used for field samples. There shall be no asbestos structures of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples is started. Detection of any asbestos on laboratory blanks should be communicated to the EMR laboratory coordinator immediately.

Re-Analysis Samples. Re-analysis samples include recount same, recount different, verified analysis, and interlaboratory QA samples.

- **Recount Same (RS).** This is a field sample TEM grid that is re-examined by the same microscopist who performed the initial examination. The microscopist examines only the same grid openings that were counted in the original examination.
- **Recount Different (RD).** This is a field sample TEM grid that is re-examined by a different microscopist than who performed the initial examination. The microscopist examines only the same grid openings that were counted in the original examination.
- **Verified Analysis (VA).** This is similar to a Recount Different but has different requirements with regard to documentation. A verified analysis must be recorded in accord with the protocol provided in National Institute of Standards and Technology (NIST) 1994.

Whenever a recount occurs in which one or more of the acceptance criteria are not met, the sample will under verified analysis described by NIST 1994, and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Each laboratory should notify the EMR laboratory coordinator of any significant exceptions and corrective actions and document all exceptions on a job-specific (temporary) laboratory modification form. The EMR laboratory coordinator will ensure that appropriate Volpe and USEPA representatives are notified for their review, comment, and approval of the job-specific laboratory modification.

Repreparation. This is a grid that is prepared from a new aliquot of the same field sample filter used to prepare the original grid. Typically this is done at the same laboratory that performed the original analysis. A different laboratory can also prepare grids from a new piece of filter upon approval request through the EMR laboratory coordinator. If the re-preparation is done within the same laboratory, the repreparation and reanalysis should be done by a different analyst than who analyzed the original grid, whenever possible.

Repreparation samples will be evaluated by comparing the total counts of the original and the repreparation samples. Acceptance criteria for this QC sample type are that the results must not be statistically different from each other at the 90 percent confidence interval, using the procedure documented in Attachment 1 to LB-000029b. If this acceptance criterion is not met, a senior analyst shall determine the basis for the discordant results, and if it is judged that the discordance is related to laboratory procedures, the laboratory shall then take appropriate corrective action.

Interlaboratory (Interlab). This is a reprepped field sample that has been identified through the EMR laboratory coordinator. The originating laboratory will receive direction to prepare 3 TEM grids of which one will be archived. After the analyst reads 5 grid openings using the TEM counting rules specified by the EMR laboratory coordinator, the 2 TEM grids will be shipped to the interlaboratory for analysis under COC and direction on which grid openings should be read. The TEM grids read at the originating laboratory will be designated as RP whereas the interlaboratory will identify the Lab QC Type as “Interlab.”

Drying Blanks (DB). This is a TEM grid that is prepared from a new, unused filter at the laboratory and is analyzed using the same procedure as used for the field samples, complying with LB-000055. When no asbestos is detected on the DB TEM grid, the oven drying process is determined to be satisfactory and no cross-contamination is suspect. Should any asbestos structures be found on the DB TEM grid, the laboratory shall immediately investigate the source of the contamination and take immediate steps to thoroughly clean the oven before any other field samples are placed into the oven. Detection of any asbestos on DBs must be immediately communicated to the EMR laboratory coordinator who will provide the laboratory with further direction. The analytical data for the field samples associated with the contaminated DB will need to be evaluated to determine if analysis on the field samples should proceed and if the data set will require a bias indicator tag.

Method Blanks (MB). This is a TEM grid that is prepared from a new, unused filter at the laboratory and is analyzed using the same indirect sample preparation procedure as that used on the associated field samples. When no asbestos is detected on the MB TEM grid, the sample preparation is determined to be satisfactory and no cross-contamination is suspect. When any asbestos structures are found on the MB TEM grid, the laboratory must immediately notify the EMR laboratory coordinator to receive further direction. The analytical data for the field samples and LB associated with the MB will need to be evaluated to determine if analysis on the field samples should proceed and if the data set will require a bias indicator tag.

Program-wide goals for QC acceptance criteria are documented in LB-000029 and acceptance criteria for re-analysis samples are detailed in LB-000029b. These laboratory modifications can be revised (updated) as acceptance criteria are re-defined.

14.2.2 ISO 10312:1995(E)

14.2.2.1 Method Description

The ISO Ambient air – Determination of Asbestos fibers – Direct-transfer Transmission Electron Microscopy Method was issued in 1995 and is referenced using the designation ISO 10312:1995(E) (CDM 2003a, Section 3). This TEM method is suitable for use to determine the concentration of asbestos structures in both indoor and outdoor ambient air environments and includes measurement of asbestos structure lengths, widths, and aspect ratios. The method can be used to determine the type(s) of asbestos fibers present but cannot be used to discriminate between individual fibers of the asbestos and non-asbestos analogues of the same amphibole mineral.

14.2.2.2 Project-Specific SOPs and Method Deviations

The ISO 10312:1995(E) method has been further modified and clarified by the following permanent project-specific laboratory modifications (CDM 2005).

- LB-000016 – Clarifies the structure counting procedures and recording definitions used to populate the project specific electronic data deliverable (EDD) and documents the method modifications concerning recording definitions, stopping rules, structure counting, the use of a grid rejection criteria of > 25 percent, the use of indirect preparation methods.
- LB-000016a – Provides a counting rule clarification.

As additional modifications are prepared for this method, the Laboratory Modification document will be revised and all laboratories will be verbally informed of the content of the modifications though regularly scheduled laboratory conference calls.

14.2.2.3 QA/QC Procedures

No additional project specific QA/QC requirements for this TEM analytical method have been established.

14.3 PLM Methods

Specific requirements for each PLM method are described in the following sections. For all PLM methods the following are project-specific requirements that ensure QA/QC of PLM analysis:

- Each day the refractive index liquids are check for asbestos contamination;
- Verification of the refractive indices of the refractive index liquids once per week;
- Verification of microscope adjustments prior to each sample set;
- Reference sample or Laboratory Control Sample results by having a analyst check the United States Geological Survey (USGS) standards daily;
- Preparation of replicate slides;
- Standard Raw data forms with handwritten results (as presented in the EDD spreadsheet);
- Standard case narrative information;
- Laboratory duplicate results (if performed); and
- Results reported in the USEPA 4-tier method (per SOP Syracuse Research Corporation SRC-LIBBY-03).

14.3.1 SRC-LIBBY-01 (PLM-Grav)

14.3.1.1 Method Description

SRC-LIBBY-01. *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy* (PLM), (CDM 2005, Section 11) was developed in 2002 and contains elements from NIOSH 9002 Issue 2 and USEPA Method 600/R-93/116. This SOP provides a screening method to examine the coarse fraction of a sieved sample with particle size greater than ¼ inch for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos by PLM method NIOSH 9002, Issue 2. The method is suitable for use on soil and other similar soil-like media to quantify all types of asbestos fibers including chrysotile and amphiboles like those characteristic of the Libby site.

14.3.1.2 Project-Specific SOPs and Method Deviations

As a screening method for the coarse fraction of sieved soils using ISSI-Libby-08, this method has no project-specific SOPs or method deviations.

14.3.1.3 QA/QC Procedures

QA/QC at the laboratory level for samples analyzed via this method is maintained by laboratory-based QC for the National Institute of Occupational Safety and Health (NIOSH) 9002 PLM method requirements specified by NIST's NVLAP. This includes daily evaluation of various blanks to check for contamination.

No additional project specific QA/QC requirements have been established for this analytical method including interlaboratory, blank, and CSF QC sample analysis.

14.3.2 SRC-LIBBY-03 (PLM-VE)

14.3.2.1 Method Description

SRC-LIBBY-03. *Analysis of Asbestos Fibers in Soil by PLM, Revision 1* (CDM 2005 Section 9) was developed in 2003 and is based on NIOSH 9002 Issue 2, USEPA Method 600/R-93/116, and California Air Resource Board (CARB) Method 435. This semi-quantitative method provides a standard approach to quantify all types of asbestos fibers including chrysotile and amphiboles like those characteristic of the Libby site using visual estimation or point counting techniques. For the visual estimation technique, guidance is provided on the classification of asbestos mineral type and the estimation of Libby amphibole (LA) mass percent using site-specific reference materials following project specified "bin" categories. For point counting, the SOP provides guidance on how to estimate mass percent of LA present. The method is suitable for use on the fine fraction of soil and other similar soil-like media that has been processed using the ISSI-Libby-01 SOP.

14.3.2.2 Project-Specific SOPs and Method Deviations

This method is further modified by the following project specific permanent laboratory modifications detailed in CDM 2003b.

- LB-000024 / LB-000024a - Provides guidance for a more precise quantification of the low levels of LA found in soil samples collected at the Site

As additional modifications are prepared for this method, CDM 2003b will be revised and all laboratories will be verbally informed of the content of the modifications though regularly scheduled laboratory conference calls.

14.3.2.3 QA/QC Procedures

QA/QC at the laboratory level for samples analyzed via this method is maintained by laboratory and method required QA/QC procedures. Laboratory-based QC for PLM is based on the requirements specified by NIST's

NVLAP. This includes daily evaluation of various blanks to check for contamination. Overall QC analysis is at a rate of at least 10 percent, including inter- and intra-analyst re-analyses, inter-lab and blank analysis.

Section 7.1 of SRC-Libby-03 defines method precision and accuracy as the frequency with which samples are assigned to the correct “bins”. Bin assignment accuracy is being collected; however, performance criteria have not yet been established.

Section 7.2 of SRC-Libby-03 contains method proficiency requirements associated with an annual blind set of performance evaluation (PE) samples prepared by USGS that will be shipped to each laboratory for analysis. At present, the acceptance criteria for these samples have not been established.

Additional project specific QA/QC requirements for this analytical method have not been established.

14.4 Analytical Sensitivity

Information regarding analytical sensitivity is discussed in this section. The following discussions are general and present factors that affect method sensitivities. The analytical sensitivities (AS) required or each investigation will vary and the investigation-specific SAPs will provide details on the analytical sensitivities required to meet investigation-specific goals.

14.4.1 TEM Methods

14.4.1.1 ISO 10312:1995(E)

Direct Method

ISO 10312:1995(E) describes analytical sensitivity as the structure concentration corresponding to the detection of one structure in the analysis. The analytical sensitivity for air samples is dependent on the volume of air sampled, the number of grid openings observed, the size of the grid openings, and the effective area of the sampling filter. The AS for this method is determined by the following equation:

$$AS = \frac{A_t}{k * A_s * V}$$

Where:

AS = analytical sensitivity; structures/liter

A_t = active area of sample collection filter; millimeters squared (mm^2)

k = number of grid openings examined

A_s = mean area of grid openings examined; mm^2

V = volume of air sampled; liters

As the AS is required to be lower, the number of grid openings examined or the volume of air sampled should be increased. Flow rates and sampling durations should be chosen using the highest flow rate and longest duration possible that can meet project objectives so that filter loading does not exceed 10 micrograms per centimeter squared (ug/cm^2) of filter surface.

Flow rates should at no time exceed 15 liters per minute, flow rates higher than this can result in damage to the sample filter. Also, flow rates should at no time should be below 1 liters per minute, flow rates lower than this result in linear velocities below those required for analysis by ISO 10312:1995(E).

Indirect Method

As described in section 1.4 of the method, the analytical sensitivity and be lowered by sampling larger volumes of air and by increasing the grid openings analyzed. This section further describes that the lowest achievable AS is determined by the total suspended particulate concentration remaining after the ashing and aqueous dispersal steps, and this depends on the chemical nature of the suspended particulate.

14.4.2 PLM Methods

14.4.2.1 SRC-LIBBY-03 (PLM-VE)

As described in SRC-LIBBY-03, PLM analysis of soil samples can be performed by visual estimation or a point count approach.

Visual estimation (VE) uses a semi-quantitative approach that requires the analysts to estimate the area fraction of the total material present in a field of view that consists of asbestos material. All visual estimates of Libby amphibole contents are performed using a set of site-specific reference materials. Using these reference materials soil results are reported as non-detect (ND), Trace (Tr), less than 1 percent, or quantification of the mass percent when it is equal to or greater than 1 percent. SRC-LIBBY-03 provides specific guidance on reporting PLM-VE results.

The point count approach is performed in general accordance with the descriptions provided in USEPA/600/R-93/116 (CDM 2003a, Section 17) and CARB Method 435. When the point count method is required the number of points to be counted will be specified on the COC. A point count of 400 points equates to an analytical sensitivity of 0.25 percent, and a point count of 1,000 points equates to an analytical sensitivity of 0.1 percent.

14.4.2.2 SRC-LIBBY-01 (PLM-Grav)

This method is based on USEPA 600-R-93/116 (CDM 2003a, Section 17). This method describes the analytical sensitivity that can be obtained by this method as 1 percent by weight. The sensitivity can be affected by the homogeneity of the sample, the accuracy of the weight measurements obtained at the laboratory, and the effectiveness of the sample reduction and filtering procedures.

14.5 Holding Times

Technical holding times are storage times allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed. No preservation requirements or holding times are established for air, dust, or soil samples collected for asbestos analysis.

The only exception to these holding times is related to filters and soil samples that are wet. Because moisture can promote the growth of mold, these samples must be refrigerated if delivery to a laboratory for drying cannot be completed within 24 hours.

14.6 Analytical Results Turnaround Times

The analytical results turn around times required for each investigation will vary. When expedited turn around times are required (less than 2 weeks) for any investigation sample, the EMR laboratory coordinator will be informed as soon as possible during the investigation planning phase. The EMR laboratory coordinator will inform the sample coordination staff on the laboratory that is to be utilized to meet required turn around times. Investigation-specific SAPs will include information regarding the expected turn around time for results related to each investigation.

15.0 Laboratory Custody Procedures and Documentation

Laboratory custody procedures are provided in the laboratories' QA management plan. The basic laboratory sample custody process is as described herein. Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipping and the individual samples. This inspection will include verifying sample integrity. The accompanying COC records will be cross-referenced with all of the samples in the shipment. The laboratory sample custodian will sign the COC records and maintain a copy for their project files; the original COC will be appended to the hard copy data report that is sent to EMR's laboratory coordinator. Next, the sample custodian may continue the COC record process by assigning a unique laboratory number to each sample on receipt. This number, if assigned, will identify the sample through all further handling at the laboratory. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, and data reporting.

16.0 Documentation and Records

16.1 Analytical Data Reports

Data reports for all samples will be submitted to the EMR laboratory coordinator and include a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed COC forms, analytical data summary report pages, a QC package, and raw data, where applicable. Raw data is to consist of instrument preparation logs, instrument printouts, and QC sample results including, instrument maintenance records, COC check in and tracking, raw data instrument print outs of sample results, analysis run logs, and sample preparation logs. All original data reports will be the EMR laboratory coordinator and stored in project files. The laboratory also will provide an electronic copy of the data to the laboratory coordinator and others as directed by the EMR laboratory coordinator.

16.2 Laboratory Data Entry Spreadsheets

Standardized data entry spreadsheets (electronic data deliverables [EDDs]) were developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. In general, a unique data entry MSExcel workbook template was developed for each type of analytical method (TEM, PLM). Since the beginning of the Libby project, the EDD has evolved to better accommodate the present and future needs of data handling, retrieval, and interpretation. An on-going refinement of the EDD continues based on laboratory and data user input.

The EDD template contains a variety of built-in QC functions that improve accuracy of data entry and help maintain data integrity. For example, data entry forms utilize drop-down menus whenever possible to standardize data inputs and prevent transcription errors. In addition, many data input cells are coded to highlight omissions, apparent inconsistencies, or unexpected values so that data entry personnel can check and correct any errors before submittal of the EDD. The spreadsheet workbook also performs automatic computations of sensitivity, dilution factors, and concentration, thus reducing the likelihood of analyst calculation errors. The EDD was designed to directly upload data into the project database, avoiding any additional data entry requirements.

16.3 Modification Forms

All deviations from project specific and method guidance documents will be recorded on the Libby Asbestos Project Record of Modification Form to Laboratory Activities. The Record of Modification Form will be used to document all permanent and temporary changes to analytical procedures. In addition, the Record of Modification Form will be used to document any information of interest as requested by USEPA project

management. As modifications are implemented, the laboratory coordinator will communicate the changes to the project laboratories.

Record of Modification Forms are completed by case manager assigned by each laboratory to the Libby project or their designate. Once a form is completed a technical review is completed by the laboratory and the EMR laboratory coordinator, and then reviewed and approved by the USEPA project leader or designate.

A record is kept to track the person each form was completed by and a brief description of the modification documented on each form. Each completed Record of Modifications Form is assigned a unique identification number and maintained by the EMR laboratory coordinator.

17.0 Data Recording, Management, and Reporting

This section describes data recording and management and also provides information related to data reporting.

17.1 Geographic Information System (GIS) Data

GPS data collected in the field is converted to usable GIS format using the general processes described in this section and CDM-LIBBY-09 (CDM 2007, Section 14).

After the conversion from GPS points to GIS files, 100 percent of the data is checked to ensure data entry errors, such as too many spaces, into the data dictionary are rectified prior to loading in the Geodatabase. In addition 10 percent of all GPS points collected per each investigation will be randomly chosen for additional field verification of accuracy. If a GPS point is found to be inaccurate by more than 6 feet a new GPS point will be collected and submitted to replace the original point. If this first tier of verification finds 5 percent or more of the points to be inaccurate, 10 percent of all points collected on the same day will also be verified and corrected if necessary. If the second tier of verification finds 50 percent or more points require correction all points collected on the same day will be verified and corrected.

17.2 Data Recording and Management

Data entry will be managed by EMR. Data entry is primarily an automated process. However, if hand-entry of data is required, EMR's data entry team will perform a 100 percent check of all data entered and correct any errors found in the data entered.

17.3 Data Reporting

As investigation-specific activities are completed, data summary reports will be generated by EMR. Summary reports will present any deviations implemented and the potential impacts of the deviations on data quality.

When data are presented associated with limitations identified though the QA/QC procedures detailed in this QAPP, all summary reports and tables of data will duly note all QA/QC deficiencies observed and provide a discussion on the potential impacts of data quality

18.0 References

CDM. 2003a. Libby Asbestos Project Analytical Guidance Documents. Volume 1. August.

CDM. 2003b. Final Draft Pre-Design Inspection Action Work Plan. November.

CDM. 2003c. Modifications to Laboratory Activities. 1st Revised December 23, 2003 with ongoing updates.

CDM. 2004. Close Support Facility Soil Preparation Plan, Revision 1. March.

CDM. 2005. Libby Asbestos Project Analytical Guidance Documents. Volume 2. December with ongoing updates.

CDM. 2007. Libby Asbestos Projec Field Standard Operating Procedures. February.

NIST. 1994. Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by TEM – Version 2.0. March.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 1
Sample Custody

1.0 Objective

Because of the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in project files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

Note: Sample custody documentation requirements vary with the specific EPA region or client. This procedure is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements shall be presented in the project-specific quality assurance (QA) project plan or sampling and analysis plan.

2.0 Background

2.1 Definitions

Sample - A sample is material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody - A sample is under custody if:

1. It is in your possession
2. It is in your view, after being in your possession
3. It was in your possession and you locked it up
4. It is in a designated secure area

Chain-of-Custody Record - A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

Custody Seal - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

Sample Label - A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag - A sample tag is attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

3.0 General Responsibilities

Sampler - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork.

Field Sample Custodian - The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or sampling and analysis plan.

4.0 Required Supplies

- Chain-of-custody records (applicable client or CDM forms)
- Custody seals
- Sample labels and/or tags
- Clear tape
- Printer
- Printer paper

5.0 Procedures

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations before the actual fieldwork. As few people as possible shall handle samples.

2. Complete sample labels or tags for each sample using waterproof ink.

3. Maintain personal custody of the samples (in your possession) at all times until custody is transferred for sample shipment or directly to the analytical laboratory.

Transfer of Custody and Shipment

1. Complete a chain-of-custody record for all samples. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.

    - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the chain-of-custody record.

    - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.

    - If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

Note: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign, and date as the relinquisher. The individual sampler(s) must sign in the appropriate block, but does (do) not need to sign and date as a relinquisher.

2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment must be accompanied by a separate chain-of-custody record. If a shipment consists of multiple coolers, a chain-of-custody record shall be filled out for each cooler documenting only samples contained in that particular cooler.

3. The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation. The shipping number from the freight bill shall be recorded on the applicable chain-of-custody record and field logbook in accordance with the Project-Specific Procedure – 2: Field Logbook Content and Control.

Procedure for Completing Typical Chain-of-Custody Record

The following procedure is to be used to fill out typical chain-of-custody record, the custody record shall be filled out in its entirety.

1. Record project number.

2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the "Remarks" box).

3. Record the name and address of the laboratory to which samples are being shipped.

4. Enter the project name/location or code number.

5. Record overnight courier's airbill number.

6. Record sample location number.

7. Record sample number.

8. Note preservatives added to the sample.

9. Note media type (matrix) of the sample.

10. Note sample type (grab or composite).

11. Enter date of sample collection.

12. Enter time of sample collection in military time.

13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.

14. List parameters for analysis and the number of containers submitted for each analysis.

15. Enter appropriate designation for laboratory quality control (e.g., matrix spike/matrix spike duplicate [MS/MSD], matrix spike/duplicate [MS/D]), or other remarks (e.g., sample depth).

16. Sign the chain-of-custody record(s). All samplers must sign each record.

17. If sample tags are used, record the sample tag number in the "Remarks" area.

18. The originator checks information entered on the form and then signs the "Relinquished by" area, prints his/her name, and enters the current date and time (military).

19. Send two copies with the samples to the laboratory; retain the third copy for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.

20. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received by" area and keeps the copy.

5.2 Sample Labels and Tags

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for projects.

1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows:

- Record the project code (i.e., project or task number).
- Enter the station number (sample number or EPA CLP identification number) if applicable.
- Record the date to indicate the month, day, and year of sample collection.
- Enter the time (military) of sample collection.
- Place a check to indicate composite or grab sample.
- Record the station (sample) location.
- Sign in the space provided.
- Indicate if a preservative was added.
- Identify the analytical parameters for which the sample is to be analyzed.
- Place or write additional relevant information where appropriate.

2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.

3. Securely attach sample tags to the sample bottle. On 2.27 liter (80 oz.) amber bottles, the tag string may be looped through the ring-style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted, and relooped around the neck until the slack in the string is removed.

4. Double-check that the information recorded on the sample tag is consistent with the information recorded on the chain-of-custody record.

5.3 Custody Seals

Two custody seals must be placed on opposite corners of all shipping containers (e.g., cooler) before shipment. The seals shall be signed and dated by the shipper.

Custody seals may also be required to be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction.

5.4 Sample Shipping

The procedure Packaging and Shipping Environmental Samples defines the requirements for packaging and shipping environmental samples.

6.0 Restrictions/Limitations

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure shall be followed.

7.0 References

U. S. Army Corps of Engineers. 2001. Requirements for the Preparation of Sampling and Analysis Plan, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. Revised March 1992. National Enforcement Investigations Center, Multi-Media Investigation Manual, EPA-330/9-89-003-R. p.85.

U. S. Environmental Protection Agency. Region IV. 1996. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Section 3.3. May.

U. S. Environmental Protection Agency. 2002. FORMS II Lite™ User's Guide, Version 5.1.

U. S. Environmental Protection Agency. 2002. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/240/R-02/009. Section 2.2.3. December.

U. S. Environmental Protection Agency. 2004. Contract Laboratory Program (CLP), Guidance for Field Samplers, EPA-540-R-00-003. Final. Section 3.2. August.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 2
Packaging and Shipping Environmental Samples

1.0 Objective

The objective of this procedure is to outline the requirements for the packaging and shipment of environmental samples. Additionally, Sections 2.0 through 7.0 outline requirements for the packaging and shipping of regulated environmental samples under the Department of Transportation (DOT) Hazardous Materials Regulations, the International Air Trans¬portation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations for shipment by air and applies only to domestic shipments. This procedure does not cover the requirements for packaging and shipment of equipment (including data loggers and self-contained breathing apparatus [SCBAs]) or bulk chemicals that are regulated under the DOT, IATA, and ICAO.

1.1 Packaging and Shipping of All Samples

This procedure applies to the packaging and shipping of all environmental samples.

1.2 Background

1.2.1 Definitions

Environmental Sample - An aliquot of air, water, plant material, sediment, or soil that represents the contaminant levels on a site. Samples of potential contaminant sources, like tanks, lagoons, or non-aqueous phase liquids are normally not “environmental” for this purpose. This procedure applies only to environmental samples that contain less than reportable quantities for any foreseeable hazardous constituents according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Custody Seal - A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the container (i.e., cooler) before offsite shipment. Custody seals are used to demonstrate that sample integrity has not been compromised during transportation from the field to the analytical laboratory.

Inside Container - The container, normally made of glass or plastic, that actually contacts the shipped material. Its purpose is to keep the sample from mixing with the ambient environment.

Outside Container - The container, normally made of metal or plastic, that the transporter contacts. Its purpose is to protect the inside container.

Secondary Containment - The outside container provides secondary containment if the inside container breaks (i.e., plastic overpackaging if liquid sample is collected in glass).

Excepted Quantity - Excepted quantities are limits to the mass or volume of a hazardous material in the inside and outside containers below which DOT, IATA, ICAO regulations do not apply. The excepted quantity limits are very low. Most regulated shipments will be made under limited quantity.

Limited Quantity - Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

Performance Testing - Performance testing is the required testing of outer packaging. These tests include drop and stacking tests.

Qualified Shipper - A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

1.2.2 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places the employer and the individual employee at risk of serious fines. The analytical holding times for the samples must not be exceeded. The samples shall be packed in time to be shipped for overnight delivery. Make arrangements with the laboratory before sending samples for weekend delivery.

1.3 Required Equipment

- Coolers with appropriate return address
- Bubble wrap (optional)
- Heavy-duty plastic garbage bags
- Ice
- Plastic zip-type bags, small and large
- Custody seals
- Clear tape
- Nylon reinforced strapping tape
- Completed chain-of-custody record or contract laboratory program (CLP) custody records, if applicable
- Duct tape
- Completed bill of lading
- A nonflammable material that is inert and absorbent
- “This End Up” and directional arrow labels

1.4 Packaging Environmental Samples

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of “environmental sample” and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the appropriate health and safety coordinator or the health and safety manager shall be observed.

2. Select a sturdy cooler in good repair. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler. Line the cooler with a large heavy-duty plastic garbage bag.

3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly.

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Binding the vials together with a rubber band on the outside of the bag, or separating them so that they do not contact each other, will reduce the risk of breakage. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with nonflammable absorbant or equivalent. Note: Trip blanks must be included in coolers containing VOA samples.

5. Place 2 to 4 inches of nonflammable absorbant into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Glass containers are less likely to break when packed vertically rather than horizontally.

6. While placing sample containers into the cooler, conduct an inventory of the contents of the shipping cooler against the chain-of-custody record. The chain-of-custody with the cooler shall reflect only those samples within the cooler.

7. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° Celsius (C) if the analytical method requires cooling. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

8. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.

9. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the opening of the cooler on opposite sides. The custody seals shall be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

10. The shipping container lid must be marked “THIS END UP” and arrow labels that indicate the proper upward position of the container shall be affixed to the cooler. A label containing the name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

2.0 References

U. S. Environmental Protection Agency. Region IV. February 1991 or current. Standard Operating Procedures and Quality Assurance Manual.

U. S. Environmental Protection Agency. 1996 or current. Sampler’s Guide to the Contract Laboratory Program, EPA/540/R-96/032.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements, 49 CFR 172.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. Shippers General Requirements for Shipments and Packagings, 49 CFR 173.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 3
Guide to Handling Investigation-Derived Waste (IDW)

1.0 Objective

This procedure presents guidance for the management of investigation-derived waste (IDW). The primary objectives for managing IDW during field activities include:

- Leaving the site in no worse condition than existed before field activities
- Removing wastes that pose an immediate threat to human health or the environment
- Proper handling of onsite wastes that do not require offsite disposal or extended aboveground containerization
- Complying with federal, state, local, and facility applicable or relevant and appropriate requirements (ARARs)
- Careful planning and coordination of IDW management options
- Minimizing the quantity of IDW

2.0 Background

2.1 Definitions

Hazardous Waste - Discarded material that is regulated listed waste, or waste that exhibits ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261.3 or state regulations.

Investigation-Derived Wastes - Discarded materials resulting from field activities such as sampling, surveying, drilling, excavations, and decontamination processes that, in present form, possess no inherent value or additional usefulness without treatment. Wastes may be solid, sludge, liquid, gaseous, or multiphase materials that may be classified as hazardous or nonhazardous.

Mixed Waste - Any material that has been classified as hazardous and radioactive.

Radioactive Wastes - Discarded materials that are contaminated with radioactive constituents with specific activities in concentrations greater than the latest regulatory criteria (i.e., 10 CFR 20).

Treatment, Storage, and Disposal Facility (TSDF) - Permitted facilities that accept hazardous waste shipments for further treatment, storage, and/or disposal. These facilities must be permitted by the U. S. Environmental Protection Agency (EPA) and appropriate state and local agencies.

2.2 Discussion

Field investigation activities result in the generation of waste materials that may be characterized as hazardous or radioactive waste. IDWs may include drilling muds, cuttings, and purge water from test pit and well installation; purge water, soil, and other materials from collection of samples; residues from testing of treatment technologies and pump and treat systems; personal protective equipment (PPE); solutions (aqueous or otherwise) used to decontaminate nondisposable protective clothing and equipment; and other wastes or supplies used in sampling and testing potentially hazardous or radiologically contaminated material.

Note: The client's representatives may not be aware of all potential contaminants. The management of IDW must comply with applicable regulatory requirements.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that all IDW procedures are conducted in accordance with this SOP. The site manager is also responsible for ensuring that handling of IDW is in accordance with site-specific requirements.

Project Manager - The project manager is responsible for identifying site-specific requirements for the disposal of IDW in accordance with federal, state, and/or facility requirements.

Field Crew Members - Field crew members are responsible for implementing this SOP and communicating any unusual or unplanned condition to the project manager's attention.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project specific quality assurance plan.

4.0 Required Equipment

Equipment required for IDW containment will vary according to site-specific/client requirements. Management decisions concerning the necessary equipment required shall consider: containment method, sampling, labeling, maneuvering, and storage (if applicable). Equipment must be onsite and inspected before commencing work.

4.1 IDW Containment Devices

The appropriate containment device (drums, tanks, etc.) will depend on site- or client-specific requirements and the ultimate disposition of the IDW. Typical IDW containment devices can include:

- Plastic sheeting (polyethylene) with a minimum thickness of 20 millimeters
- Department of Transportation (DOT)-approved steel containers

- Polyethylene or steel bulk storage tanks. Containment of IDW shall be segregated by waste type (i.e., solid or liquid, corrosive or flammable, etc.) and source location. Volume of the appropriate containment device shall be site-specific.

4.2 IDW Container Labeling

A “Waste Container” or “IDW Container” label or indelible marking shall be applied to each container. Labeling or marking requirements for onsite IDW not expected to be transported offsite are:

- Labels and markings that contain the following information: project name, generation date, location of waste origin, container identification number, sample number (if applicable), and contents (drill cuttings, purge water, PPE, etc.).

- Each label or marking will be applied to the upper one-third of the container at least twice, on opposite sides.

- Containers that are 5 gallons or less may only require one label or set of markings.

- Labels or markings will be positioned on a smooth part of the container. The label must not be affixed across container bungs, seams, ridges, or dents.

- Labels must be constructed of a weather-resistive material with markings made with a permanent marker or paint pen and capable of enduring the expected weather conditions. If markings are used, the color must be easily distinguishable from the drum color.

- Labels will be secured in a manner to ensure the label remains affixed to the container. Labeling or marking requirements for IDW expected to be transported offsite must be in accordance with the requirements of 49 CFR 172.

4.3 IDW Container Movement

Staging areas for IDW containers shall be predetermined and in accordance with site-specific and/or client requirements. Arrangements shall be made before field mobilization as to the methods and personnel required to safely transport IDW containers to the staging area. Transportation offsite onto a public roadway is prohibited unless 49 CFR 172 requirements are met.

4.4 IDW Container Storage

Containerized IDW shall be staged pending chemical analysis or further onsite treatment. Staging areas and bulk storage procedures are to be determined according to site-specific requirements. Containers are to be stored in such a fashion that the labels can be easily read. A secondary/spill container must be provided for liquid IDW storage and as appropriate for solid IDW storage.

5.0 Procedures

The three general options for managing IDW are:

(1) collection and onsite disposal,

(2) collection for offsite disposal, and

(3) collection and interim management.

Attachment 1 summarizes media-specific information on generation processes and management options. The option selected shall take into account the following factors:

- Type (soil, sludge, liquid, debris), quantity, and source of IDW
- Risk posed by managing the IDW onsite
- Compliance with regulatory requirements
- IDW minimization and consistency with the IDW remedy and the site remedy. In all cases the client shall approve the plans for IDW. Formal plans for the management of IDW must be prepared as part of a work plan or separate document.

5.1 Collection and Onsite Disposal

5.1.1 Soil/Sludge/Sediment

The options for handling soil/sludge/sediment IDW are as follows:

1. Return to boring, pit, or source immediately after generation as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate “clean” areas).

2. Spread around boring, pit, or source within the area of contamination (AOC) as long as returning the media to these areas will not increase site risks (e.g., direct contact with surficial contamination).

3. Consolidate in a pit within the AOC as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate “clean” areas).

4. Send to onsite TSDF - may require analytical analysis before treatment/disposal.

Note: These options may require client and/or regulatory approval.

5.1.2 Aqueous Liquids

The options for handling aqueous liquid IDW are as follows:

1. Discharge to surface water, only when IDW is not contaminated.

2. Discharge to ground surface close to the well, only if soil contaminants will not be mobilized in the process and the action will not contaminate clean areas. If IDW from the sampling of background upgradient wells is not a community concern or associated with soil contamination, this presumably uncontaminated IDW may be released on the ground around the well.

3. Discharge to sanitary sewer, only when IDW is not contaminated.

4. Send to onsite TSDF - may require analysis before treatment/disposal.

Note: These options may require analytical results to obtain client and/or regulatory approval.

5.1.3 Disposable PPE

The options for handling disposable PPE are as follows:

1. Double-bag contents in nontransparent trash bags and place in onsite industrial dumpster, only if PPE is not contaminated.

2. Containerize, label, and send to onsite TSDF - may require analysis before treatment/disposal.

5.2 Collection for Offsite Disposal

Before sending to an offsite TSDF, analysis may be required. Manifests are required. In some instances, a bill of lading can be used for nonhazardous solid IDW (i.e., wooden pallets, large quantities of plastic sheeting). Arrangements must be made with the client responsible for the site to sign as generator on any waste profile and all manifests or bill of ladings; the policy is not to sign manifests. The TSDF and transporter must be permitted for the respective wastes. Nonbulk containers (e.g., drums) must have a DOT-approved label adhered to the container and all required associated placard stickers before leaving for a TSDF off site. These labels must include information as required in 49 CFR 172. Bulk containers (i.e., rolloffs, tanks) do not require container specific labels for transporting off site, but must include appropriate placards as required in 49 CFR 172.

5.2.1 Soil/Sludge/Sediment

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., drummed, covered in a waste pile) or returned to its source until final disposal. The management option selected shall

take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.2 Aqueous Liquids

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., mobile tanks or drums with appropriate secondary containment) until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.3 Disposable PPE

When the final site remedy requires offsite treatment disposal, the IDW may be containerized and stored. The management option selected shall take into account potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.3 Collection and Interim Management

All interim measures must be approved by the client and regulatory agencies.

1. Storing IDW onsite until the final action may be practical in the following situations:

    - Returning wastes (especially sludges and soils) to their onsite source area would require reexcavation for disposal in the final remediation alternative.

    - Interim storage in containers may be necessary to provide adequate protection to human health and the environment.

    - Offsite disposal options may trigger land disposal regulations under the Resource Conservation and Recovery Act (RCRA). Storing IDW until the final disposal of all wastes from the site will eliminate the need to address this issue more than once.

    - Interim storage may be necessary to provide time for sampling and analysis.

2. Segregate and containerize all waste for future treatment and/or disposal.

    - Containment options for soil/sludge/sediment may include drums or covered waste piles in AOC.

    - Containment options for aqueous liquids may include mobile tanks or drums.

    - Containment options for PPE may include drums or roll-off boxes.

6.0 Restrictions/Limitations

Site Managers Shall Determine the Most Appropriate Disposal Option for Aqueous Liquids on a Site-Specific Basis.

Parameters to consider, especially when determining the level of protection, include the volume of IDW, the contaminants present in the groundwater, the presence of contaminants in the soil at the site, whether the groundwater or surface water is a drinking water supply, and whether the groundwater plume is contained or moving. Special disposal/handling may be needed for drilling fluids because they may contain significant solid components. Disposable sampling materials, disposable PPE, decontamination fluids, etc. will always be managed on a site-specific basis.

Under No Circumstances Shall These Types of Materials Be Brought Back to the Office or Warehouse.

7.0 References

Environmental Resource Center. 1997. Hazardous Waste Management Compliance Handbook 2nd Edition. Karnofsky (Editor).

Academy of Certified Hazardous Materials Manager. May 1999. Hazardous Materials Management Desk Reference. Cox.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements, 49 CFR 172.

U. S. Environmental Protection Agency. 1987. A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.1.

U. S. Environmental Protection Agency. August 1990. Low-Level Mixed Waste: A RCRA Perspective for NRC Licensees, EPA/530-SW-90-057.

U. S. Environmental Protection Agency. May 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA/540/G-91/009.

U. S. Environmental Protection Agency. January 1992. Guide to Management of Investigation-Derived Wastes, 9345.3-03FS.

U. S. Environmental Protection Agency. Region IV. November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

Attachment 1
IDW Management Options

Type of IDW	*Generation Processes*	*Management Options*
Soil	• Well/Test pit installations • Borehole drilling • Soil sampling	**Onsite Disposal** • Return to boring, pit, or source immediately after generation • Spread around boring, pit, or source within the AOC • Consolidate in a pit (within the AOC) • Send to onsite TSDF **Offsite Disposal** • Client to send to offsite TSDF **Interim Management** • Store for future treatment and/or disposal
Sludge/Sediment	• Sludge pit/sediment sampling	**Onsite Disposal** • Return to boring, pit, or source immediately after generation • Send to onsite TSDF **Offsite Disposal** • Client to send to offsite TSDF **Interim Management** • Store for future treatment and/or disposal
Aqueous Liquids (groundwater, surface water, drilling fluids, wastewaters)	• Well installation/development • Well purging during sampling • Groundwater discharge during pump tests • Surface water sampling • Wastewater sampling	**Onsite Disposal** • Pour onto ground close to well (nonhazardous waste) • Discharge to sewer • Send to onsite TSDF **Offsite Disposal** • Client to send to offsite commercial treatment unit • Client to send to publicly owned treatment works (POTW) **Interim Management** • Store for future treatment and/or disposal
Decontamination Fluids	• Decontamination of PPE and Equipment	**Onsite Disposal** • Send to offsite TSDF • Evaporate (for small amounts of low contamination organic fluids • Discharge to ground surface **Offsite Disposal** • Client to send offsite TSDF • Discharge to sewer **Interim Management** • Store for future treatment and/or disposal
Disposable PPE and Sampling Equipment	• Sampling procedures or other onsite activities	**Onsite Disposal** • Place in onsite industrial dumpster • Send to offsite TSDF **Offsite Disposal** • Client to send offsite TSDF **Interim Management** • Store for future treatment and/or disposal

Adapted from U.S. Environmental Protection Agency, *Guide to Management of Investigation-Derived Wastes,* 9345-03FS, January 1992.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 4
Field Logbook Content and Control

1.0 Objective

The objective of this project-specific procedure is to set criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

2.0 Background

2.1 Definitions

Biota - The flora and fauna of a region.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks includes field team names; observations; data; calculations; date/time; weather; and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 General Responsibilities

Field Team Leader (FTL) - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

Site Personnel - All personnel who make entries in field logbooks during onsite activities are required to read this procedure before engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities should be defined in the field plan or site-/project-specific sampling and analysis plan.

4.0 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black or blue ink pen
- Ruler or similar scale

5.0 Procedures

5.1 Preparation

In addition to this procedure, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office or vehicle for easy reference.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered before initial use of the logbook. Before use in the field, each logbook will be marked with a specific document control number issued by the document control administrator. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number (if applicable).
- Start date of entries.
- End date of entries.
- Activity (if the logbook is to be activity-specific)
- Site name and location.

The first few (approximately five) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

- Table of Contents
- Date/Description (Start Date)/Reserved for TOC pages 1-5

The remaining pages of the table of contents will be designated as such with “TOC” written on the top center of each page. The table of contents should be completed as activities are completed and before placing the logbook in the records file.

5.2 Operation

Requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information does not need to be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.

- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:

  Above notes authored by:

  - (Date)
  - (Sign name)
  - (Print name)

- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons onsite
  - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data

- Level of personal protection used
- Serial numbers of instruments
- Equipment calibration information
- Serial/tracking numbers on documentation (e.g., carrier air bills)

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g.,data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in thelogbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketchesbe oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.
- Visitors to the site.

5.3 Post-Operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the project office. At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by project personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and nonsubjective. Field logbooks, and entries within, are not to be used for personal use.

7.0 References

Sandia National Laboratories. 1991. Procedure for Preparing Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures, QA-02-03. Albuquerque Environmental Program, Department 3220, Albuquerque, New Mexico.

Sandia National Laboratories. 1992. Field Operation Procedure for Field Logbook Content and Control. Environmental Restoration Department, Division 7723, Albuquerque, New Mexico.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 5
Photographic Documentation of Field Activities

1.0 Objective

The purpose of this project-specific procedure is to provide standard guidelines and methods for photographic documentation, which include still and digital photography and videotape or DVD recordings of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This document shall provide guidelines designed for use by a professional or amateur photographer. This procedure is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

2.0 Background

2.1 Definitions

Photographer - A photographer is the camera operator (professional or amateur) of still photography, including digital photography, or videotape or digital versatile discs (DVD) recording whose primary function with regard to this procedure is to produce documentary or data-oriented visual media.

Identifier Component - Identifier components are visual components used within a photograph such as visual slates, reference markers, and pointers.

Standard Reference Marker - A standard reference marker is a reference marker that is used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates - Slates are blank white index cards or paper used to present information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black indelible marking pens.

Arrows and Pointers - Arrows and pointers are markers/pointers used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds - Contrasting backgrounds are backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

Data Recording Camera Back - A data recording camera back is a camera attachment or built-in feature that will record, at the very least, frame numbers and dates directly on the film.

2.2 Associated Procedures

- Field Logbook Content and Control

2.3 Discussion

Photographs and videotape or DVD recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used, and possible lithologic interpretation. This procedure is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. These items shall become an integral part of the "visual media" that, for the purpose of this document, shall encompass still photographs, digital photographs, videotape recordings (or video footage), and recordings on DVDs. The use of a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the visual media and ensure the representative nature of the photographic documentation.

3.0 General Responsibilities

Field Team Leader - The field team leader (FTL) is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting.

Photographer - The photographer shall seek direction from the FTL and regularly discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this procedure. Responsibilities will be defined in the project sampling plan.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or sampling and analysis plan.

4.0 Required Equipment

A general list of equipment that may be used:

- 35mm camera or disposable single use camera (35mm or panoramic use)
- Standard reference markers
- Slates
- Digital camera
- Extra batteries for 35mm camera
- Arrows or pointers
- Contrasting backgrounds

- Video camera and appropriate storage media (e.g., video tapes, DVDs)
- Medium speed, or multi purpose fine-grain, color, 35mm negative film or slide film (project dependent)
- Logbook
- Data recording camera back (if available)
- Indelible black or blue ink pen
- Storage medium for digital camera

5.0 Procedures

5.1 Documentation

A commercially available, bound logbook will be used to log and document photographic activities. Review Field Logbook Content and Control and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook may be designated as the photographic log and documentation section.

Field Health and Safety Considerations

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on location or operation. Personal protective equipment used in this operation will be site-specific and dictated through requirements set by the site safety officer, site health and safety plan, and/or prescribed by the Health and Safety Plan. The photographer should contact the site safety officer for health and safety orientation before commencing field activities. The site health and safety plan must be read before entry to the site, and all individuals must sign the appropriate acknowledgement that this has been done. The photographer should be aware of any potential physical hazards while photographing the subject (e.g., traffic, low overhead hazard, edge of excavation).

5.2 Operation

5.2.1 General Photographic Activities in the Field

The following sections provide general guidelines that should be followed to visually document field activities and site features using still/digital cameras and video equipment. Listed below are general suggestions that the photographer should consider when performing activities under this procedure:

- The photographer should be prepared to make a variety of shots, from close-up to wide-angle. Many shots will be repetitive in nature or format, especially close-up site feature photographs. Consideration

should therefore be given to designing a system or technique that will provide a reliable repetition of performance.

- All still film photographs should be made using a medium speed, or multi purpose fine-grain, color negative film in the 35mm format unless otherwise directed by the FTL.

- It is suggested that Kodak brand “Ektapress Gold Deluxe” film or equivalent be used as the standard film for the still photography requirements of the field activities. This film is stable at room temperature after exposure and will better survive the time lag between exposure and processing. It is suggested that film speed ASA 100 should be used for outdoor photographs in bright sunlight, ASA 200 film should be used in cloudy conditions, and ASA 400 film should be used indoors or for very low-light outdoor photographs.

- No preference of videotape or DVD brand along with digital storage medium is specified and is left to the discretion of the photographer.

- The lighting for sample and feature photography should be oriented toward a flat condition with little or no shadow. If the ambient lighting conditions are inadequate, the photographer should be prepared to augment the light (perhaps with reflectors or electronic flash) to maintain the desired visual effect.

- Digital cameras have multiple photographic quality settings. A camera that obtains a higher resolution (quality) has a higher number of pixels and will store a fewer number of photographs per digital storage medium.

5.2.2 General Guidelines for Still Photography

Slate Information

It is recommended that each new roll of film or digital storage medium shall contain on the first usable frame (for film) a slate with consecutively assigned control numbers (a consecutive, unique number that is assigned by the photographer as in sample numbers).

Caption Information

All still photographs will have a full caption permanently attached to the back or permanently attached to a photo log sheet. The caption should contain the following information (digital photographs should have a caption added after the photographs are downloaded):

- Film roll control number (if required) and photograph sequence number

- Description of activity/item shown (e.g., name of facility/site, specific project name, project number)

- Date and time

- Direction (if applicable)
- Photographer

When directed by the sampling plan, a standard reference marker should be used in all documentary visual media. While the standard reference marker will be predominantly used in close-up feature documentation, inclusion in all scenes should be considered. Digital media should be downloaded at least once each day to a personal computer; the files should be in either “JPEG” or “TIFF” format. Files should be renamed at the time of download to correspond to the logbook. It is recommended the electronic files be copied to a compact disc for backup.

Close-Up and Feature Photography

When directed by the sampling plan, close-up photographs should include a standard reference marker of appropriate size as an indication of the feature size and contain a slate marked with the site name and any identifying label, such as a well number or core depth, that clearly communicates to the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their in situ locations. This enables a more accurate record of their initial condition and color. When directed by the sampling plan, include a standard reference color strip (color chart such as Munsell Soil Color Chart or that available from Eastman Kodak Co.) within the scene. This is to be included for the benefit of the viewer of the photographic document and serves as a reference aid to the viewer for formal lithologic observations and interpretations.

Site Photography

Site photography, in general, will consist predominantly of medium- and wide-angle shots. A standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and caption/slate be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide-angle shots. In such a case, the film/tape control number shall be entered in the photographic logbook along with the frame number and all other information pertinent to the scene.

Panoramic

In situations where a wide-angle lens does not provide sufficient subject detail, a single-use disposable panoramic camera is recommended. If this type of camera is not available, a panoramic series of two or three photos would be appropriate. Panoramas can provide greater detail while covering a wide subject, such as an overall shot of a site. To shoot a panoramic series using a standard 35mm or digital camera, the following procedures are recommended:

- Use a stable surface or tripod to support the camera
- Allow a 20- to 30-percent overlap while maintaining a uniform horizon
- Complete two to three photos per series

5.2.3 General Photographic Documentation Using Video Cameras

As a reminder, it is not within the scope of this document to set appropriate guidelines for presentation or "show" videotape or DVD recording. The following guidelines are set for documentary videotape or DVD recordings only and should be implemented at the discretion of the site personnel.

Documentary videotape or DVD recordings of field activities may include an audio slate for all scenes. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking close-up shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

It is recommended that a standard reference marker and caption/slate be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original video recordings will not be edited. This will maintain the integrity of the information contained on the videotape or DVD. If editing is desired, a working copy of the original video recording can be made.

A label should be placed on the videotape or DVD with the appropriate identifying information (project name, project number, date, location, etc.).

5.2.4 Photographic Documentation

Photographic activities must be documented in a photographic logbook or in a section of the field logbook. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in the Field Logbook Content and Control Procedure, the following information should be maintained in the appropriate logbook:

- Photographer name.
- If required, an entry shall be made for each new roll/tape/DVD control number assigned.

- Sequential tracking number for each photograph taken (for digital cameras, the camera-generated number may be used).
- Date and time (military time).
- Location.
- A description of the activity/item photographed.
- If needed, a description of the general setup, including approximate distance between the camera and the subject, may be recorded in the logbook.
- Record as much other information as possible to assist in the identification of the photographic document.

5.3 Post Operation

All film will be sent for development and printing to a photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer shall also be responsible for arranging delivery of the negatives and photographs, digital storage medium, or videotape or DVD to the project management representative to be placed in the project files.

5.3.1 Documentation

At the end of each day's photographic session, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained as outlined in Field Logbook Content and Control Procedure.

5.3.2 Archive Procedures

- Photographs and the associated set of uncut negatives, digital media, and original unedited documentary video recordings will be submitted to the project files and handled according to contract records requirements. The project manager will ensure their proper distribution.
- Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

6.0 Restrictions/Limitations

This document is designed to provide a set of guidelines for the field amateur or professional photographer to ensure that an effective and standardized program of visual documentation is maintained.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or "show" photography.

The procedures outlined herein are general by nature. The photographer is responsible for specific operational activity or procedure. Questions concerning specific procedures or requirements should be directed to the project manager or FTL. Note: Some sites do not permit photographic documentation. Check with the site contact for any restrictions.

7.0 References

U. S. Army Corps of Engineers. 2001. Requirements for the Preparation of Sampling and Analysis Plans, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. 1992. National Enforcement Investigations Center. Multi-Media Investigation Manual, EPA-330/9-89-003-R. p. 85. Revised March.

U. S. Environmental Protection Agency. Region IV. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Athens, Georgia. November.

Rail Maintenance Activity-Based Sampling and Analysis Plan - OU6
Project-Specific Procedure - 6
Control of Measurement and Test Equipment

1.0 Objective

The objective of this project-specific procedure is to establish the baseline requirements, procedures, and responsibilities inherent to the control and use of all measurement and test equipment (M&TE). Contractual obligations may require more specific or stringent requirements that must also be implemented.

2.0 Background

2.1 Definitions

Traceability - The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification.

2.2 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that measurements and tests resulting from the use of this equipment be of the highest accountability and integrity. To facilitate that, the equipment shall be used in full understanding and compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures and in accordance with any other related project-specific requirements.

3.0 Responsibilities

All staff with responsibility for the direct control and/or use of M&TE are responsible for being knowledgeable of and understanding and implementing the requirements contained herein as well as any other related project-specific requirements.

The project manager (PM) or designee (equipment coordinator, quality assurance coordinator, field team leader, etc.) is responsible for initiating and tracking the requirements contained herein.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Requirements for M&TE

- Determine and implement M&TE related project-specific requirements
- The maintenance and calibration procedures must be followed when using M&TE
- Obtain the maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment log or a field log as appropriate
- Maintain M&TE records

- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using the appropriate procedure and calibration standards
- Identify and take action on nonconforming M&TE

5.0 Procedures

5.1 Determine if Other Related Project-Specific Requirements Apply

For all M&TE:

The PM or designee shall determine if M&TE related project-specific requirements apply. If M&TE related project-specific requirements apply, obtain a copy of them and review and implement as appropriate.

5.2 Obtain the Operating and Maintenance and Calibration Documents

For GF M&TE that is to be procured:
Requisitioner - Specify that the maintenance and calibration procedures be included.

For GF M&TE that is acquired as a result of a property transfer:
Receiver - Inspect the M&TE to determine whether maintenance and calibration procedures are included with the item. If missing or incomplete, order the appropriate documentation from the manufacturer.

For M&TE that is to be rented or leased from an outside vendor:
Requisitioner - Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, ask the procurement division to request it from the vendor.

5.3 Prepare and Record Maintenance and Calibration Records

For all M&TE:
PM or Designee - Record all maintenance and calibration events in a field log unless other project-specific requirements apply.

For GF M&TE only (does not apply to rented or leased M&TE):
If an equipment log is a project specific requirement, perform the following:

Receiver - Notify the PM or designee for the overall property control of the equipment upon receipt of an item of M&TE.

PM or Designee and User:
- Prepare a sequentially page numbered equipment log for the item.
- Record all maintenance and calibration events in an equipment log.

5.4 Label M&TE Requiring Calibration

For GF M&TE only (does not apply to rented or leased M&TE):
If calibration labeling is a project specific requirement, perform the following:

PM or Designee:
- Read the maintenance and calibration procedures to determine the frequency of calibration required.
- If an M&TE item requires calibration before use, affix a label to the item stating "Calibrate Before Use."
- If an M&TE item requires calibration at other scheduled intervals, e.g., monthly, annually, etc., affix a label listing the dateof the last calibration, the date the item is next due for a calibration, the initials of the person who performed the calibration, and a space for the initials of the person who shall perform the next calibration.

5.5 Operating, Maintaining or Calibrating an M&TE Item

For all M&TE:
PM or Designee and User - Operate, maintain, and calibrate M&TE in accordance with the maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log.

5.6 Shipment

For GF M&TE:
Shipper - Inspect the item to ensure that the maintenance and calibration procedures are attached to the shipping case, or included, and that a copy of the most recent equipment log entry page (if required) is included with the shipment. If the maintenance and calibration procedures and/or the current equipment log page (if required) is missing or incomplete, do not ship the item. Immediately contact the PM or designee and request a replacement.

For M&TE that is rented or leased from an outside vendor:
Shipper - Inspect the item to ensure that the maintenance and calibration procedures and latest calibration and standards certification records are included prior to shipment. If any documentation is missing or incomplete, do not ship the item. Immediately contact the procurement division and request that they obtain the documentation from the vendor.

5.7 Records Maintenance

For GF M&TE:
PM or Designee - Create a file upon the initial receipt of an item of M&TE or calibration standard. Organize the files by contract origin and by M&TE item and calibration standard. Store all files in a cabinet, file drawer, or other appropriate storage media at the pertinent warehouse or office location.

Receiver - Forward the original packing slip to the procurement division and a photocopy to the PM or designee.

PM or Designee and User:

- Maintain all original documents in the equipment file except for the packing slip and field log.
- File the photocopy of the packing slip in the M&TE file.
- Record all maintenance and calibration in an equipment log or field log (as appropriate). File the completed equipment logs in the M&TE records. Forward completed field logs to the PM for inclusion in the project files.

For M&TE rented or leased from an outside vendor:
Receiver - Forward the packing slip to the procurement division.

User:

- Forward the completed field log to the PM for inclusion in the project files.
- Retain the most current maintenance and calibration record and calibration standards certifications with the M&TE item and forward previous versions to the PM for inclusion in the project files.

5.8 Traceability of Calibration Standards

For all items of M&TE:
PM or Designee and User:

- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with other related project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Material Safety Data Sheets to be provided with standards.
- Note standards that are perishable and consume or dispose of them on or before the expiration date.

5.9 M&TE That Fails Calibration

For any M&TE item that cannot be calibrated or adjusted to perform accurately:
PM or Designee

- Immediately discontinue use and segregate the item from other equipment. Notify the appropriate PM and take appropriate action in accordance for nonconforming items.
- Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected and notify the appropriate PM(s) of the results of the review.

6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this procedure may be granted by the health and safety manager and/or quality assurance director. All exemptions shall be documented by the grantor and included in the equipment records as appropriate.

ENSR

Site-Specific SOP for Soil Sample Collection (CDM-LIBBY-05, Revision 2)

September 2008

Site-Specific Sampling Guidance
Libby Superfund Site

Guidance No.: CDM-LIBBY-05, Revision 2

Guidance Title: Soil Sample Collection at Residential and Commercial Properties

Approved by:

5/10/07
Technical Reviewer — Date

5/10/07
QA Reviewer — Date

05/10/07
Volpe Center Approval — Date

5/10/07
EPA Approval — Date

Section 1
Purpose

The goal of this standard operating procedure (SOP) is to provide a consistent method for the collection of 30-point composite surface soil sampling to support all investigations conducted at the Libby Superfund Site and specified in governing guidance documents. This SOP describes the equipment and operations used for sampling surface soils in residential and commercial areas, which will be submitted for the analysis of Libby amphibole asbestos. Refer to each investigation-specific guidance documents or work plan for detailed modifications to this SOP, where applicable. The EPA Team Leader or their designate must approve deviations from the procedures outlined in this document prior to initiation of the sampling activity.

Section 2
Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff with responsibility for the collection of soil samples is responsible for understanding and implementing the requirements contained herein as well as any other governing guidance documents.

Task Leader (TL) or Field Team Leader (FTL) - The TL or FTL is responsible for overseeing sample collection processes as described in EPA approved governing guidance documents (i.e., site-specific sampling and analysis plans [SAPs], quality assurance project plans [QAPPs], etc.). The TL or FTL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and all governing guidance documents. The TL or FTL will communicate with the field team members regarding the specific collection objectives and anticipated situations that require deviation from this SOP. It is also the responsibility of the TL or FTL to communicate the need for any deviations from the SOP with the appropriate EPA personnel (team leader or their designate), and document the deviations using a Field Modification Form provided in each SAP or QAPP.

Field team members - Field team members performing the sampling described in this SOP are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples at properties associated with the Libby Superfund Site. The field team members should have limited discretion with regard to collection procedures but should exercise judgment regarding the exact location of sample points, within the boundaries outlined by the TL or FTL.

Section 3
Equipment

- Measuring tape or wheel – Used to estimate the square footage of each land use area.
- Pin flags – Used to identify composite points within each sampling area.
- Trowel or push probe - For collecting surface soil samples.
- Shovel - For collecting surface soil samples.
- Stainless steel mixing bowl - Used to mix and homogenize composite soil samples after collection. Zip-top bags may also be used for homogenization if approved by the governing guidance documents.
- Gloves - For personal protection and to prevent cross-contamination of samples (disposable, powderless plastic or latex).
- Sample container - Gallon-sized zip-top plastic bags (2 per sample).
- Field clothing and personal protective equipment (PPE) - As specified in the current version of the site health and safety plan (HASP).
- Field sprayers – Used to suppress dust during sample collection and to decontaminate nondisposable sampling equipment between samples.
- Deionized (DI) water- Used in field sprayers to suppress dust and to clean and decontaminate sampling equipment.
- Plastic bristle brush - Used to clean and decontaminate sampling equipment.
- Wipes - Disposable, paper. Used to clean and decontaminate sampling equipment.
- Aluminum foil – Used to wrap decontaminated sampling equipment in between uses to prevent contamination during transport.
- Alconox – Used to clean and decontaminate sampling equipment weekly.
- 6-mil poly bag – Used to store and dispose of investigation-derived waste (IDW).
- Trash bag - Used to store and dispose of general trash.
- Field logbook/PDA - Used to record progress of sampling effort and record any problems and field observations.

- Visual Vermiculite Estimation Form (VVEF) – Used to record semi-quantitative estimates of visual vermiculite at each sub-sample location and point inspection (PI).

- Permanent marking pen - Used to label sample containers.

- Sample ID Labels (Index IDs)– Pre-printed stickers used to label sample containers.

- Cooler or other rigid container - Used to store samples while in the field.

- Custody Seals - For ensuring integrity of samples while in the field and during shipping.

Section 4
Sampling Approach

Upon arrival at each property, the field team will locate all parcels requiring sample collection depending on the investigation-specific objectives detailed in governing guidance documents. Parcels on a property will be sectioned into zones that share a similar land use. Zones established by land use areas may be subdivided based on site conditions (e.g., access, construction setup considerations, etc.). Use areas include:

- Specific Use Area (SUA): flowerbed, garden, flowerpot, stockpile, play area, dog pen, driveway (non-paved), parking lot (non-paved), road (non-paved), alley (non-paved)

- Common Use Area (CUA): yard, former garden, former flowerbed, walkway

- Limited Use Area (LUA): pasture, maintained/mowed field, overgrown areas with trails/footpaths, overgrown areas in between SUAs/CUAs

- Interior Surface Area (ISA): soil floor of garage, pumphouse, shed, crawlspace, earthen basement

- Non-Use Areas(NUA): wooded lot, un-maintained field. NUAs will be identified but will not be sampled at this time because they are not presently considered a complete exposure pathway. However, to the extent that NUAs may become a complete exposure pathway in the future, EPA may revisit NUAs at a later date.

After areas have been designated as zones (i.e., SUA zones, CUA zones, LUA zones, NUA zones, ISA zones), the field team will measure the zones with a measuring wheel and label the zone type and approximate square footage on the field sketch and/or design drawings. There is not a minimum or maximum square footage restriction on any zone.

In establishing zones at the property, no area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be

separated into unique zones for soil sampling. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the FTL but consistent among the teams. Conversely, not all land use areas previously mentioned will be applicable at every property.

It is anticipated that SUAs and ISA zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the FTL but consistent among teams. With the exception of proximal SUAs, all other land use areas will be contiguous when establishing zones at each property.

Composite sampling requires soil collection from multiple (sub-sample) points. Composite samples will be collected from similar land use areas (i.e., SUA, CUA, etc.) and will not be combined with any other use area. One composite sample will be collected from each zone that does not contain visual vermiculite.

For SUAs (e.g., driveway, garden, dog pen, etc.), composite samples will be collected from the 0- to 6–inch depth interval. If a depth of 6 in. cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook/PDA. For non-SUAs (e.g., yard, former flowerbed, crawlspace, etc.), composite samples will be collected from 0 to 3 inches. All composite soil samples will have 30 sub-samples (i.e., 30-point composite sample) of approximately equal size for a final sample volume between 2,000 and 2,500 grams. Table 1 lists the sample depth for each type of land use area.

Table 1 Sampling Area and Depth

Land Use Area	Label	Sampling Depth (Inches)
Special Use Area	SUA	0 – 6
Common Use Areas	CUA	0 – 3
Limited Use Area	LUA	0 – 3
Non-Use Area	NUA	Not Sampled
Interior Surface Zone	IS	0 – 3

As each sub-sample is collected, the soil will be inspected for visual vermiculite (VV) and the location and semi-quantitative estimates of VV will be recorded as prescribed in the SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil, Revision 1 (CDM 2007a).

Areas of SUAs with VV will not be sampled. Instead, the location will be recorded in the field logbook/PDA and on the field sketch or design drawing. If the SUA is of substantial size (greater than 1000 square feet [ft^2]), and the VV is localized, additional PIs will be collected to determine the extent of VV and a sample will be collected from the remainder of the zone that does not contain VV. If the SUA measures less than 1,000 ft^2 and VV is present, a sample will not be collected from that SUA. Proximal

SUAs will not be combined into a SUA zone if VV is present. If visible vermiculite is not observed, proceed with sample collection of the SUA zone

Section 5
Sample Collection

Don the appropriate PPE as specified in the governing HASP. A new pair of disposable gloves is to be worn for each sample collected. Segregate land use areas on the property into zones as described in Section 4. To reduce dust generation during sampling, use a sprayer with DI water to wet each sub-sample location prior to collection. Use the trowel to check beneath the surface soil layer, but do not advance more than 6 inches. If VV is observed, record the information on the field sketch or design drawing. If VV is observed within a large SUA, do not collect a sample from the area containing VV as described above.

Within each zone, select 30 sub-sample locations equidistant from each other. These 30 sub-sample locations will comprise the 30-point composite sample for that zone. All composite sub-samples will originate from the same land use area. For example, do not mix sub-samples from SUAs with sub-samples from LUAs.

Clean the sub-sample locations of twigs, leaves, and other vegetative material that can be easily removed by hand. Using the trowel or push probe, excavate a hole in the soil approximately 2 inches in diameter and 6 inches deep for SUAs, or 3 inches deep for non-SUAs, while placing the excavated material directly inside the gallon-sized zip-top plastic bag. Repeat this step for each subsequent sub-sample until the appropriate number of composite sub-samples has been collected. As each sub-sample is collected, inspect the location for VV as prescribed in the SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil, Revision 1 (CDM 2007a).

Samples collected from zones measuring greater than 3,000 ft^2 will require additional PIs to inspect the soil for VV, but no more than 30 sub-samples will be collected from a zone for each composite sample. Samples collected from zones measuring less than 3,000 ft^2 will have the same number of sub-samples as PIs unless additional PIs are required to identify the extent of localized VV.

Homogenize the sample as required by governing guidance documents. Once the sample is homogenized, fill the zip-top plastic bag to 1/3rd full (approximately 2000 grams). Affix the sample index ID label to the inside of the bag and write the index ID number on the outside of the bag, or affix an additional label using clear packing tape. Sample index ID numbers will be assigned based on the investigation-specific guidance document. Double bag the sample and repeat the labeling process for the outer bag. Decontaminate equipment between composite samples as described in Section 8.

Repeat steps outlined above until all samples from a property have been collected.

Soil field duplicate samples will be collected at the rate specified in governing guidance documents. Field duplicate samples will be collected as samples co-located in the same zone. The duplicate will be collected from the same number of sub-samples as the parent sample, but the sub-sample locations of the duplicate sample will be randomly located in the zone. The inspection for VV at each sub-sample location will follow the same protocol as referenced above. These samples will be independently collected with separate sampling equipment or with the original sampling equipment after it has been properly decontaminated. For tracking purposes, the parent/duplicate sample relationship will be recorded in accordance with sample documentation requirements stated in the governing guidance document. These samples will be used to determine the variability of sample results in a given land use area. These samples will not be used to determine variability in sampling techniques.

Section 6
Site Cleanup

IDW will be managed as prescribed in Section 3.2.10 of the Site-wide QAPP [SWQAPP] (CDM 2007b) or other applicable governing guidance documents. In general, replace the soil plug with excess sample volume. The soil should be placed back into the hole and tamped down lightly. If sandy areas such as playgrounds are sampled, refilling the soil plug is not necessary.

Rinse water, the roots of vegetation removed during sampling, and any excess soil volume may be returned to the sampled area.

Section 7
Documentation

A field logbook/PDA will be maintained by each individual or team that is collecting samples as prescribed in Section 3.2.4 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents. Guidance documents will detail conditions which require attention, but at a minimum the following information should be collected:

- Project name
- Title of governing documents
- Property address
- Date
- Time
- Team members

- Weather conditions
- PPE used
- Locations of any samples or sub-samples that could not be acquired
- Descriptions of any deviations to the SAP or SOP and the reason for the deviation
- Relinquishment of samples to project sample coordinator

Complete required documentation as detailed in applicable governing guidance documents.

Section 8
Quality Assurance/Quality Control

Quality control samples will include:

- Field duplicates

Detailed information on QC sample collection and frequency is prescribed in Section 3.1.3.2 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.

Section 8
Decontamination

All sampling equipment must be decontaminated prior to reuse. Specific instructions on sample equipment decontamination are included in the applicable governing guidance documents. In general, the procedure to decontaminate all soil sampling equipment is outlined below:

- Remove all visible contamination with plastic brush
- Use DI water and plastic brush to wash each piece of equipment
- Remove excess water present on the equipment by shaking
- Use a paper towel to dry each piece of equipment
- Wrap dried equipment in aluminum foil

Once a week all soil sampling equipment will be cleaning using Alconox and DI water.

Spent wipes, gloves, aluminum foil, and PPE must be disposed of or stored properly as IDW, specified in Section 3.2.10 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.

Section 9
Sample Custody

Field sample custody and documentation will follow the requirements described in Section 3.2.11 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.

Section 10
Glossary

Governing guidance documents - The written document that spells out the detailed site-specific procedures to be followed by the project leader and the field personnel for completing specific investigations. These documents will clearly indicate specific requirements for the implementation of this SOP.

Libby Superfund Site – The Libby Superfund Site contains all buildings and land within the boundaries of each operable unit (OU) of the site and illustrated on the most recent version of the OU boundary map.

Sub-sample - The actual location at which the sample is taken. The dimension of a sample point is 2 inches across by 3 inches deep (6 inches for SUAs).

Composite Sampling - A sample program in which multiple sample points are compiled together and submitted for analysis as a single sample.

Land Use Area - A section of property segregated by how the property owner uses the area. The area can be classified as a SUA, LUA, CUA, ISA, or NUA.

Section 11
References

CDM. 2007a. Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties, Revision 1. CDM-LIBBY-06.

CDM. 2007b. Site-Wide Quality Assurance Project Plan. Draft in review.

ENSR

Site-Specific SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil (CDM-LIBBY-06, Revision 1) with modifications

September 2008

Site-Specific Sampling Guidance
Libby Superfund Site

SOP No.: **CDM-LIBBY-06, Revision 1**

SOP Title: Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties

Approved by:

5/10/07
Technical Reviewer — Date

5/10/07
QA Reviewer — Date

05/10/07
Volpe Center Approval — Date

5/10/07
EPA Approval — Date

Section 1
Purpose

EPA will identify and delineate the extent of any visible vermiculite (VV) present in soils as part of all investigations conducted at the Libby Superfund Site and specified in governing guidance documents. The goal of this standard operating procedure (SOP) is to provide a consistent approach to identify and characterize any VV present in soils.

The semi-quantitative approach presented in this SOP for visually estimating VV in soil will be revised as required to optimize data collection as the sampling teams gain experience. This will be accomplished by expanding and/or improving this SOP, supporting pictorial standards, and additional electronic data acquisition efforts, as necessary.

Section 2
Definitions

Specific Use Area (SUA) – Discrete exterior parcels on a property with a designated specific use. Due to the nature of activities typically carried out in SUAs, residents may be especially vulnerable to exposures when Libby amphibole asbestos (LA) contaminated soil becomes airborne. SUAs may be bare or covered with varying amounts of vegetation. SUAs include:

- Flower Pot
- Flowerbed
- Garden
- Stockpile
- Play Area
- Dog Pen
- Driveway (non-paved)
- Parking Lot (non-paved)
- Road (non-paved)
- Alley (non-paved)

Common Use Area (CUA) – Exterior parcels on a property with varied or generic use. CUAs may be bare or covered with varying amounts of vegetation. CUAs include:

- Walkway
- Yard (front, back, side, etc.)
- Former Garden
- Former Flowerbed

Limited Use Area (LUA) – Exterior parcels on a property that are accessed, utilized, and maintained on a very limited basis. LUAs may be bare or covered with varying amounts of vegetation. LUAs include:

- Pasture
- Maintained/Mowed Fields
- Underneath porches/decks[1]
- Overgrown Areas (with trails/footpaths, or between SUAs/CUAs)

Interior Surface Area (ISA) – Interior soil surfaces of buildings such as garages, pumphouses, sheds, and crawlspaces.

Non-Use Area (NUA) – Exterior parcels on a property with no current use (e.g., areas that are un-maintained and not accessed). NUAs may be bare or covered with varying amounts of vegetation. NUAs include:

- Wooded Lots
- Un-maintained Fields

Since NUAs are not currently accessed, they are not presently considered a complete exposure pathway. As such, semi-quantitative visual estimates of vermiculite in soil will not be captured at this time. However, to the extent that NUAs may become a complete exposure pathway in the future, EPA may revisit these NUAs at a later date.

Zone[2] – Parcels on a property that share a similar land use or subdivisions of a land use area based on site conditions (e.g., access, construction setup considerations, etc.) or sampling requirements. No area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for visual inspection. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the field team leader but consistent among the teams.

It is anticipated that SUAs and ISA zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the field team leader but consistent among teams. No ISA will be combined with any other ISA for visual inspection. There is not a maximum square footage restriction on any zone.

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[1] The soils underneath porches and decks will be classified as LUAs depending on ground clearance and accessibility to homeowners and pets. If these areas are not accessible, they will be classified as NUAs.
[2] The restriction on the maximum square footage of SUA zones (1,000 ft2) and non-SUA zones (2, 500 ft2) was eliminated from the previous iteration of this SOP after the data were reviewed by EPA and determined to sufficiently characterize the presence of VV regardless of zone square footage. Additionally, this will allow the flexibility necessary for field teams to identify areas of zones most cost effectively for removal purposes.

Point Inspection (PI) – Used in SUA, CUA, LUA, and ISA zones. A PI is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a zone. A PI consists of the active displacement of the surface soil with a small shovel and visual inspection of the displaced soil to determine if VV is present. If VV is observed during the PI, the location and a semi-quantitative estimate of VV contamination will be recorded.

Section 3
Applicability

This SOP applies to properties within the Libby Superfund Site at varying stages of the removal process including, but not limited to, all screening and risk-based investigations, pre-design inspections, and removal actions. Investigation-specific modifications to this SOP are outlined in the governing guidance document for each investigation. The following locations on a property will be evaluated for the presence/absence of VV:

- All parcels on a property where soil samples are being collected.
- All parcels on a property where soil was non-detect for LA during previous sampling activities.
- All SUA parcels on a property that have not been previously characterized as containing VV

Section 4
Procedure

Figure 1 illustrates the procedures and decision rules for this SOP. The three primary procedural steps are listed below:

- Establish zones
- Perform PI
- Perform semi-quantification of visual vermiculite

Each is described in the following subsections.

4.1 Establish Zones

Upon arrival at the property, the field team will locate all areas requiring sample collection (i.e., where previous soil sample results were non-detect for LA or SUAs have not been previously characterized for VV). Parcels will be identified as SUA zones, CUA zones, LUA zones, NUA zones, or ISA zones. The field team will measure the zone sizes and note them on the field sketch and/or design drawings. Zones will be assigned according to the definitions provided above.

4.2 Point Inspections[3]

As defined above, a PI is an intrusive visual inspection performed for the entire surface of a zone. Professional judgment may be used to determine the exact location of PIs; however, the following guidelines will be implemented to maintain consistency.

A minimum of 30 PIs will be evaluated per zone if sampling is required within that zone. If soil sampling is not required, a minimum of 5 PIs will be evaluated within each zone. Zones larger than 500 square feet (ft^2) will require evaluation at a minimum of 1 PI per 100 ft^2 (10 ft by 10 ft area). The PI locations will be randomly selected and will be spatially representative of the entire zone. Locations of the PIs and semi-quantitative estimates of VV (i.e., low, intermediate, or high) will be recorded on the field sketch for each PI. While a minimum of 5 PIs will be conducted per zone, there is no set maximum. Rather, the maximum number of PIs is variable—dependent upon the total area of the zone and achieving the minimum required frequency of 1 PI per 100 ft^2.

The following sections outline procedures for inspecting each use area (e.g., SUA, CUA, LUA, ISA). The procedure for semi-quantification of VV is provided in the next section.

<u>SUA Zone:</u>

- Visually inspect the PI point using a spade or trowel to remove any cover material, including excess debris (e.g., mulch, rock, etc.) and organic material, from the surface of the soil. Remove and visually inspect soil to a depth of 0-6 inches below ground surface[4].
- If a depth of 6 in. cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook.
- Record semi-quantitative estimate of VV observed as described in the following section.
- Replace soil and cover material.
- Repeat as necessary employing procedure outlined above.

<u>CUA and LUA Zones:</u>

- Visually inspect the PI point using a spade or trowel, carefully removing organic material, including grass, from the surface of the soil. Remove and visually inspect soil to a depth of 0 - 3 inches below ground surface[5].

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[3] Surface Inspections- The non-intrusive visual inspection of the immediate surface of a zone was eliminated from the previous iteration of this SOP after their data were reviewed and determined by EPA to provide no additional information over that gained through Point Inspections.
[4] A soil depth of 6 inches for SUAs was chosen to approximate the depths to which digging would be expected during typical activities occurring in these SUA zones (e.g., gardening, child digging in dirt, etc.)
[5] A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these CUA and LUA zones (e.g., lawn mowing, etc.)

- Record semi-quantitative estimate of VV observed as described in the following section.
- Carefully replace all soil and organic material.
- Repeat as necessary employing procedure outlined above.

ISA Zone:

- Move items as necessary to access the soil surface.
- Visually inspect the PI points using a spade or trowel, remove and visually inspect soil to a depth of 0 - 3 inches below ground surface[6].
- Record semi-quantitative estimate of VV observed as described in the following section.
- Repeat as necessary employing procedure outlined above.

If during the PI, VV is observed to be localized within a zone, the portion with vermiculite will be denoted on the field sketch. If additional PIs are necessary to determine the boundaries of the area, approximately 10 to 20% additional PIs will be evaluated to determine the extent of localized vermiculite.

4.3 Semi-Quantification of Visual Vermiculite

During PI, the field team will estimate the quantity of vermiculite observed. Each PI location for all zones will be assigned a semi-quantitative estimate of visible vermiculite content using a 4-point scale: none (blank), low (L), intermediate (M), and high (H)[7]. For PI locations where VV is observed, semi-quantitative estimates (e.g., L, M, or H) will be recorded on the field sketch. PI locations where VV is not observed will not be recorded on the field sketch. Photographs illustrating these quantities are attached to this SOP as Figure 2. Additionally, jars of vermiculite-containing soils representing these three levels will be available for training and reference.

Under the current version of this SOP, there will be no effort to design an approach to combine vermiculite levels for PIs within or among zones. While the viability of combining semi-quantitative visual estimates within or among zones may be assessed as a pilot-scale evaluation, any PI with visible vermiculite qualifies as vermiculite-containing soil for the area represented by the inspection point or inspection zone.

[6] A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these IS zones (e.g., entering crawlspace, retrieving items from shed, etc.)

[7] Based on EPA's review of previous data, the 5-level scale VV identification scheme was not meaningful and will be reduced to a 4-level scale. As such the quantity of "Gross" VV in the previous iteration of this SOP was combined with High. Previously collected data of Gross VV should be considered analogous to High VV under this revised SOP.

Section 5
Health & Safety/Engineering Controls

All personnel will carry out visual inspections in accord with proper personal protective equipment (PPE) and other monitoring/governing requirements outlined in the most recent version of the Site Health and Safety Plan governing the work being conducted.

All visual inspections will employ appropriate engineering controls to minimize dust (e.g., wetting soil during inspection) as prescribed in the Site-Specific Standard Operating Procedure for Soil Sample Collection (CDM-LIBBY-05, Revision 2).

Section 6
Equipment Decontamination

Equipment decontamination is not required between each PI from the same zone, but is required before moving to another inspection zone. Decontamination of equipment will be conducted as required by the governing guidance documents.

Section 7
Documentation

As noted above, information about the presence of vermiculite will be recorded on the field sketch or design drawing for the property under investigation. Each zone will be marked with:

- Zone type (i.e., SUA, CUA, LUA, NUA, or ISA)
- Zone area in ft^2
- PI locations/points
- Semi-quantitative estimate of VV content for each PI (i.e., L, M, H)

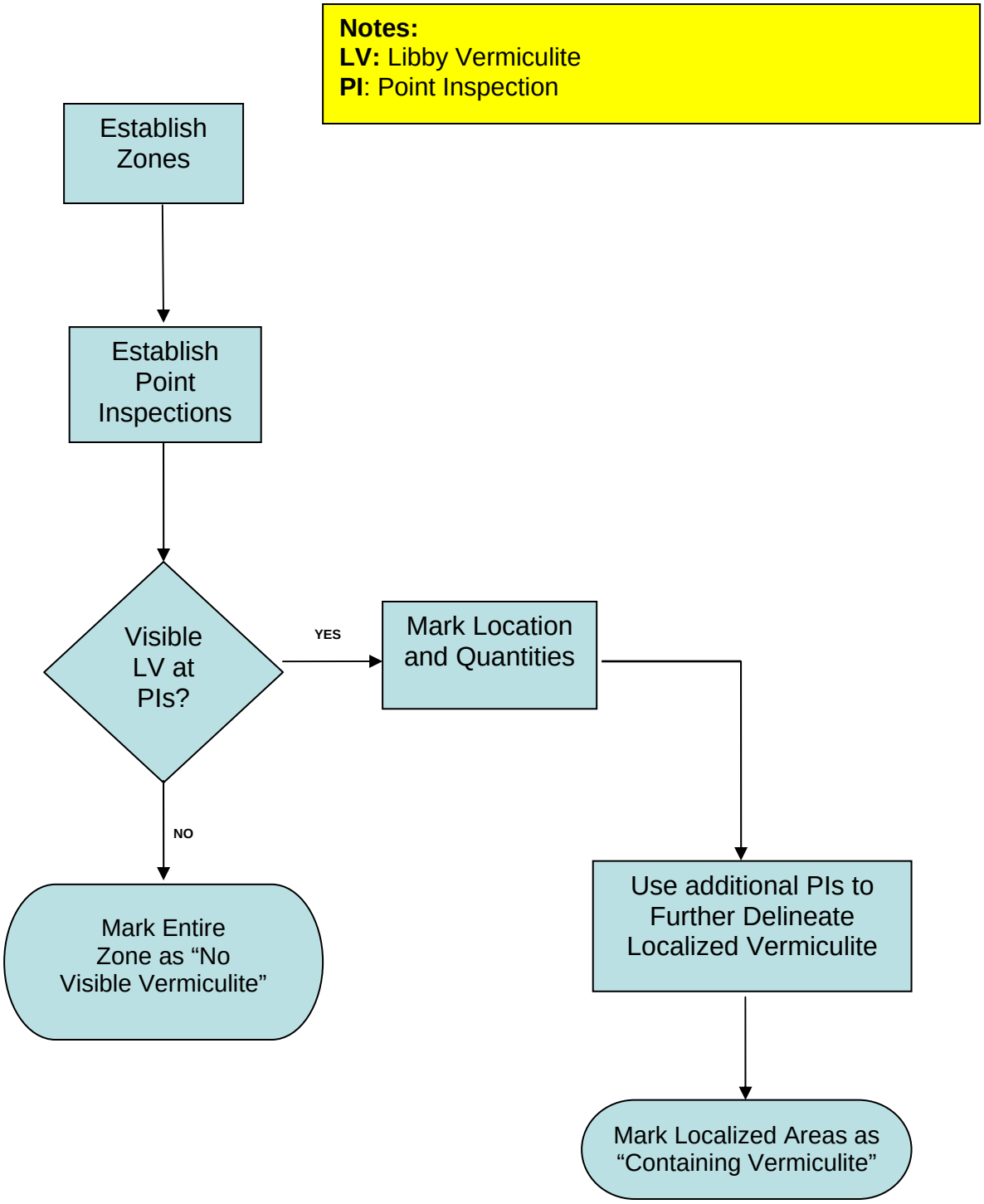
In addition to field sketch/design drawing documentation, each field team will generate a Visual Vermiculite Estimation Form (VVEF) (Figure 3) to document the semi-quantitative visual estimates of VV for each PI for possible future information use. This form will be managed according to governing guidance documents.

Section 8
Training

Every effort will be made to ensure consistency in the semi-quantitative evaluation of VV in soil to the extent possible. This will include training (e.g., field calibration), specimen examples (i.e., jars/photographs of low, intermediate, and high quantities of vermiculite, etc.), designated field staff, and oversight by the field team leader. Figures illustrating none, low, intermediate, and high quantities of vermiculite are attached to this SOP for reference (Figure 2).

To ensure consistency over time, the field team leader will verify semi-quantitative assignments at a rate of one property per team per week. The field team leader will sign off on those field sketches that were verified. If inconsistencies are noted, the field team leader will hold re-training with all teams participating simultaneously. Updates to the SOP and its attached specimen examples will occur as necessary and the EPA Project Team Leader and Technical Assistance Unit will be notified when these updates are recommended by the field team leader or field investigation manager.

Figure 1 – Visible Vermiculite Inspection Process



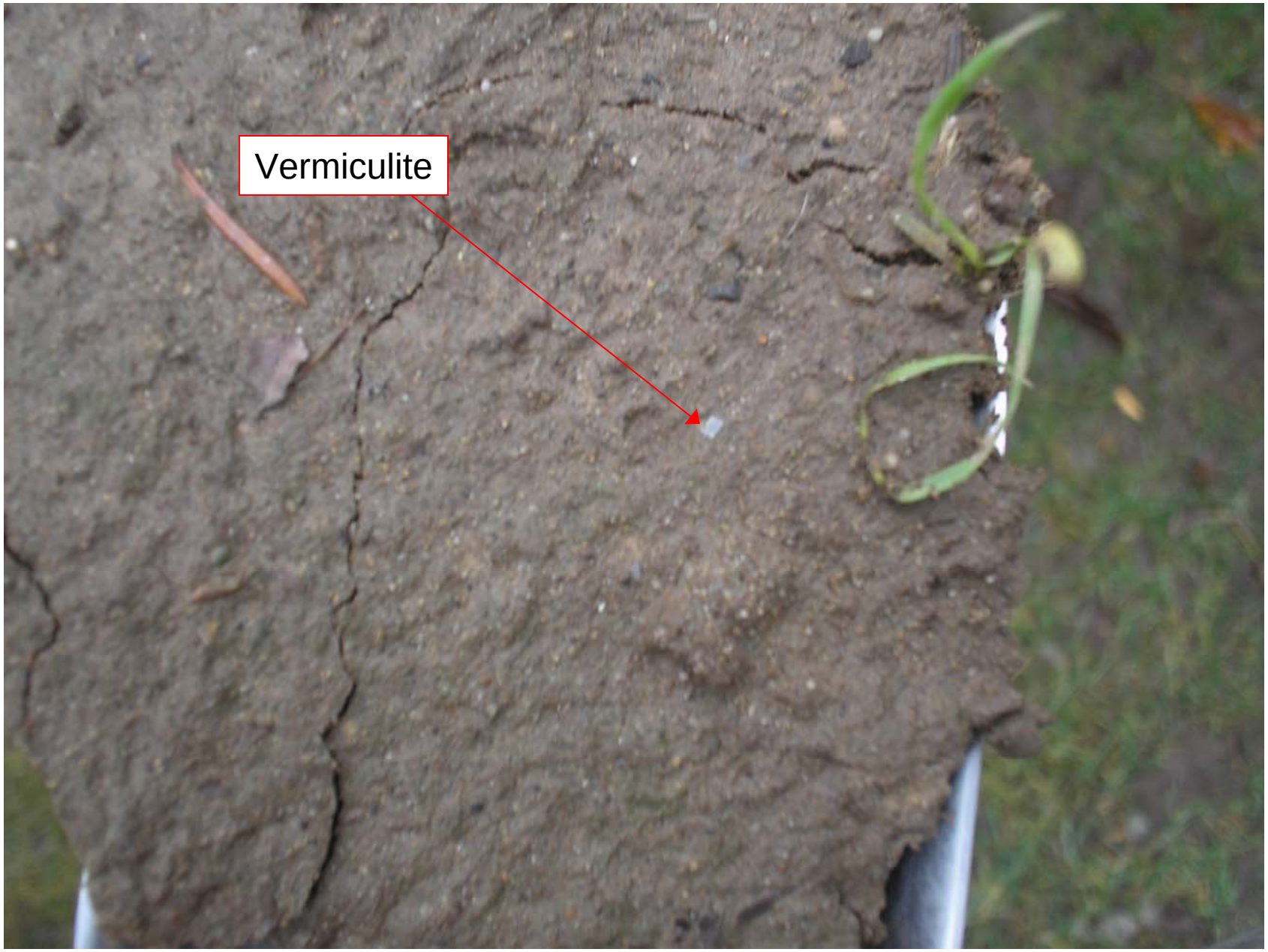


Figure 2a: Low Visible Vermiculite – A maximum of a few flakes of vermiculite observed within a given visual inspection point

Figure 2b: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

Figure 2c: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

Figure 2d: High Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

LIBBY SUPERFUND SITE
Visual Vermiculite Estimation Form (VVEF)

Field Logbook No.: _____ Page No.: _____ Site Visit Date: _____ BD Number: _____
Address: _____ Structure Description: Property
Occupant: _____ Phone No.: _____
Owner (If different than occupant): _____ Phone No.: _____
Investigation Team: _____ Investigation Name: _____
Field Form Check Completed by (100% of Forms): _____ Visual Verification by Field Team Leader (10% of forms): _____

		Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8
Type (SUA/CUA/LUA/IS)									
Description									
Area Size (square feet)									
General Comment (Cover, etc.)									
PIs (X=None, L=Low, M=Intermediate, H=High)	**X**								
	L								
	M								
	H								
	Total	0	0	0	0	0	0	0	0

Areas previously identified for removal not inspected for visible vermiculite? **Yes** **No** **NA** **Location(s):** _____

ENSR

Site-Specific SOP for Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy (SRC-LIBBY-01, Revision 2)

September 2008

Date: May 20, 2003 SOP No. SRC-LIBBY-01 (Revision 1)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson Syracuse Research Corporation

SYNOPSIS: A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	[illegible]	5/20/03
Syracuse Research Corp.	W J Brattin	5/20/03

Revision	Date	Reason for Revision
0	11/12/02	--
1	05/20/03	Provided clarification on dealing with very small particles.

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM). This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana, site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

2.0 PREREQUISITE TRAINING

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than one year of project-specific experience by "mentors" from either Reservoir Environmental Services, Inc. or EMSL.

3.0 RESPONSIBILITIES

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana, project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It

is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

4.0 EQUIPMENT

- Analytical balance - accurate to 0.01 g, range of 0.01 g to 1000 g (for weighing total sample)

- Analytical balance - accurate to 1 mg (for weighing asbestos)

- Traceable standards - major asbestos types

- Microscope - binocular stereomicroscope, 5-60X approximate magnification

- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions

- Light Sources - incandescent or fluorescent

- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools

- Microscope slides and cover slips

- Refractive index liquids

- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers

- HEPA-filtered or Class 1 biohazard hood negative pressure

- Three-ring binder book- binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser, and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of Libby amphibole asbestos, other amphibole asbestos, and chrysotile asbestos in the coarse soil fraction will be calculated from the mass of each asbestos type positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate container. The weight of each coarse sample will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination and particle segregation as depicted Figure 1. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

NOTE: Although the coarse fraction is prepared by sieving with a 1/4" screen, particles smaller than 1/4" may be present in the fraction due to adherence between coarse and fine particles. This may even include some very fine asbestos fibers. Because of the technical difficulty, the analyst should not attempt to physically segregate and weigh particles smaller than about 2-3 mm (1/10 inch). A particle this size is expected to have a mass of about 10-20 mg, which is less than 0.1% of a sample whose total mass is 25 grams. If no particles larger than 2-3 mm are present, this should be noted in the data sheet for each category of asbestos using the following code system:

- ND = No asbestos observed
- Tr = Trace levels of asbestos observed but not quantified

The weight fraction for any asbestos type marked "ND" or "Tr" in a given sample is not calculated and is left blank.

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either "non-asbestos" or "tentatively identified asbestos." The tentatively identified asbestos particles will be examined by PLM, as described below. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.

5.2 PLM

The coarse material tentatively identified as asbestos by stereomicroscopic examination will be subject to confirmation using PLM, as described in SOP SRC-LIBBY-03 (Revision 0) ("Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy"). The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos, and will be assign each particles to one of three categories:

LA = Libby amphibole
OA = Other amphibole
C = Chrysotile

If OA is observed, the type of OA observed should be noted in the data sheet using the following code system:

- AMOS = Amosite
- ANTH = Anthophyllite
- CROC = Crocidolite
- UNK = Unknown

The total weight of each type of positively identified asbestos (LA, OA, C) will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials and the date of the examination.

6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

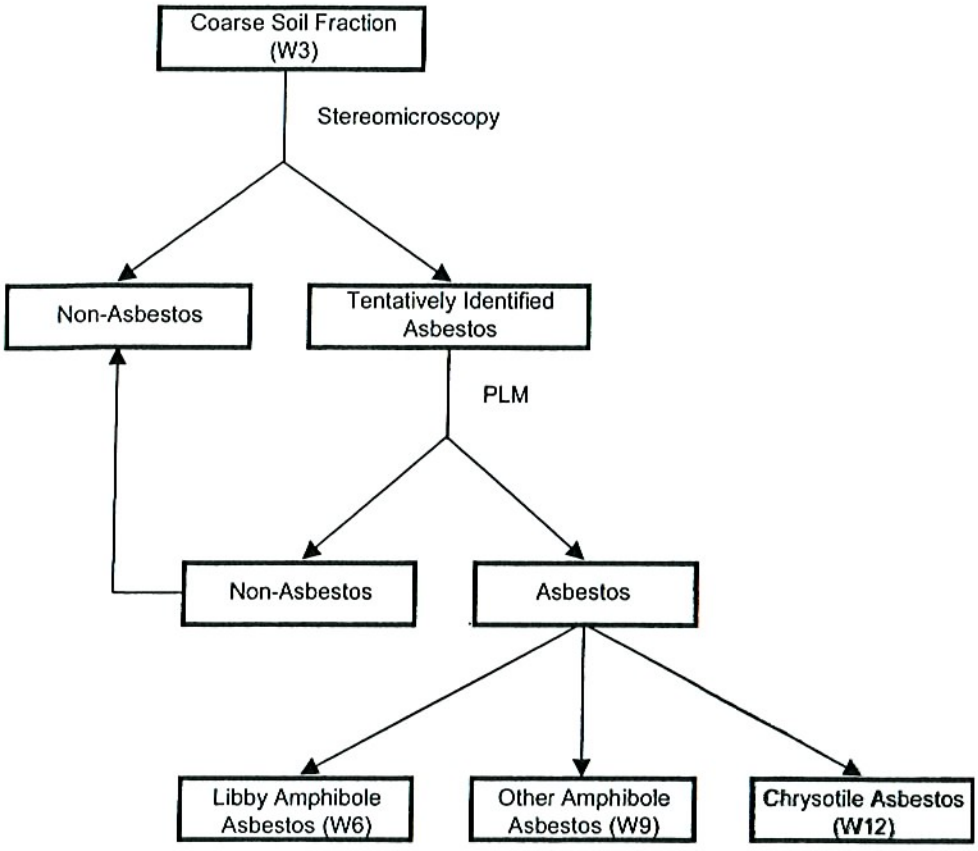
7.0 REFERENCES

CDM 2002. *Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4.* 3282-116-PP-SAMP-14187. Camp, Dresser and McKee Denver, Colorado. April.

NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials*. 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W3 = Original coarse soil fraction mass (g)
W6 = If present in measurable quantities, mass (mg) of Libby amphibole
W9 = If present in measurable quantities, mass (mg) of other amphibole
W12 = If present in measurable quantities, mass (mg) of chrysotile

Codes used in the illustration (e.g., W3) correspond to Data Log Sheet

ATTACHMENT 1

MICROSCOPIC EXAMINATION LOGBOOK SHEET

See attached electronic file "SRC-LIBBY-01 Data sheet and EDD v6.xls"

Example hard copy of data entry sheet shown on next page (for illustration purposes only).

Data Log Sheet v6 for SRC-LIBBY-01
Stereomicroscopic and Gravimetric Analysis of Coarse Soil

Lab Name:
SOP Version:
Lab Job No.

Page _____ of _____

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

EPA Index ID	Index Suffix	Lab Job-Sample No	Status (1 = Analyzed, 2 = Missing, 3 = Contam, 4 = Cancelled)	QA Type (Not QA)	Total Sample Weight (g): Tare Weight (g) Empty Container	Total Sample Weight (g): Mass of Sample + Container (g)	Total Sample Weight (g): Mass of Sample (g)	Analysis Details: Analyst Initials	Analysis Date	Mass of Asbestos Particles (mg): Libby Amphibole (LA) Asbestos: LA Qual* (ND, Tr)	LA: Tare Weight - Container (mg)	LA: Mass of LA + Container (mg)	LA: Mass (mg) LA	Other Amphibole (OA) Asbestos: OA Qual* (ND, Tr)	OA: OA Type** (AMOS, ANTH, CROC, UNK)	OA: Tare Weight - Container (mg)	OA: Mass of OA + Container (mg)	OA: Mass (mg) OA	Chrysotile (C) Asbestos: C Qual* (ND, Tr)	C: Tare Weight - Container (mg)	C: Mass of C + Container (mg)	C: Mass (mg) C	% LA	% OA	% C	Comments (see Notes below)

Notes:

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
UNK = Unknown

Comment Codes (user-defined):

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Site-Specific SOP for Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy (SRC-LIBBY-03, Revision 1)

September 2008

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE IN LIBBY PE STUDY ONLY

ANALYSIS OF SOIL-LIKE MEDIA FOR ASBESTOS BY POLARIZED LIGHT MICROSCOPY

Date: April 20, 2004 SOP No. SRC-LIBBY-03 (Revision 1)

Title: **ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY**

Author: William Brattin

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy (PLM) is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project-specific modifications intended specifically for use at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8	[illegible]	4/20/04
Syracuse Research Corp.	William J Brattin	04/20/04

Revision	Date	Principal Changes
0	03/03/03	--
1	12/11/03	Clarify binning assignment of samples at 0.2%

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using polarized light microscopy (PLM). This SOP is specifically intended for application at the Libby Superfund site.

2.0 SCOPE AND APPLICATION

This method is intended mainly for analysis of asbestos in soil or other similar soil-like media. This method is appropriate for the analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including those that are characteristic of the Libby site.

3.0 RESPONSIBILITIES

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from EPA for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

4.0 METHOD DESCRIPTION

The soil sample to be evaluated for asbestos content by PLM is examined under stereomicroscopy and under PLM (3-5 slides per sample) to estimate the amount of asbestos present. Quantification of the amount of asbestos present may be done either using a visual estimation approach or by a point counting approach, as specified in the Chain of Custody request. In either case, the concentration of Libby amphibole asbestos in the sample is estimated in terms of mass fraction (i.e., percent asbestos by weight) based on the use of project-specific reference materials (calibration standards).

5.0 DETAILED METHOD

5.1 Basic Methods

All qualitative and quantitative analyses are to be performed in general accordance with the methods and techniques specified in NIOSH 9002, EPA 600/R-93/116, and CARB Method 435. Project-specific modification, clarifications, and requirements are provided below.

5.2 Visual Estimation Approach

5.2.1 Classification of Asbestos Mineral Type

Based on fiber attributes (morphology, refractive index, color, birefringence, etc.), asbestos in the sample is classified into one of three categories:

Code	Description	Notes
LA	Libby Amphibole	Refractive index values for LA span the standard values for tremolite/actinolite (EPA 1993), but may include values for other similar amphiboles (e.g., winchite, richterite) characteristic of the mine at Libby. Based on analysis of 4 different samples from the mine (Wylie and Verkouteren 2000; USGS, unpublished data; Verkouteren, personal communication), observed refractive indices of Libby amphiboles range from about 1.629-1.640γ and 1.614-1.623α, with a birefringence of about 0.017. The full range of refractive indices of samples from the mine may be somewhat greater.
OA	Other amphibole	Includes amphibole forms (e.g., amosite, crocidolite, anthophyllite) that are not thought to occur in significant amount at the mine in Libby
C	Chrysotile	

5.2.2 Estimation of LA Mass Percent

The visual area estimation is a semi-quantitative approach that requires the microscopist to estimate the area fraction of the total material present in a field of view that consists of asbestos material. Because this estimation may be difficult, especially at low concentration values, and because the desired output is an estimate of mass fraction (rather than area fraction), all visual estimates of Libby amphibole content will be performed using a set of site-specific reference materials (calibration standards) as a frame of reference. These reference material will contain either 0.2 % or 1.0% Libby amphibole by weight[1], and have been prepared for analysis using the same approach as for field samples. Using the two reference concentrations (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the field sample and report the results as follows:

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	Asbestos was not observed in the field sample
Tr		B1	Asbestos was observed in the field sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	Asbestos was observed in the field sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	Asbestos was observed in the field sample at a level that appeared to equal or exceed the 1% standard. In this case, the mass percent is estimated quantitatively.

"ND" (not detected) in the Qualifier column is used for all samples in which asbestos is not observed under stereomicroscopy and is also not detected in five (5) different PLM slides

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.

"Tr" (trace) in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in at least one out of 3-5 PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be less than the 0.2 % reference material. These samples are assigned to **Bin B1**.

"<" (less than) in the Qualifier column and 1 in the Concentration column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be equal to or greater than the 0.2 % reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.

A numeric value (1, 2, 3, etc) in the Concentration column without an entry in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 1 % reference material. These samples are assigned to **Bin C**.

Note that because these reference materials are based on Libby amphibole, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, other types of asbestos). Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

5.3 Point Counting Approach

5.3.1 Counting Procedure

Any analysis in which evaluation by point counting is requested will be performed in general accordance with the descriptions provided in EPA/600/R-93/116 and CARB Method 435. The total number of particles to be counted (generally 400 or 1000) will be specified in the Chain of Custody request.

Take eight sub-samples of the soil sample and mount each separately with the appropriate refractive index liquid. The preparations should not be heavily loaded. Each sample should be uniformly dispersed to avoid overlapping particles and allow 25-50% empty area within the fields of view.

An ocular reticule (point array) or cross-hair is used to visually superimpose points on the microscope field of view. Count 1/8 of the total points required on each of the 8 slides (e.g., 50 non-empty points per slide for a 400 point count and 125 non-empty points per slide for a 1000 point count). For each non-empty point counted, assign the particle that is present at the point into one of four bins:

- Not asbestos
- Libby asbestos (LA)
- Other asbestos (OA)
- Chrysotile asbestos (C)

In order for a particle to be counted as asbestos, the aspect ratio must be ≥ 3:1.

After the required total number of non-empty points have been counted, record the total number of points in the LA, OA and C bins on the point counting data sheet.

5.3.2 Estimation of Mass Percent

Like visual estimation, the output of the point counting approach is an estimate of area fraction, not mass fraction. For this site, point-count estimates of area fraction for Libby amphibole particles will be converted into estimates of mass fraction using a standard curve approach.

The standard curve will be prepared using a series of site-specific reference materials (calibration standards) containing 0%, 0.2%, 0.5%, 1%, or 2% Libby amphibole. The area fraction of each reference material will be estimated by the point counting approach in quadruplicate. The standard curve will be prepared by plotting the mean area fraction determined by point counting versus the mass percent in the reference material. The mass fraction of a field sample will be determined by measuring the area fraction of the field sample and locating the mass fraction that corresponds to that area fraction on the standard curve.

Because the standard curve is based on Libby amphibole, it is not appropriate to utilize this standard curve for other types of asbestos. Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

6.0 APPARATUS AND MATERIALS

Polarized light microscope, with lens and filters
Stereomicroscope (approximately 10-45x)
Petri dish for stereomicroscopic sample examination
Spatula and forceps
Glass slides and cover slips
Refractive Index (RI) oils
Reference Materials (Calibration Standards)
- Soil containing 0.2% LA by mass
- Soil containing 0.5% LA by mass
- Soil containing 1.0% LA by mass
- Soil containing 2.0% LA by mass

Laboratory log book
Data recording sheet (Attachment 1)

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Precision and Accuracy

PLM by visual estimation and point counting are both semi-quantitative methods. For the purposes of this project, the accuracy and precision of the method are evaluated by measuring the frequency with which samples are assigned to the correct "bins". Data on precision and accuracy of bin assignment will be collected in the future and used to establish performance criteria for this project.

7.2 Method Proficiency

At present, sufficient data are not available to establish a quantitative procedure for method proficiency demonstration. As results become available, a procedure will be established and

applied, based on the analysis of a set of blind Performance Evaluation materials and assessing the frequency of correct bin assignments. If the assignments reported by a laboratory are within acceptance criteria bounds (see Section 7.1), then that laboratory will be deemed proficient. If not, remedial actions must be taken to address the errors before work may begin by that laboratory.

8.0 RECORDS

8.1 PLM Data Forms

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in Attachment 1. Note that there are two different electronic forms; one is for use in visual area estimation, and the other is for use in point counting. Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

8.2 Instrument Maintenance Logbook

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

8.3 Data Storage and Archival

Electronic Data. Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

Hardcopy Data. All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

9.0 REFERENCES

CARB 435. California Environmental Protection Agency Air Resources Board, Method 435, Determination of Asbestos Content in Serpentine Aggregate. June 6, 1991.

EPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. United States Environmental Protection Agency, Office of Research and Development. EPA/600/R-93/116. July 1993.

EPA. 2003. Technical Memo 8. Procedure for Combining Mass Fraction Estimates for Coarse and Fine Fractions of Soil. Prepared by US EPA Region 8 with technical assistance from Syracuse Research Corporation.

NIOSH. 1994. Asbestos (Bulk) by PLM. NIOSH Manual of Analytical Methods, Fourth Edition. National Institute of Occupational Safety and Health. August 15, 1994.

Wylie AG and Verkouteren JR. 2000. Amphibole Asbestos from Libby, Montana: Aspects of Nomenclature. American Mineralogist 85:1540-1542.

ATTACHMENT 1

PLM DATA RECORDING SHEETS

PLM (VE and PC) Data Sheet and ED.xls
(Check with Volpe or SRC to determine the latest version number)

PLM VISUAL ESTIMATION DATA RECORDING SHEET

Page ____ of _____

Laboratory Name _____

Job Number _____

Date Received _____

SOP Name/Revision _____

EPA Index ID	Index Suffix ID	QA Type (NOT QA or LD)	Lab Sample ID	Date Analyzed	Analyst Name	Sample Appearance	Ref Material (B or T)	Libby Amphibole		Other Amphibole			Chrysotile		Deviation?	Comments (list below)
								Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, Tr, <)	Area Fract (%)	QA Type (AMOS, ANTH, CROC, UNK)	Qual (ND, Tr, <)	Area Fract (%)		

Comments (Use back if needed)

PLM POINT COUNT DATA RECORDING SHEET

Laboratory Name _____

Job Number _____

Date Received _____

SOP Name/Revision _____

EPA Index ID	Index Suffix ID	QA Type (NOT QA or LD)	Lab Sample ID	Date Analyzed	Analyst Name	Sample Appearance	Points Counted	Libby Amphibole	Other Amphibole		Chrysotile	Deviation?	Comments (list below)
								Counts	Counts	QA Type (AMOS, ANTH, CROC, UNK)	Counts		

Comments (use back if needed)

Site-Specific SOP for GPS Coordinate Collection and Handling (CDM-LIBBY-09, Revision 0)

September 2008

Project-Specific Standard Operating Procedure
Libby Asbestos Project

SOP No.: CDM-LIBBY-09, Revision 0
SOP Title: Global Positioning Satellite (GPS) Coordinate Collection and Handling
Project: Libby Asbestos Project
Project No.: 2616
Client: U.S. Department of Transportation (DOT)/Volpe Center

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Date: 5/21/07

Terry Crowell
CDM Quality Assurance Reviewer

1.0 Objective

The objective of this standard operating procedure (SOP) is to provide a standardized approach for the collection and handling of GPS data at the Libby Asbestos Site (Site).

2.0 Background

2.1 Definitions

Libby_Sampling Data Dictionary – All Trimble handheld units used at the Site are pre-programmed with the Libby _Sampling data dictionary, specific to the spatial data collection needs for the Libby Asbestos Project. All personnel required to collect GPS data will be familiar with the contents of the Libby_Sampling data dictionary, which contains the following features: Soil Sample, Air Sample, Dustfall (Settled Dust) Sample, Water/Sediment Sample, Building Location, Interest Point, Sample Area, and Interest Area. The Trimble units also are loaded with a generic data dictionary that handles collection of generic lines, points and areas.

2.2 Discussion

The following attributes are required to be collected as indicated in Table 1 for each feature type when a GPS coordinate is collected:

Table 1 – Attributes Collected in the Libby_Sampling Data Dictionary

Feature Name	Attributes Collected
Building Location	LocationID, Address, Comments
Soil Sample	LocationID, IndexID, Sample_Type, SamplGroup, Upper_Depth, Lower_Depth, Comment
Air and Dustfall Samples	LocationID, IndexID, Sample_ Type, SamplGroup , Comment
Water/Sediment Sample	LocationID, IndexID, Matrix_Type, Comment
Interest Point	Location, Land_Use, Comment
Interest Area	Location, Land_Use, Comment
Sample Area	LocationID, IndexID, Num_of _Composites, Upper_Depth, Lower_Depth, Comment

These attributes are discussed in detail in Section 4 of this document.

3.0 Responsibilities

GPS data is collected by investigation, pre-design, and removal oversight staff as specified in the sampling and analysis plans specific to those programs. Transfer of GPS data from the field

equipment to the onsite server, as well as initial data review, processing, and transmittal of data off-site will be performed by a designated on-site IMS staff member during peak field season (April through November), and by administrative support staff during the off season. These additional procedures are documented separately and are posted on CDM's e-room at: https://team.cdm.com/eRoom/R8-RAC/Libby/0_290a.

4.0 Procedures

The following sections describe how GPS points are collected and handled for features commonly used at the Site.

4.1 GPS Point Collection

Building Locations

For building locations, a GPS point is collected near the front door or main entrance of the building. Location IDs beginning with the prefix "BD" (indicating a building point), are used for such locations.

Soil Samples

For **Grab** samples, a GPS point is collected directly above the location where each sample is collected. Location IDs beginning with the prefix "SP" (indicating a sample point), are used for such locations.

For **Composite** samples, a GPS point is collected at the approximate center of each sample area. In the case of an irregular-shaped sample area or sample area that is non-continuous (e.g., a flowerbed that wraps around a house), a GPS point is collected at the center of the largest continuous sample area. Location IDs beginning with the prefix "SP" are used for such locations.

Outdoor Stationary Air and Dustfall (Settled Dust) Samples

For permanent (i.e., samples represent a consistent monitoring zone or area and are collected on a routine schedule) outdoor stationary air and dustfall sample locations, a GPS point is collected at each unique sample location. All subsequent samples taken at that location will be assigned the same Location ID and X,Y coordinates. The GPS point is only collected once. Location IDs beginning with the prefix "SP" (indicating a sample point), are used for such locations.

GPS points are **not** collected for the following features:

- Stationary air, dust, and soil samples collected inside or beneath structures (locations are associated with the X,Y coordinate of the building where the sample was collected)
- Stationary air samples, with the exception of permanent monitoring locations as designated in site-specific removal work plans or Response Action Work Plan Addenda
- Duplicate or Replicate air or dust samples (assigned the same location ID as the parent sample)
- Soil samples taken at depth from the same X,Y location as a previously collected sample. The at-depth soil sample will be assigned the same Location ID as the shallower sample in order to relate both samples to the same X,Y coordinate.
- Duplicate or split soil samples (assigned the same location ID as the parent sample)
- Personal air samples (locations are associated with the X,Y coordinate of the building or property where the sample was collected)

Interest Point, Interest Area, Sample Area
GPS points for these features are not routinely collected on the Libby Asbestos Project. However, they are included in the Libby_Sampling data dictionary in the event that a GPS point is collected for an area where no sampling is involved, or a series of points is collected to document the perimeter of an interest area or sample area.

4.2 Operation of Trimble Pro XRS and GeoXT Handheld Units:

Operators must be standing at the sample location *before* the unit starts to collect positions. Once the unit has started collecting positions, the operator must remain standing at the sample location until the minimum required positions have been collected. A minimum of **30** positions will be collected for each GPS location. More positions will be required in circumstances where the position dilution of precision (PDOP) is greater than the default setting of 4.5. Plan GPS collection around satellite availability & times when PDOP is < 4.5.

Record-keeping Requirements:

Serial numbers of the Trimble datalogger, receiver, and antenna will be recorded in a field logbook. GPS filenames will be recorded in the logbook and on field sample data sheets (FSDSs).

Data Collection Instructions for Trimble Pro XRS:

Turn on the unit and select *Data Collection* from the main menu. You will be prompted to create a new file, open an existing file, or create a base file. Choose *create new file* and press Enter. There will be a generic default file name that begins with "*RO…*" followed by the date. Create a new file name using the following naming convention: **T1A10204**, where **T1** refers to the specific Trimble unit you are using, **A** refers to the first file of the day (**B** would be the second file of the day, and so on), and **10204** refers to October 20, 2004. You are limited to only 8 characters so the date notation will be MMDDY. The setting for data dictionary should always be set to Libby_Sampling. Press Enter to bring up the Start Feature menu.

From the Start Feature menu you will select the type of location data that you want to collect. Press the F1 key to pause the unit until you are ready to start collecting data. Highlight the appropriate data type and press Enter. (Note, if you do not have the unit paused it will start collecting data as soon as you press Enter.) Using the alphanumeric keypad and the directional keypad enter the *Index* and *Location ID* exactly as they appear on the printed labels. Under the *Sample Type* field you will see an arrow indicating a drop-down menu with preset options. If you scroll to the right while *Sample Type* is highlighted you will see the available options. Select the option you want and then scroll to the right again to exit the drop down menu.

Enter any additional information such as Owner, Sample Grid, Sample Location, etc. in the *Comments* field. Press the F1 key to *resume* collecting positions. The unit will beep for every position it collects displaying the total positions in the lower right corner. After the counter has reached the desired number of positions (30 positions), press Enter and then F1 to confirm and save your data point. Repeat this process for every new location.

Review all entries and correct any mistakes before downloading. You can view and edit the data you have collected by pressing F2 (*Review*) from the Start Feature menu. Use the directional pad to scroll through the locations and press Enter to view the sample information.

If changes are made to the data, be sure to press Enter to save the changes, otherwise just press Esc. Press F2 (*New*) to return to the Start Feature menu.

Additional useful handheld features:

- **Review feature –** allows you to quickly view keyed data for errors, making changes as necessary.
- **Repeat feature –** saves time & reduces keystroke errors when collecting multiple samples of the same type.
- **Offset –** reduces the headache and extra time associated with trying to capture GPS data under bridges, large trees, porches, facades and awnings, or while standing close to a building or other object that can deflect satellites signals from the GPS receiver.
- **Delete Feature –** allows you to delete a feature from a file if, for example, no positions were collected or the sample is voided. This will save time & confusion during the QC process.
- **Rename File –** will allow you to browse through the file names you have created, and quickly edit them if necessary. This will save time if it is done *before* the files are downloaded.
- **Delete File –** will allow you to delete a file from the handheld when necessary. This will save time during the QC process if it is done *before* the files are downloaded.

Data Collection Instructions for Trimble GeoXT:

Turn on the unit and with the stylus, select *GPS* from the lower right menu. This will open the Terra Sync software. Wait for the GPS status screen to recognize at least 4 satellites. Depending on your location, this can take several minutes. If you do not wait long enough, you will you not succeed in collecting your data. The connected satellite names will appear on the left side of the screen – they will be highlighted to indicate that they are connected. Select *Data* from the drop down menus at upper left. There will be a generic default file name that begins with "*RO…*" followed by the date. Create a new file name using the following naming convention: **T1A10204**, where **T1** refers to the specific Trimble unit you are using, **A** refers to the first file of the day (**B** would be the second file of the day, and so on), and **10204** refers to October 20, 2004. You are limited to only 8 characters so the date notation will be MMDDY. The setting for data dictionary should always be set to Libby_Sampling. Select *Create.* Confirm the antennae height by selecting *ok.* Highlight the appropriate feature name and select *Create*. The unit will begin logging the point automatically. Enter the attribute data using the stylus and the keyboard icon located at the bottom of the touch screen. When you are finished recording, hit *ok*, which saves the file and location information. If you have other points to collect within the same file, select the *Options* menu then select *Repeat*.

4.3 GPS Data Transfer

GPS File Transfer to Libbysvr02 from Trimble Pro XRS

- Turn on the Trimble Unit
- *The unit will try to connect to the GPS receiver* - press the **Esc** button
- Select **File Manager**
- Select **File Transfer** - *currently the data consists of .ssf files and is transferred to Libbysvr02\Pfdata\Libby - the file is named with an 8character identifier: T+TrimbleUnitNo+ file number(A for first file collected that day)+mmddy*
- Open Pathfinder Office

- Select **Utilities**
- Select **Data Transfer**
- Select **Add**
- Select **Datafile –** *Pathfinder will search for a connection to the Trimble Unit*
- Connect the cable from the computer to the Trimble Unit
- A list of files will appear when the connection is complete
- Select **Open**
- Select **Transfer All**
- When the download is complete, close the data transfer window – *if downloading files from several units, close and reopen this window between downloads*
- Delete files from the Trimble Unit – *all of the files will be listed - double check that all the files were transferred to libbysvr02 before deleting*

GPS File Transfer to Libbysvr02 from Trimble Pro GeoXT

The Trimble GeoXT connects to a PC through the charger unit using a USB cable *(type A to type B)*, and Microsoft Active Sync software. *(There are Active Sync connection settings to enable or disable once the device is connected to the PC. From the Active Sync menu, select Tools, select Options. These connect the Trimble to other Windows applications on the PC eg; email, task managers, etc. The main reason to disable these settings at Libby, is that the Trimble Units are shared and it does not make sense to activate them.)*

- Turn on the Trimble Unit
- Select **GPS** - from lower right corner *(This opens up the TerraSync GPS software.)*
- Select **Setup**
- Select **Options**
- Select **Disconnect from GPS**
- Select **Data**
- At the bottom of list, select **File Manager**
- Open Pathfinder
- Select **Utilities**
- Select **Data Transfer**
- From the Device list, select **GIS Datalogger on Windows CE**
- Click on the connect icon (the button with the checkmark circled in green). *A picture on the right will indicate the connection status.*

4.4 Preliminary On-site Data Quality Control

Following the download of files from the Trimble units, a copy of each file is made and filed in *Libbysvr02\Pfdata\Libby\RawFiles*. The raw files are not modified but kept as the only copy of the complete set of original downloaded data files. Using the Pathfinder export utility, shapefiles (.shp) of the non-quality control checked (QC'd) files located in *Libbysvr02\Pfdata\Libby* are exported. These shapefiles are opened in ArcMap. A new export file of the attribute tables from Arcmap is created and saved as a .dbf file, then opened and saved in Excel workbook format. The Excel file is imported as a new table into a recent copy of the Electronic Libby Asbestos Sample Tracking Information Center (eLASTIC). A report is generated linking the index_id of the imported table with the index_id of the eLASTIC sample

table. This report is saved in Excel. An Excel comparison function is used to compare location ids from the GPS files with the eLASTIC Location IDs. Any discrepancies are researched to determine if the error resides on the FSDS, was a data entry error in eLASTIC, or a data entry error in the GPS .ssf file. Errors in the .ssf files are corrected using Pathfinder Office. Files used for this data review process (.shp, .dbf files and .xls files) are not retained. The QC'd .ssf files are then emailed in a .zip file from the Libby Office to off-site GIS staff for processing. The QC'd and .zip files are moved to *Libbysvr02\Pfdata\Libby\QC and sent zip files.*

For reference on using Pathfinder export and ARCMap attribute tables see Eroom: Libby GIS folder: GPS to GIS procedure posted by Mike Schultz on August 29, 2006.

4.5 Equipment, Software & Configuration

For Trimble Pro XRS or Trimble GeoXT:

Software used for data transfer: GPS Pathfinder Office 2.90 and 3.10
TerraSync

Software used for on-site QC: GPS Pathfinder Office 2.90 and 3.10
ArcGIS ArcMap
Microsoft Excel
eLASTIC

Configuration Settings (TSC1 5.27 software)
Software can vary with rental equipment. Some settings can be changed to accommodate data collection needs.

Table - 2 Configuration Settings for Trimble Pro XRS		
GPS Rover Options - Logging Options		
Logging Intervals	Point feature	1 s
	Line / area	3 s
	Not in feature	none
	Velocity	none
Confirm end feature	no	
Minimum Positions	30	
Carrier phase	Carrier mode	off
	Minimum time	10mins
GPS Rover Options – Position Filters		
Position mode	Manual 3D	
Elevation mask	15 degrees	
SNR mask	6.0	
DOP type	PDOP	
PDOP mask	6.0	
PDOP switch	4.0	
GPS Rover Options – Real-time input		
Preferred correction source	use uncorrected GPS	
GPS Rover Options – General real-time settings		
Correction age limit	10s	
GPS Rover Options – Antenna options		
Height	6.000USft	

Measure	Vertical	
Confirm	Never	
Type	Integrated GPS/ Beacon/Sat	
Part number	33580-50	
GPS Rover Options – Initial Position		
North	USft	
East	USft	
GPS Rover Options – 2D altitude		
Altitude(MSL)	USft	
Computed at	time	
Computed at	date	
GPS Base Station Options – Logging Options		
Logging Intervals	Measurements	5s
	Positions	30s
Audible Click	Yes	
Log DOP data	Yes	
GPS Base Station Options – Position Filters		
Position mode	Manual 3D	
Elevation mask	15 degrees	
SNR mask	4.0	
PDOP mask	6.0	
PDOP switch	4.0	
GPS Base Station Options – Real-time output options		
Real-time output mode	off	
Radio type	Custom	
Baud rate	9600	
Data bits	8	
Stop bits	1	
Parity	Odd	
RTCM options	Station	1
	Message type	Type 1
	Message interval	5s
	Message suffix	None
	CTS flow control	Off
	CTS xmit delay	0ms
	RTS mode	High
	RTS edge delay	0ms
GPS Base Station Options – Reference position		
Datum	NAD 1983 (Conus)	
Zone	11 North	
NMEA/TSIP Output options		
Output	TSIP	
Baud rate	38400	
Coordinate System	UTM	
Map display options	All show with no background	
Units and Display		
Units	Distance(2D)	US Survey Ft
	Area	Square feet
	Velocity	Miles/Hour
	Angle format	DDMMSSss
	Order	North/East
	North reference	True
	Magnetic declination	Auto

	Null string	
	Language	English
Time and Date	24 hour clock	Yes
	Time	##:##:##
	Date format	MM/DD/YYYY
	Date	MM/DD/YY weekday
Quickmarks	Attributes	Repeat
	Confirm	No
Hardware(TSC1) software version 5.27		

Table - 3 Libby Sampling Data Dictionary
"Libby Sampling", Dictionary
"Soil Sample", point, "", 1, seconds, 1, Code
"LocationID", text, 30, required, "SP-000001", required, SP-
"IndexID", text, 30, required, required, Label1
"Sample_Type", menu, required, required, Label2
"COMPOSITE", default
"GRAB"
"SamplGroup", menu, required, required
"BARN"
"BARROW SOURCE"
"BASEMENT"
"BLANK"
"DRIVEWAY"
"FIELD"
"FLOWER BED"
"GARAGE"
"GARDEN"
"HOUSE"
"PARK"
"PROPERTY"
"ROAD"
"SCHOOL"
"SHED"
"WALKWAY"
"YARD", default
"STOCKPILE"
"Upper_Depth", text, 30, required, "Inches", required
"Lower_Depth", text, 30, required, "Inches", required
"Comment", text, 30, normal, normal
"Air Sample", point, "", 1, seconds, 1, Code
"LocationID", text, 30, required, required
"IndexID", text, 30, required, required, Label1
"Sample_Type", menu, required, required, Label2
"PERSONAL"
"STATIONARY", default
"SamplGroup", menu, required, required
"BARN"
"BARROW SOURCE"
"BASEMENT"
"BLANK"
"DRIVEWAY"
"FIELD"

"FLOWER BED"
"GARAGE"
"GARDEN"
"HOUSE", default
"PARK"
"PROPERTY"
"ROAD"
"SCHOOL"
"SHED"
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ENSR

Activity-Based Air Sampling for Asbestos (USEPA Emergency Response Team [ERT] #2084) with modifications

September 2008

STANDARD OPERATING PROCEDURES

ACTIVITY-BASED AIR SAMPLING FOR ASBESTOS

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1.0 SCOPE AND APPLICATION

As a result of a directive issued by the United States Environmental Protection Agency (U.S. EPA) Office of Solid Waste and Emergency Response (OSWER Directive 9345.4), estimating asbestos exposures resulting from suspension of soils is an area of increased importance to the Superfund Program. Such exposures may be estimated via monitoring and/or modeling methods. At present, models are not available to accurately estimate asbestos exposure associated with the disturbance of contaminated soil. Therefore, personal monitoring in the form of activity-based sampling (ABS) is the most appropriate technique to estimate exposure. Personal exposure is influenced by the activities performed, the duration of the activity and the site-specific soils of interest.

At a number of diverse sites across the county (Clear Creek Management Area, San Benito County, California (CA), El Dorado Schools, North Ridge Estates, Klamath Falls, Oregon, Slodusty Road, Garden Valley CA, Ambler Alaska), the U.S. EPA has demonstrated that disturbance of soil with low levels of asbestos (including soil concentrations less than 1.0 percent (%) as measured by Polarized Light Microscopy) can potentially result in significant concentrations (>0.1 structures per cubic centimeter) of respirable asbestos fibers in the breathing zone of individuals engaged in various physical activities. This may result in a cancer risk in excess of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) remedial objectives.

Since personal monitoring is more representative of actual exposure than samples obtained from a fixed downwind location (McBride 1999, Rodes 1995, Hildemann 2005), personal monitoring results are generally most relevant to CERCLA risk characterizations. Thus the best measure of actual exposure to an individual would be through the collection of personal air samples over the exposure period of interest (NIOSH 1977). However, at CERCLA sites, it is neither always possible nor practical to do so. EPA has thus developed a sampling procedure called ABS, designed to mimic the activities of a potential receptor.

As part of ABS, U.S. EPA or contractor personnel trained in hazard recognition and mitigation, serve as surrogates for the potentially exposed populace of interest. ABS simulates routine activities in order to mimic and evaluate or predict personal exposures from disturbance of materials potentially contaminated with asbestos. Similar sampling approaches have been used to assess exposures to pesticides and lead (U.S. EPA 2000) and this technique has long been a cornerstone of industrial hygiene wherein workplace exposures are routinely assessed via personal exposure monitoring.

This document provides guidance for ABS for a particular set of activities or scenarios. Personal monitoring may be conducted during various activities such as raking, All-Terrain Vehicle (ATV) riding, rototilling, digging, a child playing in the dirt, weed whacking, lawn mowing, walking with a stroller, bicycling, and playing basketball.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

This document is not intended to be used as a substitute for a site-specific Quality Assurance Project Plan (QAPP) or a detailed Sampling and Analysis Plan (SAP). This document is intended to be used as a reference for developing site-specific QAPPs and SAPs.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

There are two types of ABS that can be employed in the field: generic ABS and site-specific ABS. Generic ABS can be used with potentially contaminated soil and utilizes a rake to disturb the soil over a known area in conjunction with the collection of air samples to characterize potential exposure. Site-specific ABS is also used with contaminated soil; however, it utilizes site-specific activities to disturb the soil, such as riding ATVs, jogging or riding bikes. Although site-specific ABS provides a more realistic measure of fiber release, it can also be more resource intensive and it is recommended to be used after the generic ABS, if results deem necessary.

For all ABS events, asbestos samples should be collected from the breathing zones of the subjects at an appropriate flow rate. Special consideration should be given to characterizing exposure to children as it has been hypothesized that children are more prone to exposure than adults (U.S. EPA 2000) because they tend to be closer to the source. Sample flow rates, duration and final volume will need to be weighed against the number of grid openings that must be counted (cost factor) to obtain the needed sensitivity. Sampling periods should be of sufficient durations (averaging time) to facilitate collection of a representative sample and achieving the required level of sensitivity.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container, and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers or a static charge that could disturb the dust deposited on the filter media.

2. Samples must be handled gently with the filter inlet facing upward to avoid disturbing the particulate deposited on the filter and to minimize the potential of imparting a static charge to the cassette, which might alter the particulate deposition on the filter media.

3. Place the cassette individually in a manila-type envelope. Each envelope should be marked with the sample identification number, total volume, and date.

4. To the best extent possible, the sampling cassettes in the manila envelopes should be placed right side up so that the cassette inlet cap is on top and cassette base is on bottom. Place samples into a shipping container and use enough packing material to prevent jostling or damage. Samples must be handled gently so as not to disturb the dust deposited on the filter media. Do not use vermiculite or any other type of fibrous packing material for samples. If possible, hand carry to lab.

5. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 Area Selection

When selecting areas for ABS, consideration should be given to the potential for off-site migration of contaminants and possible exposure of the public. Within the constraints of ABS, to the degree practical, particulate generation migration off-site should be minimized, and constraints or mitigation protocols established to eliminate public exposure. These constraints/mitigation protocols may include conducting the ABS in remote areas of the site, dust suppression using water mist, building a containment structure, etc. Air sampling should be conducted to document the airborne concentration of asbestos at the site perimeter during activities.

4.2 Flow Rate Considerations

For activities that generate a large quantity of dust (i.e., particulates), sample flow rates may need to be reduced accordingly to avoid overloading the filters. For example, a sampling pump flow rate of approximately 3.0 liters per minute (L/min) was found most effective at one site for monitoring for asbestos while riding ATVs on dusty soils while high soil moisture and reduced particulate generation at another site permitted a 5.0 L/min flow rate.

High flow rates may result in filter damage due to failure of its physical support associated with increased pressure drop, leakage of air around the filter mount so that the filter is bypassed or damage to the asbestos structures (breakup of bundles and clusters) due to increased impact velocities (ISO 10312). High flow rates can also tear the filters during initial pump startup due to the shock load placed on the filter when the pump is first started.

Sampling larger volumes of air and analyzing greater areas of the filter media can theoretically lower the limit of detection indefinitely. In practice, the total suspended particulate (TSP) concentration limits the volume of air that can be filtered as TSP can obscure asbestos fibers. The International Organization for Standardization (ISO) Method 10312 states that the direct analytical method cannot be used if the general particulate loading exceeds approximately 10% coverage of the collection filter. An airborne concentration of approximately 10 micrograms per cubic meter

($\mu g/m^3$), corresponding to clean rural air, results in approximately 10% coverage of the filter media based on a 4000-L sample.

The following formula from ISO 10132 may be used to calculate the analytical sensitivity:

$$S = \frac{A_t}{KA_gV}$$

Where:

S = Analytical sensitivity expressed in structures per liter

A_t = Active area in square millimeters of the collection media or filter

A_g = Mean area in square millimeters (mm^2) of the grid openings examined,

K = Number of grid openings examined

V = Volume of air sampled, in liters

NOTE: 25-millimeter (mm) cassettes have an effective filter area of 385 mm^2 and 37-mm cassettes have an effective filter area of 855 mm^2. The typical grid opening is 0.0057 mm^2. Note: Grid size will vary between laboratories and dimensions should be verified prior to calculating the number of grid openings that must be counted to achieve a particular level of sensitivity.

Table 1 provides an example of the minimum number of grid openings that must be counted in order to achieve various sensitivity and detection limits.

It is frequently more efficient to employ co-located samplers to collect a high and low volume of air. This increases the likelihood of at least one of the two samples being readable using the direct analytical method (ISO 10312) than to lose the sample due to overloading or having to analyze by the indirect method (ISO 13794).

4.3 Transmission Electron Microscopy (TEM) Specimen Preparation Methods

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because other particulate material with which they are associated conceals many of the asbestos fibers present. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.3.1 Direct-Transfer TEM Specimen Preparation Methods

Direct-transfer preparation methods are intended to retain all particles in the same relative positions with respect to each other on the final TEM grids as on the original filter. The membrane filter, or a portion of it, is placed on a microscope slide with the sample face upward, and then collapsed by exposure to acetone vapor. The cleared filter is then etched in a low-temperature plasma asher, subsequently coated with carbon in a sputtering device and then peeled from the glass slide. A portion of the collapsed, etched and carbon-coated filter is then transferred to an electron microscope grid and then extracted with dimethylformamide, glacial acetic acid and water to remove the filter. Once the process is complete, the particles originally collected on the filter are bound in the carbon film and the grids can be observed on a transmission electron microscope (ISO 1995). Direct-transfer TEM specimen preparation methods have the following significant interferences:

- The particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled, restricts the achievable detection limit.

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.

- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting may not be possible.

4.3.2 Indirect TEM Specimen Preparation Methods

In the indirect preparation method the membrane filter, or a portion thereof, is placed on a microscope slide, sample face downward, and ashed in a low temperature asher until complete calcination of the filter is achieved. The ash is then recovered in distilled water and the solution then filtered on a polycarbonate filter. The indirect transfer method re-distributes the particulate on a new membrane filter.

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified (clusters, matrices bundles, etc. may be broken up during sample preparation).

- There is increased opportunity for fiber loss or introduction of extraneous contamination from laboratory glassware, process water, etc.

- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

The direct analytical method (ISO 10312) is the preferred method and every reasonable effort should be made to prevent overloading of the filter, which would necessitate use of the indirect method. Samples that are overloaded may, at the discretion of the project management team, be analyzed by ISO Method 13794 "Ambient air – Determination of asbestos fibres – Indirect-transfer transmission electron microscopy method" (ISO 1999). Results of the ISO 13794 analysis should be reviewed discrete of the ISO 10312 samples and a decision made regarding combining the two data sets.

4.4 Sampling Cassette Orientation

Air sampling cassettes must be oriented with the open face pointing down to preclude large non-respirable particles from falling or settling onto the filter media.

5.0 EQUIPMENT/APPARATUS

- Personal sampling pumps, providing a flow rate from 0.020 L/min up to 4.0 L/min, battery powered
- High flow sampling pumps (i.e., Quik Take 30 or AirCon II), capable of providing a flow rate from 4.0 to 12 L/min, battery or alternating current (AC)
- Mixed cellulose ester (MCE) filter cassettes, 0.45 or 0.8 micrometer (µm), 25-mm diameter, purchased from a certified vendor with appropriate documentation (low filter background counts, consistent filter area, certified leak-free cassettes)
- Sampling setups, Tygon® tubing with Luer type adaptor
- Backpacks
- Sampling stands, for perimeter sampling
- Duct tape
- Tools, miscellaneous (e.g., screwdrivers, pliers, cutting tool, etc.)
- Envelopes, manila-type
- Whirlpak® bags
- Sample labels

- Chain of custody (COC) records
- Logbook and/or sampling worksheets
- Precision rotameter or primary flow standard appropriate for sampling flow rate
- Personal protective equipment (PPE), including but not limited to respirators, boots, gloves, eye protection, hard hat, to be determined based on type of activity and possible exposure
- Decon equipment (Plastic sheeting, liquinox®, buckets, brushes, water, Hudson sprayers, garbage bags, etc.)
- Power sources, e.g., line power, solar recharging batteries, power inverters, generators, etc.

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Pre-Site Sampling Preparation

1. Determine the extent of the sampling effort (number of locations, repetitions, number of samples, etc.), the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).
3. Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan (HASP).
4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.
5. After calibrating the sampling pump, mobilize to the sampling location.

7.2 Calibration Procedures

To determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the equipment. Sampling pumps should be calibrated on a routine basis and prior to use.

A rotameter can be used provided it has been calibrated with a primary calibrator. Typically rotameters are calibrated on a yearly basis. Sampling pumps can be calibrated prior to coming on-site in order to expedite on-site calibration. However, calibration must be verified on-site prior to use.

7.2.1 Calibrating a Personal Sampling Pump with a Rotameter

1. Refer to the manufacturer's manual for the Rotameter Operational Instructions.

2. Set up the calibration train using a rotameter, sampling pump and the sampling cassette that will be used during the sampling event. This train may be set up prior to field mobilization and will be checked in the field again prior to use.

3. To set up the calibration train, attach one end of the polyvinyl chloride (PVC) tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter. Insure that the tubing and rotameter used to calibrate the pump do not restrict the airflow.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6 degrees (^{o}) of vertical (Omega 1987).

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the pre-calibrated flow rate value on the rotameter. Note: rotameters should be marked with the previous calibration date and corresponding flow rates and scale.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.2.2 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. Refer to the manufacturer's manual for operational instructions.

2. Set up the calibration train using a sampling pump, electronic calibrator, and the actual sampling cassette or a representative filter cassette. The same lot of cassettes used for sampling should also be used for calibration.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Select a flow rate to calibrate.

5. Turn the flow-adjust screw or knob on the pump until the desired flow rate is attained on the rotameter.

6. Using the primary calibrator, obtain approximately 10 readings three times until the flow rate of ± 5% of the required flow is attained.

7.3. Meteorology

It is recommended that an onsite, portable, 3-meter meteorological station be established. If possible, sample after two to three days of dry weather and when wind conditions are representative for the climatology of the location based on month and time of day. Historical hourly wind speed and wind direction data should be analyzed before mobilization. Wind speed, wind direction, temperature, and station pressure should be recorded on the meteorological station data logger and real-time data should be available for review on the station display panel. Suggested meteorological station specifications can be found in Table 2, Appendix A or ERT SOP #2129, *Met One Remote Meteorological Station.* Alternatively, a nearby representative meteorological station, as determined by a meteorologist, may be used to acquire the necessary data.

7.4 General Sampling Information

For all activity-based sampling events, except as noted otherwise, asbestos samples will be collected from the breathing zones of the event participants. The breathing zone can be visualized as a hemisphere approximately 6 to 9 inches around an individual's face. Breathing zone samples provide the best approximation of the concentration of contaminants in the air that an individual is

actually breathing. Specific breathing zone heights should be determined on a project-by-project basis based on the anthropometrics for the study population and the participants' positions during the performance of each task.

If it is necessary to relieve a participant from the activity, another sample collector should be suited and ready to participate in the ABS prior to the personnel exchange. The participant will stop the activity, remove the backpack or belt, and pass it to the relief participant similar to the transfer of a baton in a relay race. The original participant will assist the relief participant with donning and adjusting the backpack or belt. The exchange is anticipated to take less than 60 seconds, therefore the sampling pumps and event time clock will not be halted during the exchange. If the exchange requires more than 60 seconds, the pump and event clock will be stopped until activity is re-initiated.

Sample volumes and detection/quantification limits should be specified in the site-specific QAPP with flow rates and sampling periods adjusted accordingly. Typical sensitivity limits that have been employed for risk assessment have been approximately 0.001 S/cc for ABS samples and 0.0001 S/cc for background or reference samples. Based on ISO 10312 Table 1, a sensitivity limit of 0.001 S/cc would require a sample volume of greater than 500 liters to keep the number of grid openings to be counted below 100. Similarly, a sample volume greater than 5000 L would be required to reach 0.0001 S/cc and count fewer than 100 grid openings. For all asbestos sampling, an asbestos sampling train consisting of 0.8-µm, 25-mm mixed cellulose ester (MCE) filter connected to a personal sampling pump will be used. The top cover from the cowl extension on the sampling cassette shall be removed ("open-face") and the cassette oriented face down for all asbestos filters. All samples should be collected open-faced unless a specific requirement for sampling closed-faced exists.

For activity based sampling, a personal sampling pump (or equivalent) or SKC Quick Take 30 will be calibrated to collect between 2 and 12 L/min of air through the filter depending on the capacity of the pump. The flow rate will be based upon the duration of time required to collect a minimum target volume of 560 L and provide a sensitivity limit of 0.001 S/cc.

Generally each activity based sampling event should be repeated a minimum of three times in an area to expose trends. This can be accomplished by a single participant repeating the activity three or more times or by having a single simulation with three or more participants. If soil moisture or seasonal variability is a concern, then three events for each different season or meteorological conditions may be appropriate.

The sampling pumps used should provide non-fluctuating airflows through the filter, and should maintain the initial volume flow rate to within ± 10% throughout the sampling period. A constant flow or critical orifice controlled pump typically meets these requirements. If the flow rate changes by more than 5% during the sampling period, the average of the pre- and post-sampling

rates will be used to calculate the total sample volume. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, sampling should be terminated. Depending on the type of sampling pump used, it may be possible to salvage the sample if sufficient volume was collected; however, it may not be representative of the time it takes for the actual activity to be completed. Depending on the type of sampling pump used, the actual sampling time in hours and minutes before the sampling fault may be displayed and an actual sample volume calculated. If the fault was due to battery failure, it may be possible to check the post-sampling flow.

During certain ABS activities, participants may be fitted with two sampling pumps to collect a high-flow or volume and a low-flow or volume sample. Co-located samples are collected to sample a high and low volume of air to increase the likelihood of at least one of the two samples being readable using the direct analytical method (ISO 10312). Approximately 560 L (40 CFR 763) is collected for the low-flow samples and up to 4000 L for the high-flow samples. The targeted high volume is typically 1200 L, which permits counting approximately 54 grid openings for a sensitivity level of 0.001 S/cc.

7.5 Generic Activity-Based Sampling Scenario / Raking

The raking scenario, also referred to as the generic scenario, is appropriate for all sites with soils potentially contaminated with asbestos. Generic ABS should be employed in a grid pattern to evaluate the potential for fiber release from soil over a portion of the site. If the analytical results are above the criteria that were derived for the site, then remediation or institutional controls should be implemented or additional site-specific ABS should be undertaken. If the analytical results are below the criteria that were derived, then no further action may be necessary.

In this activity or simulation a participant will rake a lawn or garden area to remove debris such as rocks, leaves, thatch and weeds using a leaf rake with a rake width of approximately 20 to 28 inches. Participants should strive to disturb the top half-inch of soil with an aggressive raking motion. This depth will vary based on the objective of the scenario.

Each raking participant donning appropriate PPE will be fitted with a personal sampling pump contained in a backpack with the cassette secured to the shoulder straps near the operator's lapels in the breathing zone. Personnel will rake a lawn or garden area to remove debris for a minimum of 1 to 2 hours (flow rate and sensitivity level dependent). Raking will occur in a measured area with vegetation, soil or rocks/gravel and will occur in an arched motion raking from the left of the participant to the right. The participants will rake the debris towards themselves facing one side of the square for 15 minutes then the participant will turn 90 degrees clockwise and begin a new side. Participants will continue to rake each side of the square and rotate 90 degrees. Once several small piles of debris have been made, the participant shall pick up the debris and place it in a trashcan. The sequence of raking, rotating and picking up debris shall be repeated for the duration of the sampling period. The participant should stay in the same plot for the entire sampling period.

7.6 Site-Specific Activity-Based Sampling Scenarios

If site-specific ABS is undertaken, the number and types of activities as well as the types of scenarios should be based on current and potential land use. Reference to current and currently planned future land use and the effectiveness of institutional or legal controls placed on the future use of the land should be evaluated. Probable land use should be selected based on zoning and the existing land use of the site and adjacent areas.

Land use assumptions should be based on a factual understanding of site-specific conditions and reasonably anticipated use. The land use evaluated for the assessment should be based on a residential exposure scenario (i.e., the default worst-case) unless residential land use is not plausible for the site. Future land use assumptions should be consistent with reasonably anticipated future land use based on input from planning boards, appropriate officials, and the public.

7.6.1 ATV Riding

This scenario might be appropriate for recreational areas or other areas where ATVs are typically ridden where asbestos contamination is present. This activity is designed to be representative of two or more ATV participants riding on a course or trail. Riders should maintain their relative position (lead, middle, tail) throughout the activity.

Each ATV rider wearing appropriate PPE will be fitted with two personal sampling pumps set at two distinct flow rates, to collect approximately 560 and 1200 liters of air, because of filter overloading concerns. The cassettes for the personal sampling pumps will be attached to the shoulder straps of the backpack proximal to the riders' lapels in the breathing zone. It may be beneficial to attach a dust monitor (e.g., DataRAM) to the tail ATV to record dust levels and gauge dust loading. The sampling pumps will be carried in a backpack while the dust monitor, if used, will be mounted to the ATV.

Personnel will ride the ATVs around a course at the same time until a sufficient volume of air has been collected to achieve the required sensitivity limit of 0.001 S/cc of air. The riders, one lead rider and one following rider, will vary the vehicle speed between 5 and 30 miles per hour (mph). Riders will strive for an average speed of 10 mph. The average speed is a target speed only; vehicle speeds will be adjusted to meet track conditions. Vehicles will be equipped with a speedometer and odometer to record speeds and distance traveled. ATV riding and sampling should be conducted for 30 to 120 minutes in duration, depending on dust loading and required detection limits.

ATVs and ATV tires should be selected as appropriate for the area being studied. Specifically, the size (i.e., weight, horsepower, etc.) of the ATV should be appropriate for

the study area. The vehicle tires should have a tread pattern that is representative of those typically used in the area. Local ATV shops or ATV clubs should be consulted for guidance.

7.6.2 Child Playing in the Dirt

This scenario might be appropriate for sites where schools, playgrounds, parks or residential areas, etc. are contaminated with asbestos; the overarching criteria being areas where a child might be expected to play or dig in the dirt. This scenario was designed to be representative of a child playing in the dirt with a shovel and pail.

The event participant wearing appropriate PPE will be fitted with a personal sampling pump; the inlet to the filter will be at a height of approximately 1 to 3 feet above the ground to simulate a child's breathing zone. The actual pump unit should be secured in a backpack or on a belt.

A participant should sit on the ground while digging or scraping the top 2 to 6 inches of surface soil, placing it in a small bucket or pail and dumping it back on the ground. The activity will be paced such that soil will be placed in the bucket and dumped approximately every two to five minutes, regardless of the amount of material in the bucket. The bucket should be emptied rapidly from a height of approximately 12 inches, based on observations of two to four-year-olds playing in a sandbox.

A sampling period and flow rate to collect a sufficient volume of air will be determined as to achieve the project-specific detection/quantification limit. The sampling period will be divided into equal sub-periods to facilitate having the participant face each compass direction for an equal amount of time during the activity. This approach is designed to mitigate the effect of wind direction on potential exposure. Random head and body movement during the activity should further mitigate the impact of wind direction on exposure. Ideally, the participants will face each compass direction at least twice during the sampling event. For example, during a two-hour or 120-minute event, the participant might face North for 15 minutes, rotate to the East for 15 minutes, then South for 15 minutes, then West for 15 minutes and return to the North to repeat the cycle. Participants should move to a fresh patch of soil after the completion of each cycle (360 degree rotation).

7.6.3 Gardening/Rototilling

This scenario might be appropriate for sites where gardening or surface disturbance to a depth of approximately one foot is anticipated. This activity is designed to be representative of individuals participating in gardening activities using a rototiller.

Each rototilling participant donning appropriate PPE will be fitted with a personal sampling pump. The actual pump unit will be contained in a backpack with the cassette secured to the shoulder straps near the operator's lapels in the breathing zone.

Personnel will operate a rototiller for a minimum of two hours to loosen soil in the yard to a depth of approximately 12 inches. The depth chosen is area-specific and will need to be determined on a case-by-case basis. A rear tine rototiller in the six to eight horsepower range will be selected. Other types or sizes of tillers may be appropriate based on the soil conditions and type of gardening being conducted.

A 100 to 720-square-foot plot of land will be selected to till. The average size of a community garden in New Jersey was 720 square feet based on a survey conducted by Rutgers University in 1991 (Patel 1991). The edges will be delineated. Square plots are preferred. The rototiller operator will conduct typical associated activities such as removing rocks and debris from the tilled area. To account for the effects of varying wind direction on potential exposure, the operator will till the soil back and forth towards each side of the square continuously for 10 minutes, shut down the machine or place it in neutral, and rake or sort through the material for five minutes. The operator will then turn 90 degrees in a clockwise direction and repeat the previous 15-minute procedure. The operator will continue to rotate 90 degrees clockwise every 15 minutes until the two-hour sampling period is complete. The participant should stay in the same plot for the entire sampling period.

7.6.4 Weed Whacking/Cutting

This scenario might be appropriate for sites where lawn maintenance might be conducted such as in residential and commercial areas. This activity is designed to simulate a person trimming weeds and grasses.

Each weed-whacking participant will be fitted with a personal sampling pump. The actual pump unit will be contained in a backpack with the cassette secured to the shoulder straps near the operator's lapels in the breathing zone. Personnel wearing appropriate PPE will operate a gas or electric-powered string trimmer. A 25 to 35-cc gas or electric-powered trimmer with a 16 to 18- inch cutting swath will be selected. Trimming and edging will occur in a measured area with thick vegetation (typically 100 to 720-square feet, based on a typical residential garden) (Patel 1991). Trimming will be done using a side to side sweeping motion with the operator moving in a series of straight lines back and forth towards one side of the selected area for 10 minutes, resting five minutes, and turning 90 degrees in a clockwise direction before repeating this 15-minute procedure for the

duration of the sampling period. The participant should stay in the same plot for the entire sampling period.

7.6.5 Digging

Digging might be appropriate for sites where construction projects are likely to occur or where plants might be planted. Digging will occur in a measured area with vegetation, soil or rocks/gravel.

Each digger participant donning appropriate PPE will be fitted with a personal sampling pump contained in a backpack with the cassette secured to the shoulder straps near the operator's lapels in the breathing zone. The participants will dig a hole to approximately two feet deep and two feet (representative of planting a small shrub or digging a fencepost; site-specific dimensions should be specified in the QAPP/SAP) in diameter (Vodak 2004) and will place the soil next to the hole. The participants will then refill the hole with the soil that had been removed. Participants will then rotate 90 degrees in a clockwise direction and continue to dig and refill additional holes until the sampling period is complete. The sequence of digging, filling and rotating shall be repeated for the duration of the sampling period.

7.6.6 Lawn Mowing

Lawn mowing might be appropriate for sites where lawn maintenance might be conducted such as residential and commercial areas.

Each lawn-mowing participant will be fitted with a personal sampling pump contained in a backpack with the cassette secured to the shoulder straps near the operator's lapels in the breathing zone. Personnel wearing appropriate PPE will operate a gas-powered lawn mower. Mowing will occur in a measured area with thick vegetation and will occur in a shrinking square pattern. Participants will divide the area into a number of squares that decrease in size towards the center of the square by the width of the mower swath. Mower blades will be set at approximately 2 to 2.5 inches. A bag-less side discharge 3- to 5-horsepower lawn mower will be used for this exercise.

7.6.7 Walker with Stroller

This scenario might be appropriate for sites such as parks, paths or open-space. The actual pump unit will be secured in a backpack. The cassette for the personal sampling pump will be attached to the shoulder straps of the backpack proximal to the walker's lapel in the breathing zone. A second pump will be placed in the stroller at a child's breathing zone height.

During these events, walkers wearing appropriate PPE pushing a stroller will walk back and forth along a portion of a path until a sufficient volume of air has been collected to achieve the required detection limit. The walkers will vary their speed between 1.5 and 4 mph. Walkers will strive for an average speed of 2 mph. The average speed is a target speed only; speeds will be adjusted to meet trail conditions. Walkers should be equipped with a global positioning system (GPS) unit to estimate average speed and distance traveled.

7.6.8 Jogging

This scenario might be appropriate for sites such as parks, paths or open-space. The actual pump unit will be secured in a backpack. The cassette for the personal sampling pump will be attached to the shoulder straps of the backpack proximal to the jogger's lapel in the breathing zone.

During these events, joggers wearing appropriate PPE will run/jog back and forth along a portion of a path until a sufficient volume of air has been collected to achieve the required detection limit. The joggers will vary their speed between 2.5 and 5 mph. Joggers will strive for an average speed of 4 mph. The average speed is a target speed only; speeds will be adjusted to meet trail conditions. Joggers should be equipped with a GPS unit to estimate average speed and distance traveled.

Two or more joggers can participate in this activity. When multiple joggers participate, they should maintain their relative position throughout the event (lead, middle, tail). Joggers should be spaced five feet apart.

7.6.9 Two Bicycles

Bicycling might be appropriate for sites such as parks, paths or open-space. Two bicyclists wearing appropriate PPE will ride back and forth with one leading and one following along the length of the site portion of a path or ride around a site (no trail) until a sufficient volume of air has been collected to achieve the required detection limit.

The bicycling participants will each be fitted with personal sampling pumps. The actual pump units will be contained in backpacks with the cassettes secured to the shoulder straps near the cyclists' lapels in the breathing zone.

During these events, the bicycle riders will vary their speed between 3 and 15 mph. Riders will strive for an average speed of 8 mph. The average speed is a target speed only; bicycle speeds will be adjusted to meet trail conditions. Bicycles will be equipped

with a GPS to estimate average speed and distance traveled. Riders should maintain their relative position (lead, tail) throughout the activity.

7.6.10 Basketball Scenario

This scenario might be appropriate for sites where basketball courts are present. The basketball scenario was developed to simulate a group of recreational basketball players gathering to play a casual game of basketball for 120 minutes on an outdoor concrete or macadam court. Between four and 10 players wearing appropriate PPE can participate in this exercise.

- From 0 to 15 minutes, two of the players will sweep court with push brooms from the perimeter of the court to the center. While these two people are sweeping the court, the remaining personnel should mill about under the basket and take a few shots.

- From 15 to 30 minutes, shot practice participants stand around the key as for a free throw, with the exception that one of the participants is positioned under the basket to retrieve the ball after each shot. The player closest to the basket on the left side (facing the basket) takes two shots and the ball/shooter rotates counter clockwise after those two shots. Each person shoots consecutively until everyone has taken two shots. The entire group then rotates clockwise. This sequence should be repeated until time expires. Ideally, each player should shoot from each key position and take a turn retrieving the ball under the basket.

- From 30 to 45 minutes, each player takes turns practicing lay-ups. All players line up on the left side of the basket (facing the basket) and shoot one after another. The first person shoots then retrieves the ball for next person in line and so on. Players should use two basketballs with the second person bouncing the ball outside of the key as the first person shoots. Players should run a full cycle from left then a full cycle from right; repeating the left, right cycles until the interval time is up.

- From 45 to 60 minutes, shot practice as described in the 15 to 30 minute interval above will be conducted.

- From 60 to 75 minutes, a half-court game will be played to the degree practical.

- From 75 to 100 minutes, shot practice as described in the 15 to 30-minute interval above will be conducted.

- From 100 to 120 minutes, a lay-up drill as described in the 30 to 45 minute interval above will be conducted.

7.7 Cumulative Exposure Scenario

A cumulative exposure study might be appropriate for sites where individuals move about a site during the course of a day, with varying levels of exposure at multiple indoor and outdoor locations. The objective is to estimate aggregate and cumulative exposure to asbestos over the course of a day. Cumulative exposure studies should be conducted in order to increase understanding of linkages between sources of asbestos and subsequent exposure and dose to humans for use in mitigating risk and reducing exposure and disease.

Over periods of weeks, years or decades, exposures to environmental agents such as asbestos occur intermittently rather than continuously. Yet long-term health effects, such as cancer, are routinely projected based on an average dose over the period of interest (typically years), rather than as a series of intermittent exposures. Consequently, long-term doses are usually estimated by summing doses across discrete exposure episodes and then calculating an average dose for the period of interest (e.g., year, lifetime).

For the cumulative exposure studies, representative members of the population of interest should be selected for 24 hour sampling. The volunteers should be instructed to go about their day as usual. That is, they should not modify their schedule or activities just because they will be wearing a sampling pump.

A minimal description of exposure for a particular route must include exposure concentration and the duration. This is the method of choice to describe and estimate short-term doses, where integration times are of the order of minutes, hours or days. When projecting long term exposures, on the order of years or a lifetime, since it is typically impractical to sample for the entire exposure period, short-term exposure estimates are assumed to be representative of long-term periods and are integrated to estimate long-term exposures, typically with a safety factor to account for variability.

Observations of activities should be recorded throughout each cumulative exposure study, together with the other relevant factors including locations and activities during the study.

Samples will be collected using a personal air pump with a flow rate of approximately 3.5 L/min. Samples shall be collected open-faced with the inlet facing downward at a personal breathing zone height of 4 to 6 feet for 24 hours. Because the battery life for a personal monitor is typically eight to10 hours, the pump shall be changed out at approximately 8-hour intervals (keeping the same filter cassette). Each pump shall be pre-calibrated to 3.5 L/min prior to use. Each monitor shall be worn at normal breathing height during all waking hours. During sleep, the monitor will be placed

in the same room as the sleeping individual. The sampling cassette will be placed proximal to the breathing zone of the reclined participant.

Should a study subject participate in a high dust generating activity such as riding an ATV, the 24 hour sampling cassette event should be paused and a short term exposure sample should be collected on a separate cassette with an appropriately calibrated sampling pump. Once the high dust activity has been terminated, the original 24-hour cassette and pump should be resumed for the remainder of the sampling period. Results of the 2 or more samples, depending on the number of high dust generating events should be summed to derive the total 24-hour exposure data.

7.8 Background/Reference Sampling

Background/reference samples should be collected for all sampling events. A background or reference sample is defined as a sample collected upwind at a distance sufficient to prevent being influenced by the simulated activities and outside the site perimeter. To the degree practical, the area selected for background or reference sampling should be free of known asbestos contamination. The background level should reflect the concentration of asbestos in air for the environmental setting on or near a site or activity location and can be used to evaluate whether or not a release from the site or activity has occurred. Background level does not necessarily represent pre-release conditions or conditions in the absence of influence from source at the site. A background level may or may not be less than the detection limit, but if it is greater than the detection limit, it should account for variability in local concentrations. Background or reference samples should be collected concurrent with ABS using stationary sampling pumps. Sampling and analytical parameters (sample volume grid opening count, etc.) should be prescribed to permit a detection limit approximately an order of magnitude below that of the ABS detection limit.

An Aircon II sampling pump (or equivalent) will be calibrated to collect 10 L/min for on-site and off-site air samples through the filter. The flow rate will allow a minimum target volume of 4000 L and will provide a sensitivity limit of 0.0001 S/cc. Lower volume air samples will be collected concurrently at the ambient air sampling locations. Personal sampling pumps will be utilized in the same manner with the same media at a flow rate between 2- and 3- L/min in order to collect a sample volume of approximately 1000 L. The target sensitivity of these samples is also 0.0001 S/cc when additional grids are counted in accordance with the method. Co-located samples are collected to sample a high and low volume of air to increase the likelihood of at least one of the two samples being readable using the direct analytical method (ISO 10312).

7.9 Perimeter Sampling

Perimeter samples are defined as samples collected upwind, downwind or crosswind of a specific activity. When selecting areas for ABS, consideration should be given to the potential for off-site migration of contaminants and possible exposure of the public. Within the constraints of ABS, to

the degree practical, particulate generation migration off-site should be minimized, and constraints or mitigation protocols established to eliminate public exposure. These constraints/mitigation protocols may include conducting the ABS in remote areas of the site, dust suppression using water mist, building a containment structure, etc. Air sampling should be conducted to document the airborne concentration of asbestos at the site perimeter during activities. Perimeter air monitoring should be conducted to:

- Document air quality during ABS and establish background or upwind levels of asbestos during site activities
- Monitor and document air quality during site activities near sensitive receptors
- Provide risk management information and address public confidence
- Reduce possible liabilities associated with ABS

Perimeter air sampling should be performed to ensure that ABS activities do not result in excessive airborne asbestos emissions from the site. Air samples should be collected and analyzed to determine the concentrations of asbestos at the site perimeter.

An Aircon II sampling pump (or equivalent) will be calibrated to collect 10 L/min for on-site and off-site air samples through the filter. The flow rate will allow a target volume of 4000 L and will provide a sensitivity limit of 0.0001 S/cc. Lower volume air samples will be collected concurrently at the perimeter sampling locations using personal sampling pumps, if loading is an issue. These pumps will be utilized in the same manner with the same media at a flow rate between 2- and 3-L/min in order to collect a sample volume of approximately 1000 L. The target sensitivity of these samples is also 0.0001 S/cc when additional grids are counted in accordance with the method. Co-located samples are collected to sample a high and low volume of air to increase the likelihood of at least one of the two samples being readable using the direct analytical method (ISO 10312).

7.10 Soil Sampling

A sufficient number of soil samples should be collected to characterize the study area. Since particulates are expected to be released from the entire study area, the primary objective of the soil sampling is to estimate the populations mean concentration. Composite samples are appropriate for characterizing study areas and a sampling design program such as Visual Sampling Plan is recommended for calculating the number and location of samples with the appropriate confidence intervals. Soil sampling should be conducted in accordance with ERT SOP #2012, *Soil Sampling*.

Soil characteristics should be documented in conjunction with the activity-based personal exposure monitoring using American Society of Testing and Materials (ASTM), Method D2488 - 00: *Description and Identification of Soils (Visual-Manual Procedure)*, soil moisture by ASTM Method D2216-05*: Standard Test Methods for Laboratory Determination of Water (Moisture)*

Content of Soil and Rock by Mass and grain size by ASTM Method D6913-04e1: *Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis* or Method D422-63 (2002): *Standard Test Method for Particle-Size Analysis of Soils.*

Soil samples should be representative of the soil. Table 3 provides examples of soil sampling depths, which may be disturbed by the activity being performed.

The relationship between the concentration of asbestos in a source material (typically soil) and the concentration of fibers in air that results when the source is disturbed is very complex, depending on a wide range of variables. To date, no method has been found that reliably predicts the concentration of asbestos in air given the concentration of asbestos in the source. Because of this limitation, this SOP emphasizes an empiric approach, where concentrations of asbestos in air at the location of a source disturbance are measured rather than predicted.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, and field blanks).

The concentration result is calculated by dividing the number of asbestos structures reported after the application of the cluster and matrix counting criteria by the sample volume (concentration = number of asbestos structures / sample volume).

9.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

The following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks. Record the following: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

2. All instruments/equipment must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

3. Field blanks should be collected at a rate of one per twenty samples or one per sampling event, whichever is greater

4. Lot blanks should be collected at a rate of at least two per lot

5. Collocated samples should be collected at the frequency of one per sampling event

For TEM analysis, the following QC procedures apply:

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation or handling.

3. Examine laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not commonly available for Removal Program Activities; however, they should be considered on a case-by-case basis.

10.0 DATA VALIDATION

Results of QC samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air-purifying respirator (PAPR) (full face-piece) is necessary in conjunction with high-efficiency particulate air (HEPA) filter cartridges. See applicable regulations for action levels, permissible exposure levels (PEL) and threshold limit values (TLV). If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

For all ABS, appropriate PPE, including Tyvek coveralls, protective gloves and foot wear, and a respirator with HEPA filter cartridges (P-100 or equivalent) should be worn to protect participants. Details regarding PPE and other protective measures should be specified in the site-specific Health and Safety Plan. Special

consideration should be given to the physical safety of the event participants as well as heat stress associated with performing vigorous activities in impermeable clothing.

12.0 REFERENCES

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13.0 APPENDICES

TABLE 1. Minimum Number of Grid Openings Required To Be Counted to Achieve a Given Analytical Sensitivity and Detection Limit. (Adapted from ISO 10312)

TABLE 2. Suggested Meteorological Station Specifications

TABLE 3. Soil Sampling Depth Based on Activities Performed

TABLE 1. Minimum Number of Grid Openings Required To Be Counted to Achieve a Given Analytical Sensitivity and Detection Limit. (Adapted from ISO 10312)

Analytical Sensitivity Structures/cc	Limit of Detection Structures/cc	Volume of Air Sampled (Liters)					
		500	1000	2000	3000	4000	5000
0.0001	0.0003	1066	533	267	178	134	107
0.0002	0.0006	533	267	134	89	67	54
0.0003	0.0009	358	178	89	60	45	36
0.0004	0.0012	267	134	67	45	34	27
0.0005	0.0015	214	107	54	36	27	22
0.0007	0.0021	153	77	39	26	20	16
0.001	0.003	107	54	27	18	14	11
0.002	0.006	54	27	14	9	7	6
0.003	0.009	36	18	9	6	5	4
0.004	0.012	27	14	7	5	4	4
0.005	0.015	22	11	6	4	4	4
0.007	0.021	16	8	4	4	4	4
0.01	0.030	11	6	4	4	4	4

TABLE 2. Suggested Meteorological Station Specifications

Variable	Accuracy	Resolution
Wind Speed (horizontal and vertical)	± (0.2 m/s + 5% of observed)	0.1 m/s
Wind Direction (azimuth and elevation)	± 5 degrees	1.0 degrees
Ambient Temperature	± 0.5" C	0.1" C
Precipitation	± 10% of observed or ± 0.5 mm	0.3 mm
Pressure	± 3 mb (0.3 kPa)	0.5 mb
Solar Radiation	± 5% of observed	10 W/m^2

m/s = meters per second
"C = degrees Centigrade
mm = millimeters
mb = millibar
W/m^2 = watts per square meter
kPa = kilopascal

TABLE 3. Soil Sampling Depth Based on Activities Performed

Activity Based Sampling Scenario	**Soil Sampling Depth**
Raking (metal garden rake)	Surface to 3 inches
Raking (leaf rake)	Surface to 2 inch
ATV riding	Surface to 2 inch
Rototilling	Surface to 12 inches
Digging	Surface to depth of excavation
Child Playing in the dirt	Surface to 3 inches
Weed Whacking	Surface to 2 inches
Lawn Mowing	Surface to 2 inch
Walking with Stroller	Surface to 2 inch
Two Bicycles	Surface to 2 inch
Activities on solid surfaces such as asphalt or concrete	Microvacuum ASTM D 5755

(EPA-LIBBY-09 Rev1) Site-Specific SOP for TEM Data Review and Data Entry Verification

September 2008

Date: March 5, 2008 SOP No. EPA-LIBBY-09 (rev 1)

Title: STANDARD OPERATING PROCEDURE FOR TEM DATA REVIEW AND DATA ENTRY VERIFICATION

Author Lynn Woodbury, Syracuse Research Corporation (SRC)

SYNOPSIS: This standard operating procedure provides a standardized method for review of raw TEM data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses for review and verification, b) review of the original laboratory TEM bench sheets, and c) verification of the transfer of results from the bench sheets into the Libby2 Database. This method is applicable for use only at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	**SIGNATURE/TITLE**	**DATE**
EPA, Region 8		
SRC		

Revision	Date	Reason for Revision
0	12/7/06	--
1	3/5/08	▪ Modify selection procedure to exclude: 1) records associated with files uploaded due to error corrections, and 2) samples that will be validated under other review efforts. ▪ Modify SOP to include a check of samples with errors to ensure that corrections were made properly. ▪ Change review time period from monthly to quarterly. ▪ Add consistency review of data entered in accord with LB-000066. ▪ Refer to LB-000016 (ISO) and LB-000031 (AHERA/ASTM) for appropriate aspect ratio recording rules.

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for review of raw transmission electron microscopy (TEM) data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses that will undergo a data consistency review and verification, b) performing a consistency review of the original laboratory TEM bench sheets to verify that TEM analysts working on the Libby project are performing analyses in accord with project-specific recording rules, and c) verifying the correct transfer of results from the bench sheets into the Libby2 Database.

2.0 PERSONNEL QUALIFICATIONS

Personnel performing data review and verification under this SOP must be skilled and/or trained in interpretation of raw data sheets and electronic data files in support of TEM analysis for the Libby Superfund Site. Personnel must be well-versed in TEM counting rules and Libby project-specific counting and recording rules in order to perform the required consistency reviews.

3.0 APPLICABILITY

A representative portion of TEM data, analyzed for the Libby Superfund Site, will be selected for review and verification to ensure consistency in data collection and data entry. The frequency of samples selected for review is discussed in subsequent sections.

4.0 SELECTION OF TEM RECORDS FOR REVIEW

The goals for selecting a representative subset of TEM results for review and verification are provided below. Selections should be made to ensure representation across several areas: 1) the fraction of total samples analyzed by TEM; 2) the types of programs (SAPs, QAPPs, etc.) carried out at the Site; 3) the laboratories performing TEM analysis.

> Total Samples. Over the course of the Libby project (that begins with the date of this approved SOP), a minimum of ten percent (10%) of all TEM analyses should be selected for review and verification. Samples will be selected in a manner that ensures representation across the different types of programs and the laboratories performing the TEM analysis.
>
> Types of Programs. If there are important differences in sampling and analysis protocols between sampling programs, data reviews and verifications will be stratified by program. At the request of EPA, the frequency of data review may be increased for specific programs of interest (i.e., investigative samples associated with ambient air monitoring, activity-based sampling, and cleanup efficacy evaluations). Of specific interest is ensuring reviews are stratified across programs that reflect differences in structure recording and/or counting rules.
>
> Laboratories performing TEM analysis. Data reviews and verifications will be performed for each laboratory participating in TEM analysis in support of the Site sampling programs.

Specific details for selecting TEM records for review are outlined below.

1. Interlab samples will be selected on a quarterly basis – 1st Quarter = January 1 - March 31, 2nd Quarter = April 1 - June 30, 3rd Quarter = July 1 - September 30, 4th Quarter = October 1 - December 31. At the beginning of each quarterly review period, compile a list of all TEM ISO 10312 and all TEM AHERA/ASTM samples for which new results were uploaded into the Libby2 Database in the preceding quarter (e.g., for the 1st

Quarter, specify a date range of January 1 - March 31). Samples will be selected for review separately for TEM ISO 10312 and AHERA/ASTM.

The Libby2 Database query will be based on the analysis upload date rather than the analysis date to ensure that analyses with an upload in a different quarter as the analysis date are not excluded. For example, consider the case where the TEM ISO 10312 analysis for sample X-12345 was performed on September 22 (in the 3rd Quarter) and the results were uploaded on October 3 (in the 4th Quarter). The selection query performed on October 1 for the 3rd Quarter results, if limited to all results analyzed from July 1 - September 30, would not capture the results for X-12345 because they had not yet been uploaded. The selection query performed on January 1 for the 4th Quarter results, if limited to all results analyzed from October 1 - December 31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

However, use of the analysis upload date has the potential to include both new analyses and corrections to older analyses resulting from earlier validation efforts. To avoid having to re-review analyses that have already been validated and corrected, only new analyses should be selected for review. To do this, the list of candidate samples will be compared to a running list of all previously validated samples[a]. Any samples that have been validated previously will be excluded from selection. In addition, samples that will be validated under other review efforts associated with specific investigations (e.g., ambient air) will also be excluded from selection.

2. A minimum of 10% of all TEM ISO 10312 and TEM AHERA/ASTM analyses will be selected for review each quarter. To the extent practical, these will be first stratified by analyst, with the number of samples from each analyst being in proportion to the total number of samples analyzed by each analyst. If there are important differences between sampling programs (e.g., differences in counting and/or recording protocols), samples will also be stratified by program. In addition, samples will be stratified according to detect/non-detect, with approximately 50% of the samples selected being detects, and 50% being non-detects. The following table illustrates the selection process:

	Analyzed			Selected		
Analyst	Detect	ND	Total	Detect	ND	Total
1	14	112	126	11	6	17
2	20	421	441	16	22	38
3	2	4	6	2	1	3
4	0	8	8	0	1	1
Total	36	545	581	29	30	59

	Goal	Actual
Total	58	59
Detect	29	29
Non-detect	29	30

In this example, there are a total of 581 new TEM ISO 10312 analyses available for the quarter (36 detects + 545 non-detects), analyzed by four analysts. Thus, the total number of TEM ISO 10312 analyses to be selected for review is 10% · 581 = 58.1 (rounded to 58). This total is to be split evenly between detects (29) and non-detects

[a] This running list of all validated samples will include validation efforts associated with specific investigations (e.g., 2006 demolition investigation, ambient air investigation, SQAPP sampling), as well as TEM validation efforts from preceding quarters.

(29). The number of detects and non-detects selected per analysis is calculated by multiplying the target number (29) by the fraction of the total detects and non-detects evaluated by the analyst. For example, for Analyst 1:

Number of detects = 29 · (14/36) = 11.3 (rounded to 11)
Number of non-detects = 29 · (112/545) = 5.9 (rounded to 6)

If an analyst has analyzed at least one sample in a category (detect or non-detect), the minimum number of samples to be selected is one. For example, for Analyst 4, the number of detects analyzed is zero, so the number of detects selected is zero. For non-detects, the number to be selected (computed using the approach above) is:

Number of non-detects = 29 · (8/545) = 0.4 (rounded to 0)

In this case, the number selected is set to the minimum of 1.

As seen, this procedure will tend to select a higher proportion of detects (29 of 36 analyses, 81%) than non-detects (30 of 545 analyses, 6%). This approach is used because it is considered likely that the incidence of errors may tend to be higher in samples with one or more detected structures than in samples with no detected structures.

3. Stratify the list of newly uploaded samples according to program (if applicable), analyst, and detection status (detect, non-detect), and select the appropriate number of samples for each category at random.

4. Based on the samples selected for review, create a list of all the unique analytical laboratory jobs which will be needed to review the selected analyses. Submit the list of analytical laboratory jobs to EPA's project file manager (Volpe).

5. Volpe will provide SRC with electronic copies (as Adobe Acrobat PDFs) of the requested analytical laboratory jobs via CD, an FTP site, or another electronic transfer mechanism.

5.0 CONSISTENCY REVIEW OF LABORATORY BENCH SHEETS

The purpose of the consistency review is to inspect data entered on the laboratory bench sheets in order to identify the occurrence of any data omissions, apparent inconsistencies, or potential errors in structure.

5.1 Consistency Review Procedure for TEM ISO 10312

1. For each TEM ISO 10312 analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.

2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with ISO 10312 counting rules (as modified in Libby Laboratory Modification LB-000016). The types of information that will be reviewed include:

   - The recorded structure types are consistent with the counting rules. Valid structure types include F, B, CC, CD, CF, CR, MC, MD, MF, and MR.

   - Disperse complex structures are broken down in accord with ISO 10312 counting rules and compact complex structures are not broken down. For example, a CD43 should provide 4 secondary structures, with 3 secondary structures greater than 5 um. In this example, the structure type for each of the recorded secondary structures should begin with the "C" prefix (e.g., CF, CB, CR).

- The primary and total columns have been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.

- If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.

- All recorded fibers (F, CF, and MF) meet the appropriate aspect ratio requirement. [See Libby Laboratory Modification LB-000016 for aspect ratio recording rules for ISO 10312.]

- The mineral class is populated for all structures.

- If Libby Laboratory Modification LB-000066 is applicable, the mineral type (e.g., WRTA) and appropriate spectra code (e.g., NaK) is recorded in the structure comment field for all recorded LA, OA, and NAM structures.

- Structure comments (e.g., < 3:1) are supported by recorded data.

- The stored values in the Libby2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.2 Consistency Review Procedure for TEM AHERA/ASTM

1. For each TEM AHERA/ASTM analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.

2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with AHERA/ASTM counting rules (as modified in Libby Laboratory Modification LB-000031). The types of information that will be reviewed include:

    - The recorded structure types are consistent with the counting rules. For AHERA/ASTM, valid structure types include F, B, M, and C.

    - The total column has been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.

    - If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.

    - The recorded structures meet the counting rule requirements. For AHERA/ASTM, all recorded fibers and matrices meet the appropriate aspect ratio requirement. [See Libby Laboratory Modification LB-000031 for aspect ratio recording rules for AHERA/ASTM.]

    - The recorded dimensions for matrices are the protrusion dimensions, not the matrix dimensions (provided sketches will be used to qualitatively assess dimensions).

    - The mineral class is populated for all structures.

    - If Libby Laboratory Modification LB-000066 is applicable, the mineral type (e.g., WRTA) and appropriate spectra code (e.g., NaK) is recorded in the structure comment field for all recorded LA, OA, and NAM structures.

    - Structure comments (e.g., < 5:1) are supported by recorded data.

- The stored values in the Libby 2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.3 Corrective Action

The data reviewer will prepare a list of any apparent inconsistencies, omissions, or other suspected errors. This list will be provided to EPA and to the Libby laboratory coordinator (CDM), who will forward the list to the appropriate laboratories and analysts for review and response.

At the laboratory, the analyst that performed the analysis and the Quality Assurance (QA) personnel that signed off on the TEM electronic data deliverable (EDD) will review the issues identified and determine which of the issues identified are authentic errors that require correction. All errors will be corrected and a revised TEM EDD and/or hard copy bench sheet will be submitted to the Libby laboratory coordinator (CDM). Each laboratory will provide re-training for analysts and QA reviewers, as needed, to minimize the occurrence of errors at the level of the bench sheet and EDD.

6.0 VERIFICATION OF DATA TRANSFER FROM BENCH SHEET TO DATABASE

6.1 Verification Procedure

The purpose of verification is to ensure that the data from the bench sheet have been transferred into the Libby 2 Database without error or omission. The following steps will be performed as part of the data verification procedure.

1. Compare the analysis-specific information provided in the Libby2 Database to the original lab job documentation (e.g., internal laboratory chain of custody, preparation logs, etc.). [Note: Whenever possible, verification will be performed against hand-written notations, NOT internal laboratory summary tables prepared from hand-written notes. Every attempt should be made to obtain the original hand-written notes. If laboratory summary tables are used instead of hand-written notes, this should be documented and specific rationale should be provided.] The following fields will be verified:

    - Analysis Method (TEM-ISO10312, TEM-AHERA, ASTM)
    - Analysis Date
    - Lab Name
    - Lab Job Number
    - Lab Sample Number
    - Preparation Method (Direct, Indirect, or Indirect with Ashing)
    - Filter Status (Analyzed, Overloaded, Damaged, Missing, Cancelled)
    - Primary Effective Filter Area (EFA, mm^2)
    - Secondary EFA (mm^2) [For indirect preparations only]
    - Grid Opening Area (Ago, mm^2) [b]
    - F-factor [For indirect preparations only, direct prep F-factor = 1]
    - Air Volume (L) or Sample Area (cm^2) [c]
    - Analysis Comments

[b] If the grid opening area is not within the expected range (0.005 - 0.015 mm^2), the value should be confirmed with the laboratory.
[c] To account for potential rounding issues, if the reported analysis air volume or sample area different from the value reported for the sample but is within 0.5% this will be noted in the summary report, but the value will be considered to be correct.

2. Verify the calculation of the F-factor for indirect preparations as follows:

   F-factor = Fraction of primary filter used · Volume of resuspension fluid applied to secondary filter / Total resuspension volume

3. Verify the amphibole sensitivity recorded in the Libby2 Database as follows:

   Air Sensitivity = EFA / (GOx · Ago · V · 1000 · F-factor)
   Dust Sensitivity = EFA / (GOx · Ago · SA · F-factor)

   where: EFA = Effective Filter Area (mm^2) [d]
   GOx = Grid Openings Counted for Libby amphibole
   Ago = Area of a Grid Opening (mm^2)
   V = Air Volume (L)
   SA = Dust Sample Area (cm^2)
   F-factor = indirect preparation dilution factor

4. Count the total number of unique grid openings evaluated in the original hand-written laboratory bench sheets, and compare to the number in the field titled "AnalysisGOCounted" in the Libby2 Database. [Note: If more than one analysis has been performed for the same sample, determine if the grid openings recorded in the second analysis were inclusive or exclusive of the grid openings in the first analysis. This check helps identify cases where an updated or revised EDD is added to the database as a new file rather than replacing (overwriting) an old file, thereby resulting in the duplication of some data.]

5. Using the original hand-written laboratory bench sheets, count the total number of "countable" Libby amphibole (LA) structures across all grid openings evaluated, and compare this number with the "binned" LA values stored in the Libby2 Database.

   - For ISO 10312 analyses, LA counts will be compared to Bin G for LA, which is equal to the total number of countable LA.

   - For AHERA/ASTM, LA counts will be compared to the "S<5um" and "S>5um" bins for LA.

6.2 Corrective Action

For each sample where an issue has been identified, the data reviewer will obtain a hard copy of the laboratory bench sheet. Based on a review of the bench sheet, each issue will be classified as either a) an omission or data entry error at the level of the EDD, or b) an error at the level of the data upload from the EDD into the Libby2 Database.

The data reviewer will prepare a list of any noted discrepancies or omissions for each sample, along with the apparent type of error. This list will be provided to EPA and to the Libby laboratory coordinator (CDM) for review and response.

In cases of apparent data omission or error at the level of the EDD preparation, the laboratory coordinator will contact the laboratory and identify the apparent error(s). At the laboratory, the individual responsible for data entry from the bench sheet into the EDD and the QA personnel that signed off on the EDD will review the issue and make corrections to the EDD as needed. If corrections are made, a revised EDD will be submitted to EPA's database manager for re-entry into the Libby 2 Database. Re-training of data entry and QA review personnel may be implemented, as needed.

[d] For direct preparations this will be the primary EFA. For indirect preparations, this will be the secondary EFA.

If the error is due to a database upload error, EPA's database manager (Volpe) will be contacted and notified of the issue. At Volpe, the TEM upload procedure will be reviewed to identify the source of the issue and modified to ensure that future TEM EDDs will be uploaded correctly. Depending on the nature of the issue, it may be necessary to identify other TEM analyses in the Libby 2 Database that would have been similarly impacted. Any potentially impacted TEM analyses should be removed from the Libby2 Database and re-uploaded after the upload procedure has been corrected.

7.0 CHECKING CORRECTIONS

Each quarter, the data reviewer will review the Libby2 Database and the lab job documentation to ensure that the appropriate corrections have been made for all analyses where one or more issues were identified during previous verification efforts. In cases where a revised EDD was uploaded into the database, the data reviewer will ensure that the incorrect EDD has been removed. A comprehensive summary of all issues and their status will be maintained by the data reviewer. As needed, this summary will be provided to EPA's database manager and the Libby laboratory coordinator for follow-up.

8.0 REPORTING

The data reviewer will prepare a report which summarizes the results of the consistency review and data verification for the sample set and identifies areas for improvement. Attachment A provides an example of this report. As seen, this report includes a detailed summary of the consistency review and data verification findings, and includes a summary of the potential implications of the review and verification findings on the data quality and use of the TEM analyses in the Libby2 Database. This report will also provide copies of all electronic spreadsheets generated which track any identified discrepancies and the resolution status of each issue.

Based on the results of the review and verification, EPA may choose to modify (either increase or decrease) the frequency of TEM samples selected for review and verification and/or the selection/review/verification process.

9.0 REFERENCES

Asbestos Hazardous Emergency Response Act (AHERA). 1986. Title 20, Chapter 52, Sec. 4011. Public Law 99-519.

American Society for Testing and Materials (ASTM). 2003. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D 5755-03. American Society for Testing and Materials. October 2003.

International Organization for Standardization (ISO). 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

ATTACHMENT A

EXAMPLE OF TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

Date: _____ Prepared by: _____

Reporting Date Range: _____

SUMMARY OF FINDINGS AND DATA QUALITY IMPLICATIONS

_____

_____

_____

_____

_____

_____

_____

Recommendations for future review and verification: _____

_____

_____

_____

_____

TEM-ISO 10312 SELECTION AND CONSISTENCY REVIEW RESULTS

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-ISO 10312 Analyses			Number of Analyses Selected for Review		
	Detect	Non-Detect	Total	Detect	Non-Detect	Total
Analyst #1, Lab Name						
Analyst #2, Lab Name						
…						
Total						

	Goal	Actual
Selected Total	_____	_____
Selected Detects	_____	_____
Selected Non-Detects	_____	_____

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Reported structure types are inconsistent with ISO guidance

_____ Primary and/or total columns are not populated correctly

_____ NAM structures are recorded and not identified as non-countable

_____ Fibers recorded as countable do not meet aspect ratio criteria (LB-000016)

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with LB-000066

_____ Structure comments are inconsistent with recorded data

_____ Structure attributes in the database do not match the bench sheet

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

TEM-AHERA/ASTM SELECTION AND CONSISTENCY REVIEW RESULTS

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-AHERA/ASTM Analyses			Number of Analyses Selected for Review		
	Detect	Non-Detect	Total	Detect	Non-Detect	Total
Analyst #1, Lab Name						
Analyst #2, Lab Name						
…						
Total						

	Goal	Actual
Selected Total	_____	_____
Selected Detects	_____	_____
Selected Non-Detects	_____	_____

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Reported structure types are inconsistent with AHERA/ASTM guidance

_____ Total column is not populated correctly

_____ NAM structures are recorded and not identified as non-countable

_____ Fibers recorded as countable do not meet aspect ratio criteria (LB-000031)

_____ Recorded dimensions for matrices are matrix dimensions not protrusion dimensions

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with LB-000066

_____ Structure comments are inconsistent with recorded data

_____ Structure attributes in the database do not match the bench sheet

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

DATA TRANSFER VERIFICATION RESULTS

Number of analyses verified[1]: _____ (_____% of total analyses selected)

Number of analyses with data transfer issues identified: _____ (_____% of total analyses verified)

Types of data transfer issues identified:

_____ Incorrect/missing information on analysis details (e.g., lab job number, analysis date, filter status)

_____ F-factor calculation is incorrect or inputs are missing

_____ Air volume or dust area reported by laboratory is inconsistent with field value

_____ Number of grid openings counted is incorrect

_____ Sensitivity calculation is incorrect or inputs are missing

_____ Total number of countable LA structures is incorrect

Do the data transfer issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

Comments: _____

_____

_____

_____

ISSUE RESOLUTION AND STATUS

_____

_____

_____

_____

_____

_____

[1] Only those analyses that have passed the bench sheet consistency review are included in the data transfer verification.

ENSR

(EPA-LIBBY-08) Site-Specific SOP for Indirect Preparation of Air and Dust Samples for TEM Analysis

September 2008

Libby Standard Operating Procedure
Indirect Preparation of Air and Dust Samples for TEM Analysis
Approved for Use at the Libby Superfund Site Only

Date: 1/23/07 SOP No. EPA-LIBBY-08

Title: INDIRECT PREPARATION OF AIR AND DUST SAMPLES FOR TEM ANALYSIS

Author: Ron Mahoney, Ed Cahill EMSL Analytical, Inc.

SYNOPSIS: A standardized method is presented for indirect preparation of air and dust samples for analysis by TEM.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	[illegible]	1/23/07

REVISION LOG

Revision	Date	Reason
0	11/28/06	--
1	1/23/07	Clarification of filter configuration, secondary and tertiary dilution procedures.

1.0 PURPOSE

Some air samples collected at the Libby Superfund site are overloaded with debris and/or have obvious non-uniform loading, so analysis for asbestos by Transmission Electron Microscopy (TEM) requires an indirect preparation of the sample. All dust samples collected at the Libby Superfund site are prepared for TEM analysis using an indirect preparation. The purpose of this SOP is to provide a standardized procedure for the indirect preparation of air and dust samples that minimizes the loss of sensitivity. In addition, this SOP allows for the retention of a portion of the original air sample filter for archive whenever possible.

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that all laboratories participating in the analysis of air samples at the Libby site are aware of this SOP and that all analysts follow this SOP. Laboratory managers and analysts are responsible for communicating to the Libby laboratory coordinator (CDM), Volpe Center and appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist any recommended changes or proposed improvements to the SOP.

3.0 EQUIPMENT

Equipment needed to perform indirect preparations of air samples includes the following:

- Transmission electron microscope (NVLAP compliant)
- Energy dispersive X-ray system (NVLAP compliant)
- High vacuum carbon evaporator with rotating stage
- HEPA hood (NVLAP compliant)
- Exhaust or fume hood
- Particle-free water
- Glass container for ashing
- Disposable single use containers of at least 100 ml capacity
- Waterproof marker
- Forceps
- Ultrasonic bath
- Appropriate disposable glass or variable pipets with disposable tips
- Disposable 25 mm filter funnels
- Side arm filter flask
- Cellulose support pad, 25 mm diameter
- MCE filters, 25 mm diameter, $\leq$ 0.22 µm and 5.0 µm pore size
- Storage container for 25 mm filter
- Glass slides, approximately 25 x 76 mm in size
- Scalpel blades, # 10 or equivalent and handle
- Desiccator or low temperature drying oven
- Acetone, reagent grade
- Glacial acetic acid

- Plasma asher, low temperature
- pH paper
- Tygon tubing, or equivalent
- Small vacuum pump for filtration
- Glass petri dishes
- Jaffe washer
- Carbon evaporator rods
- Wash bottles, plastic
- Reagent alcohol

4.0 METHOD SUMMARY

Figure 1 presents a simplified overview of the TEM indirect preparation procedure for overloaded air samples and dust samples. As seen, there are two general indirect preparation procedures, one that includes ashing of the primary filter and one that does not include ashing of the primary filter.

Laboratory modification LB-000053 provides a list of which sample prefix codes shall be prepared using an ashing procedure and which should not be prepared using an ashing procedure. In cases where there is a conflict regarding sample type between the sample prefix as defined by the most recent version of LB-000053 and the chain of custody instructions, the chain of custody instructions take precedent. Additionally, once sample preparations have begun, there may be cases where the analyst determines that ashing is necessary to obtain acceptable filter loading. Samples for which ashing may be warranted include indoor air or dust samples collected from properties with elevated levels of organic particulates (e.g., due to cigarette smoke or use of a wood-burning stove). In these samples, ashing may further reduce particulate loading, thus allowing for an improved analytical sensitivity.

The sections below present the detailed steps associated with each procedure. For all indirect preparations, specimen preparation should be performed in a clean facility that is separate from both bulk and air preparation areas and preparation shall take place in a negative flow HEPA hood to prevent any possible contamination of the laboratory or personnel.

4.1 PROCEDURE 1: Indirect Preparation with Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should be performed. For the purpose of the Libby Superfund Site, air samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ISO 13794, but has been modified to increase the total solution volume from 40 ml to 100 ml and to retain a portion of the original filter. The use of a 100 ml final volume is selected because it allows for preparation of a

series of indirect samples with volumes that are sufficiently large that secondary dilution is not needed to ensure uniform deposition on the filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is similar to the method specified in ASTM D-5755, but has been modified to include an ashing of the primary filter.

4.1.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.

4.1.2 Within a safety hood, carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.1.6.**

4.1.3 Any loose material that is present in the cassette should be poured into a disposable 50 ml glass beaker or similar container.

4.1.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it in the same disposable 50 ml glass beaker or similar container with the side containing the sample facing down.

4.1.5 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl into a new 25 mm diameter disposable filtration funnels. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Apply vacuum. When all solution has passed through, rinse sides of filter funnel with a stream of particle free water to dislodge any particulate that might be adhering to the sides of the filter funnel. Once filtration is complete turn off vacuum, remove filter from unit and dry. Once the filter is dry, place it in the container with the original filter and **proceed to Step 4.1.8**.

4.1.6 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.

4.1.7 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter

in a clean, single use disposable glass container with the side containing the sample facing down.

4.1.8 Cover the container with aluminum foil, forming a tight seal around the mouth.

4.1.9 Perforate the foil in 15-20 places with a syringe needle to allow for gas exchange during plasma ashing.

4.1.10 Place the sample container in the plasma asher chamber. Depending on the size of the plasma asher chamber, several samples may be ashed simultaneously.

4.1.11 Operate the plasma asher using the minimum power at which a glow-discharge is observed, until the filter appears to be completely ashed. Loss of particulate and fibers from the container will occur if the plasma asher is operated at excessive radio-frequency power. During ashing of mixed cellulose ester (MCE) filters, a critical point is reached during the oxidation at which a sudden, violent ignition may occur if the radio-frequency power is excessive. This may result in a loss of fibers from the container, contamination of the interior of the chamber, and possible cross-contamination of the samples. For this reason, ashing of the blank should be observed closely during the early stages of oxidation, in order to ensure that the radio-frequency power setting is such that sudden ignition does not occur.

4.1.12 After 100% ashing is complete based on visual observation, increase the plasma asher power to maximum and ash for a period of one additional hour.

4.1.13
While final ashing is in progress, set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm diameter disposable filtration funnels shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied to help ensure an even distribution of particulate on the filter. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and applying a light vacuum. This will ensure that the filters are flat and that there are no air bubbles.

4.1.14 After ashing is complete, admit air slowly to the chamber and remove the samples from the plasma asher chamber and place back into a safety hood.

4.1.15 Remove the aluminum foil from the top of the sample container.

4.1.16 Using particle free water in a squirt bottle, carefully rinse the ashed residue from the ashing container into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. Rinse the residue into the 100 ml container to an initial volume of approximately 90 ml. Adjust pH to approximately 3-4 using a 10% solution of glacial acetic acid, and checking with pH paper. Bring the final volume to 100 ml and cap tightly.

4.1.17 Briefly hand shake (3 seconds) the capped container containing the sample suspension.

4.1.18 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes. The liquid level in the bath should be ½ to ¾ the height of the sample containers. Wipe the outside of the sample containers dry when removing them from the bath.

4.1.19 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.

4.1.20 For each sample, prepare three secondary filters by applying volumes of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation proves to be overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series, either a greater or lesser volume, in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.1.21), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.

4.1.21 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.

4.1.22 Disassemble the filtration units. Carefully remove the filters from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place each filter in a labeled petri dish or other similar container, active side up and dry.

4.1.23 Select the secondary filter from the dilution series yielding the largest possible f-factor (highest possible volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.

4.1.24 Perform a standard TEM sample preparation procedure.

4.1.25 If TEM examination of the lowest volume aliquot filtered is deemed overloaded (>25% particulate), consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.

4.1.26 Carefully label and place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.

4.1.27 Place any remaining sample solution in a graduated cylinder or pipet. The largest known quantity of the remaining solution should be filtered through a 25 mm disposable filtration unit with a $\leq$ 0.22 µm/5.0 µm pore size MCE filter set in conjunction with a cellulose support pad and dried after removal from the filtration unit. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. The dried filter shall be placed in an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.

4.1.28 Discard the remaining portion of the sample solution using standard laboratory protocols.

4.2 PROCEDURE 2: Indirect Preparation without Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should not be performed. For the purpose of the Libby Superfund Site, samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ASTM D-5755, but has been modified to allow for an archive of the original filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is equivalent to the method specified in ASTM D-5755.

4.2.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.

4.2.2 Carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.2.5**.

4.2.3 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl down onto the sample collection filter (still inside the sampling cassette).

4.2.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. **Proceed to Step 4.2.7**.

4.2.5 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.

4.2.6 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter in a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup.

4.2.7 Bring the total volume of the suspension up to approximately 90 ml using particle-free water only.

4.2.8 Adjust the suspension to a pH of 3-4 using a 10 % solution of acetic acid. Use pH paper to test.

4.2.9 Bring the total volume up to 100 ml using particle-free water and cap tightly.

4.2.10 Set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm disposable filtration funnels (such as Whatman cat. #:1922-1820) shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus, prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied resulting in even distribution. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and

applying a light vacuum. This will assure that the filters are flat and that there are no air bubbles. Ensure that suspension is filtered within 24 hours to avoid problems associated with bacterial and fungal growth.

4.2.11 Briefly hand shake (3 seconds) the capped container containing the sample suspension.

4.2.12 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes.

4.2.13 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.

4.2.14 For each sample, prepare three secondary filters by drawing aliquots of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation is overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.2.15), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.

4.2.15 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.

4.2.16 Disassemble the filtration unit. Carefully remove the filter from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place the filter in a labeled petri dish or other similar container, active side up and dry.

4.2.17 Select the secondary filter from the dilution yielding the largest possible f-factor (highest volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.

4.2.18 Perform a standard TEM sample preparation procedure.

4.2.19 If TEM examination of the lowest volume aliquot filtered is deemed overloaded, consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.

4.2.20 Place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.

4.2.21 Place any remaining sample solution in a graduated cylinder or pipet and add to a prepared 25 mm filtration unit containing a $\leq$ 0.22 µm/5.0 µm pore size filter set with a cellulose support pad in a disposable filtration unit with a small volume of particle free water to facilitate the production of a homogeneous solution and record the volume of sample solution added. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. Add 10 ml particle free water to the sample container containing the residual filter and sonicate for three minutes. Add this solution to the filtration unit for the corresponding filtration unit for each sample as described in the first part of this paragraph. Do not include this 10 ml in the volume calculation of the sample solution added. This solution should then be filtered through the filtration unit and dried after removal from the filtration unit. The dried filter shall be placed an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.

4.2.22 Discard the remaining portion of the sample solution using standard laboratory protocols.

5.0 DOCUMENTATION AND ARCHIVE STORAGE

Project-specific Index IDs are recorded on all air samples. During each indirect preparation step, this Index ID is noted on the sample-specific beakers, containers, and filtration units.

In those cases where no loose material is present in the cassette or adhering to the cowl, the remaining portion of the original primary filter is placed in a suitable container and clearly labeled with the sample number and indicated that it is the original primary filter. In those cases where secondary or tertiary filters are prepared, all filters or remnants of filters will be archived into suitable containers, and clearly labeled with the sample number and the volume of the aliquot applied to each filter.

Analysis-specific details about the indirect preparation will be recorded in the sample TEM electronic data deliverable (EDD) spreadsheet. In the TEM EDD, if the sample is prepared using Procedure 1 (see Section 4.1) the preparation method should be identified as “IA – Indirect, ashed” and the appropriate inputs should be recorded in the fields provided. If the sample is prepared using Procedure 2 (see Section 4.2), the preparation method should be identified as “I – Indirect” and the appropriate inputs should be recorded in the fields provided. The spreadsheet is designed to automatically calculate the dilution factor, or f-factor, which is used in the calculation the sample air or dust concentration.

6.0 QUALITY ASSURANCE

All quality control sample results will be monitored for potential contamination. If sample results indicate cross-contamination, the laboratory will identify the affected samples and notify the USEPA Regional Chemist and project laboratory coordinator (CDM). Laboratory procedures will be re-assessed and appropriate changes will be made and documented accordingly by the project laboratory coordinator.

6.1 Lot Blanks

All cassettes utilized in the Libby project are screened for contamination by either TEM analysis or a combination of TEM and PCM analysis. One lot blank is prepared and analyzed from each carton of cassettes prior to using the lot of cassettes for sampling. The entire carton of cassettes will be rejected if any asbestos fiber is detected on the lot blank.

6.2 Filter blanks

Prior to filtration of the sample aliquot, 100ml particle-free water should be filtered. Acceptance criteria for filter blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

6.3 Plasma asher blanks

To ensure that contamination is not introduced during the ashing process, a container with an unused filter should be run as a blank with each batch of samples ashed. This sample will be prepared using the standard TEM sample preparation procedure and examined as per the established QC sequence. Acceptance criteria for plasma asher blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

7.0 DECONTAMINATION

All non-disposable equipment used during sample preparation must be decontaminated prior to use. Because the prescribed filtration units used to prepare the secondary filters are disposable, decontamination of filtration units is not required.

8.0 GLOSSARY

EDD - Electronic Data Deliverable. A Libby-specific spreadsheet designed to capture the detailed analysis and raw structure data generated during a TEM analysis. Contact the project laboratory coordinator (CDM) for the current TEM spreadsheet version.

HEPA - High Efficiency Particulate Air

MCE - Mixed Cellulose Ester

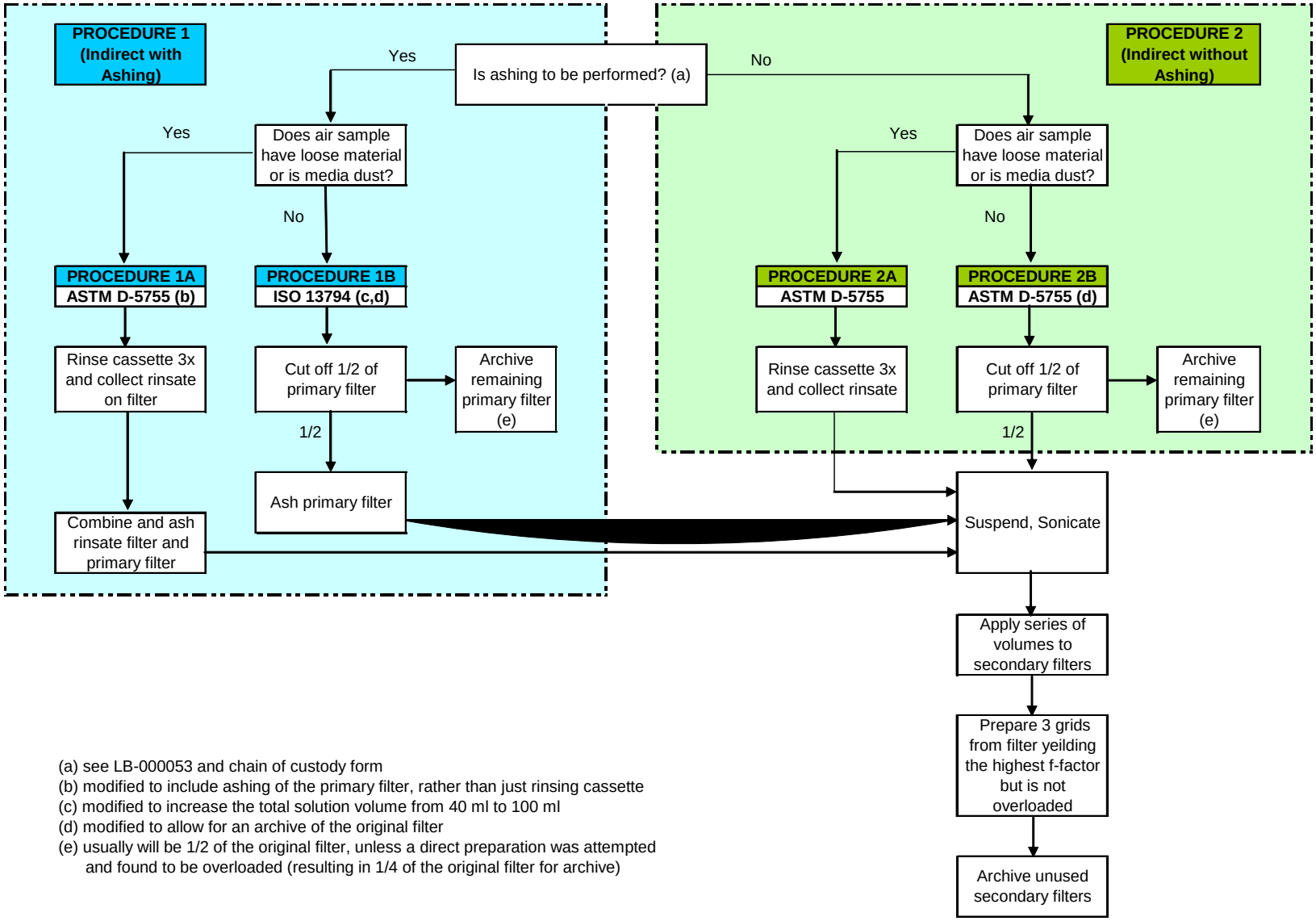
TEM - Transmission Electron Microscopy

9.0 REFERENCES

ISO 13794. Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method. International Organization for Standardization (ISO) 13794:1999. November 15, 1999.

ASTM D-5755. Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D 5755-03. October 2003.

FIGURE 1. INDIRECT PREPARATION OF OVERLOADED AIR SAMPLES AND DUST SAMPLES FOR TEM ANALYSIS



(a) see LB-000053 and chain of custody form
(b) modified to include ashing of the primary filter, rather than just rinsing cassette
(c) modified to increase the total solution volume from 40 ml to 100 ml
(d) modified to allow for an archive of the original filter
(e) usually will be 1/2 of the original filter, unless a direct preparation was attempted and found to be overloaded (resulting in 1/4 of the original filter for archive)

(LB-000016) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000016

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, [TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:_____

Requester: Jeanne Orr Title: President
Company: Reservoirs Environmental, Inc. Date: December 2, 2002

Description of Modification:
Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using ISO 10312. The purpose of the attached is to document permanent historic modifications & clarifications.

Reason for Modification:
To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:
Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by TEM analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
Please see the attached for the description of the TEM-ISO clarifications/modifications

Technical Review: R.K. Mahony EMSL Date: 23 April 2003
(Laboratory Manager or designate)

Project Review and Approval: [signature] Date: 4 April 2003
(Volpe: Mark Raney)

Approved By: [signature] Title: Project Chemist Date: 3 April 2003
(USEPA: Mary Goldade)

1. **Modification:**
   The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.
2. **Modification:**

   ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

3. **Clarification:**
   Stopping rules for ISO analyses are completion of the grid opening on which the 100th asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

   If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100th chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6*.
   This clarification in structure counting and recording is <u>to provide consistency in analytical procedures and data recording in the project laboratories.</u>

4. **Modifications and clarifications: Structure counting and recording**
   a. **Modification:** Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
   b. **Modification:** The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
   c. **Modification:** Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
   d. **Modification:** If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
   e. **Clarification:** If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

   These modifications and clarifications in structure counting and recording are <u>to provide consistency in analytical procedures and data recording in the project laboratories.</u>

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Tuesday, April 22, 2003 11:09 AM
To: 'Mahoney, Ron'
Subject: FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017

LB-000015_rev (MR 4-4-03 email... LB-000016_rev (MR 4-4-03 email... LB-000017_rev (MR 4-4-03 email...

FYI

> -----Original Message-----
> From: Raney, Mark
> Sent: Friday, April 04, 2003 9:31 AM
> To: 'Beckham, Richard'; 'Goldade.mary@EPAmail.epa.gov'; 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017
>
>
> Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).
>
> * The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"
> * Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."
>
> If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.
>
> Mark.
>
> > <<LB-000015_rev (MR 4-4-03 email).doc>> > > <<LB-000016_rev (MR 4-4-03 email).doc>> > > <<LB-000017_rev (MR 4-4-03 email).doc>>
>
>
> -----Original Message-----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Tuesday, April 01, 2003 10:47 AM
> To: 'Goldade.mary@EPAmail.epa.gov'; 'RANEY@VOLPE.DOT.GOV';
> 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: FW: LB-000015, LB-000016, and LB-000017
>
>
> For your review and approval.
>
> - Richard Beckham
>
> -----Original Message-----
> From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
> Sent: Monday, March 31, 2003 6:11 PM
> To: Beckham, Richard
> Subject: LB-000015, LB-000016, and LB-000017
>
>
> Richard,
>
> These should be final. The only recent revision is the addition of the
> Effective Date. These need to go to Mark and Mary for their final blessing.

> <<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
> <<LB-000017 rev(3_31_03).doc>>
>
> R.K. Mahoney
> Senior Analyst
> Special Projects Coordinator
> EMSL Analytical, Inc.
> Westmont, NJ
> 800.220.3675, x1218
> rmahoney@emsl.com
>
> << File: LB-000015(rev 3_31_03).doc >> << File: LB-000016 rev. (3_31_03).doc >> << File: LB-000017 rev(3_31_03).doc >>

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Wednesday, April 23, 2003 9:02 AM
To: 'Mahoney, Ron'
Subject: FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

LB-000015(rev 3_31_03).doc LB-000016 rev. (3_31_03).doc LB-000017 rev(3_31_03).doc

Ron,

I almost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs LB-15, 16, & 17.

Let me know if you have any questions.

Mark.

----Original Message----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, April 03, 2003 5:49 PM
To: Beckham, Richard
Cc: Autio, Anni; 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'
Subject: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

Richard,
Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date is: Historical.
EPA approves these mods with this changed completed.

"Beckham, Richard" <BeckhamRE@cdm.com>
To: Mary Goldade/EPR/R8/USEPA/US@EPA, '"RANEY@VOLPE.DOT.GOV"' <RANEY@VOLPE.DOT.GOV>, '"mgoldade@peakpeak.com"' <mgoldade@peakpeak.com>
cc: "Autio, Anni" <AutioAH@cdm.com>
Subject: FW: LB-000015, LB-000016, and LB-000017
04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

----Original Message----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Monday, March 31, 2003 6:11 PM
To: Beckham, Richard

Subject: LB-000015, LB-000016, and LB-000017

Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.
<<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
<<LB-000017 rev(3_31_03).doc>>

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

(See attached file: LB-000015(rev 3_31_03).doc)(See attached file: LB-000016 rev. (3_31_03).doc)(See attached file: LB-000017 rev(3_31_03).doc)

(LB-000019) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000019

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval. File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 21 January 2003

Description of Modification:
Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.

Reason for Modification:
The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the bench sheets as well as the electronically deliverables.

Potential Implications of this Modification:
There are no potential negative implications resulting from this clarification of terms.

Laboratory Applicability (circle one): All Individual(s) EMSL Analytical, Inc.

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 21 January 2003
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney Date: 27 March 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Raney Date: 7 March 2003
(Volpe: Mark Raney)

Approved By: Mary Goldade Date: 7 March 2003

Title: EPA Regional Chemist
(USEPS: Mary Goldade)

Lab Modification Form Revision 5

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Friday, March 07, 2003 2:50 PM
To: 'Beckham, Richard'; 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; Raney, Mark; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
Subject: RE: MOD LB-000019

I find Laboratory Request for Modification # LB-000019 acceptable as written and here by provide Volpe approval to this MOD.

Richard, Please make sure MOD ID#s get inserted onto the mod forms themselves (not just the file ID), so you will be able to identify the IDs based upon hardcopy alone. Also, even though this MOD is applicable to an individual lab, all MODs are to be forwarded to all labs for informational purposes and to give them an opportunity to provide comments. All labs however are REQUIRED to provide comments to only MODs that are applicable to all labs.

Mark Raney
Environmental Engineer

US DOT / Volpe Center
Environmental Engineering Division, DTS-33
phone: 617-494-2377
cell: 617-694-8223
fax: 617-494-2789
raney@volpe.dot.gov

-----Original Message-----
From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
Sent: Thursday, March 06, 2003 9:54 AM
To: 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
Subject: MOD LB-000019

This MOD impacts only EMSL. For your review and comment:

<<LB-000019.doc>>
- Richard Beckham

Mahoney, Ron

From: Mary Goldade [mgoldade@peakpeak.com]
Sent: Friday, March 07, 2003 12:29 PM
To: Raney, Mark
Cc: Jeff G. Montera; rmahoney@emsl.com; Autio, Anni; William Brattin; Goldade.Mary@epamail.epa.gov
Subject: Re: MOD LB-000019

I agree that this mod form is acceptable, and should be discussed on the next lab call to be certain similar issues are not encountered at other labs.
Mary
—— Original Message ——
From: "Raney, Mark" <RANEY@VOLPE.DOT.GOV>
To: "'Goldade, Mary (HOME)'" <mgoldade@peakpeak.com>
Sent: Friday, March 07, 2003 10:18 AM
Subject: FW: MOD LB-000019

>
> FYI
>
>
> -----Original Message-----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Thursday, March 06, 2003 9:54 AM
> To: 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio,
> Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com';
> 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
> Subject: MOD LB-000019
>
>
> This MOD impacts only EMSL. For your review and comment:
>
> <<LB-000019.doc>>
> - Richard Beckham
>
>
>

(LB-000024) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000024

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 13 March 2003

Description of Modification:
In addition to the traditional asbestos minerals, those comprising the Libby Amphibole complex will also be considered applicable analytes. As of December 16, 2002 samples of 0.2 % and 1.2 % by weight Libby amphibole bulk reference materials, prepared by the USGS, Denver (for use during ISTM2), are used as comparison materials for quantification of soil samples. Also, results will be categorized into four bins: "A" None Detected, "B1" asbestos detected but determined to be < or = 0.2%, "B2" asbestos detected but determined to be > 0.2% and <1.0 %, and "C" = or > 1.0 %. Results will be reported as "A" – None Detected, "B1" – Trace, "B2" - < 1.0 %, and "C" – will be reported as a whole number percent.

Reason for Modification:
This modification has been implemented to facilitate the more precise quantification of the relatively low levels of Libby amphibole found in soil samples from the Libby, MT site.

Potential Implications of this Modification:
There are no potential negative implications resulting from this clarification of data enumeration, recording, and reporting formats.

Laboratory Applicability (circle one): All Individual(s)

Duration of Modification (circle one):
Temporary Date(s):
Analytical Batch ID:
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: December 16, 2002
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney Date: 27 March 2003
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 14 March 2003
(Volpe: Mark Raney)

Approved By: Mary Goldade Title: EPA Regional Chemist Date: 25 March 2003
(USEPA: Mary Goldade)

Mahoney, Ron

From: Goldade.Mary@epamail.epa.gov
Sent: Tuesday, March 25, 2003 11:09 AM
To: Beckham, Richard
Cc: Autio, Anni; 'Bill Egeland'; 'Bob.Shumate@battaenv.com'; 'brattin@syrres.com'; 'Charlie LaCerra'; 'corbin77@atc-enviro.com'; 'dmazzaferro@mastest.com'; 'Gustavo Delgado'; 'Garth B. Freeman'; 'jeanneorr@resienv.com'; 'mgoldade@peakpeak.com'; 'Naresh C. Batta'; 'Raney@volpe.dot.gov'; 'rdemalo@emsl.com'; 'rhatfield@mastest.com'; 'rmahoney@emsl.com'; 'Shu-Chun Su'; 'William Longo'
Subject: EPA APPROVED MOD: LB-000024

LB-000024_rev (3-25-03 email).... pic19067.gif

The newly attached document is acceptable w/ changes. EPA approves.
(See attached file: LB-000024_rev (3-25-03 email).doc)
(Embedded image moved to file: pic19067.gif)

"Beckham, Richard" <BeckhamRE@cdm.com>
03/14/03 08:30 AM

To: 'Charlie LaCerra' <clacerra@emsl.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'William Longo' <wlongo@mastest.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, 'Shu-Chun Su' <scsu@delanet.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'brattin@syrres.com'" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>
cc:
Subject: LB-000024

This MOD impacts all labs. For your review and comment:

<<LB-000024.doc>>
- Richard Beckham

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Friday, March 14, 2003 12:37 PM
To: 'Beckham, Richard'; 'Charlie LaCerra'; 'jeanneorr@resienv.com'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; 'William Longo'; 'rhatfield@mastest.com'; 'Bill Egeland'; 'Bob.Shumate@battaenv.com'; 'Naresh C. Batta'; 'Shu-Chun Su'; 'corbin77@atc-enviro.com'; 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; Raney, Mark; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; 'dmazzaferro@mastest.com'; 'mgoldade@peakpeak.com'
Subject: LB-000018, 20_rev, 21, 22_rev, 23, & 24

LB-000024_rev (3-14-03 email)....

Below are the results of my review of the following Requests for Modifications to Laboratory Activities:

LB-000018
* Missing info, "Potential Implications of this Modification" is a required field and must be filled out even if it is to say the modification will have no implication on results, etc.
* "Laboratory Applicability" should be "Individual" NOT "All", since it only applies to Hygeia.
* Duration of Modification" should be "Temporary" not "Permanent" since the modification impacted only two jobs.

LB-000020_rev
* Looks good, I provide Volpe's approval as is.

LB-000021
* "Laboratory Applicability" should be Individual NOT All, since it only applies to Hygeia.

LB-000022_rev
It appears this revised version was provided prior to receiving my earlier comments, which still apply (see below):
* Under "Description of Modification" make it clear that the standards are from "ISTM2"
* Add: when completing data entry into the EDD instead of inputting a "B" or "T" into the "Ref Material (B or T)" field of the "Visual_data entry" tab the Labs should input "ISTM"
* Note: for this and other future TEMPORARY MODs, the MOD should not recommend proposing a written modification to the SOP (Method) itself. There is no reason to revise the method for temporary modifications.

LB-000023
* Does this MOD also affect TEM dust results? Were any dust analysis performed by Hygeia between 6/1/02 and 11/30/02?
* "Laboratory Applicability" should be "Individual" NOT "All", since it only applies to Hygeia.

LB-000024
See the attached revised MOD, where the following changes have been made:
* This MOD should be a permanent, rather than a temporary MOD, since some soils may still be analyzed via NIOSH 9002 in the future, such as at the mobile lab, etc. (i.e., applicable from 12/16/2002 forward)
* Under "Description of Modification": (1) specified reference materials as "ISTM2"; (2) specified for comparison materials for quantification "of soil samples"; and (3) clarified Bin "B2" is for <1.0% and Bin "C" is for = or > 1.0% and reported as a whole number (same Bin classifications as stated within SOP SRC-Libby-03 Rev. 0).

Please respond (reply ALL) with any questions or comments to the above points.

Mark.

<<LB-000024_rev (3-14-03 email).doc>>

(LB-000024a) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000024A

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: SRC-Libby-03 (Revision 1)

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 10 December 2003

Description of Modification:
In addition to the traditional asbestos minerals, those comprising the Libby Amphibole complex will also be considered applicable analytes. As of December 16, 2002 samples of 0.2 % and 1.2 % by weight Libby amphibole bulk reference materials, prepared by the USGS, Denver (for use during ISTM2), are used as comparison materials for quantification of soil samples. Also, results will be categorized into four bins: "A" None Detected, "B1" asbestos detected but determined to be < 0.2%, "B2" asbestos detected but determined to be > or = to 0.2% and <1.0 %, and "C" = or > 1.0 %. Results will be reported as "A" – None Detected, "B1" – Trace, "B2" - < 1.0 %, and "C" – will be reported as a whole number percent.

Reason for Modification:
This modification has been implemented to facilitate the more precise quantification of the relatively low levels of Libby amphibole found in soil samples from the Libby, MT site.

Potential Implications of this Modification:
There are no potential negative implications resulting from this clarification of data enumeration, recording, and reporting formats.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: 16 December 2002
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney Date: 6 January 2004
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 12-31-03
(Volpe: Mark Raney)

Approved By: _____ Title: Project Chemist Date: 2/5/04
(USEPA: Mary Goldade)

EMSL Mobile Lab - Asbestos

From: "Raney, Mark" <RANEY@VOLPE.DOT.GOV>
To: '"EMSL Mobile Lab - Asbestos"' <mobileasbestoslab@emsl.com>
Cc: '"Kwiatkowski, Joseph"' <KwiatkowskiJJ@cdm.com>; '"Autio, Anni"' <autioah@cdm.com>
Sent: Wednesday, December 31, 2003 7:42 AM
Subject: RE: Comments Lab Mod LB-000024A

Ron,

I concur with Mary and provide Volpe's approval of Mod LB-00024A as written. Please distribute through the signature process.

Thanks,

Mark.

-----Original Message-----
From: EMSL Mobile Lab - Asbestos [mailto:mobileasbestoslab@emsl.com]
Sent: Monday, December 29, 2003 4:45 PM
To: Mark Raney
Subject: Fw: Comments Lab Mod LB-000024A

Hi Mark,

I hope you are having a Happy Holiday Season. Just a reminder re mod LB-000024A . If I could get email comments from you, we can put this puppy to bed.

Here is the original email with Mary's comments. Also, on the original I left on the approval dates from the first time that will have to be changed.

Talk to you tomorrow.

Ron
----- Original Message -----
From: <Goldade.Mary@epamail.epa.gov>
To: "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@emsl.com>
Cc: "Anni Autio" <autioah@cdm.com>; "Charlie LaCerra" <clacerra@emsl.com>; "Kim Carr" <kcarr@emsl.com>; "Mark Raney" <Raney@volpe.dot.gov>; "Rob DeMalo" <rdemalo@emsl.com>; <brattin@syrres.com>; "Bill Longo (E-mail)" <wlongo@mastest.com>; "Bob Shumate (E-mail)" <bob.shumate@battaenv.com>; "Denise Mazzaferro (E-mail)" <dmazzaferro@mastest.com>; "Gustavo Delgado (E-mail)" <gdelgado77@atc-enviro.com>; "Jeanne Orr (E-mail)" <jeanneorr@resienv.com>; "Kwiatkowski, Joseph" <KwiatkowskiJJ@cdm.com>; "Kyeong Corbin (E-mail)" <corbin77@atc-enviro.com>; "Shu-Chun Su (E-mail)" <scsu@delanet.com>
Sent: Thursday, December 11, 2003 7:09 AM
Subject: Re: Comments Lab Mod LB-000024A

>
>
>
>
> EPA approves this mod as written which was written as a
> clarification/stop gap for the NIOSH 9002 method while we were
> finalizing the SRC SOP.
>
> As I look at this mod. w/ Ron's 2nd question in mind, I believe we
> should refer to SRC-Libby-03. The SOP is vague about what to do w/
> readings at 0.2%.
> Bill, please prepare a new mod number that will clarify the binning
> process for concs at 0.2%. That is, prepare a modification form that
> describes B2 as "Asbestos was observed in the field sample at a level
> that appeared to be at or above the 0.2% reference material but was less
> than the 1% reference material."
> Thanks,
> (Embedded image moved to file: pic22648.gif)
>
>
>
> EMSL Mobile Lab -
> Asbestos To: Mark Raney
<Raney@volpe.dot.gov>, Mary Goldade/EPR/R8/USEPA/US@EPA, Anni
> <mobileasbestosla Autio <autioah@cdm.com>
> b@emsl.com> cc: Charlie LaCerra
<clacerra@emsl.com>, Kim Carr <kcarr@emsl.com>, Rob DeMalo
> <rdemalo@emsl.com>
> 12/10/03 02:10 PM Subject: Lab Mod
LB-000024A
>
>
>
>
>
>
> Attached you will find lab mod LB-000024A for your review.
>
> Please consider two points. Should the effective date remain 16
> December
> 2002? Mary and I think yes. Another point is should the applicable
> method
> cite SRC-Libby-03 (Revision 0) instead of and/or in addition to
> PLM-NIOSH
> 9002?
>
> Ron
>
> EMSL Mobile Asbestos Lab

> 107 W 4th St.
> Libby, MT 59923
> PH: (406) 293-9066
> FAX: (406) 293-7016
> http://www.emsl.com
> (See attached file: LB-000024A.doc)
>
>

(LB-000028) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000028

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator
Company: EMSL Analytical, Inc. Date: 17 June 2003

Description of Modification:
This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

Reason for Modification:
This clarification is intended to provide more complete TEM re-analysis data.

Potential Implications of this Modification:
There are no negative implications to this clarification.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 17June 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Rance Date: 7/18/03
(Volpe: Project Technical Lead or designate)

Approved By: Mary Goldade Date: 6/24/03

Title: Project Chemist
(USEPA: Project Chemist or designate)

Mary Goldade

06/24/03 01:20 PM

To: "Beckham, Richard" <BeckhamRE@cdm.com>
cc: "Autio, Anni" <AutioAH@cdm.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'brattin@syrres.com'" <brattin@syrres.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'Shu-Chun Su' <scsu@delanet.com>, 'William Longo' <wlongo@mastest.com>
Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.

LB-000028 (MG 6-24-03).

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

"Beckham, Richard" <BeckhamRE@cdm.com>

"Beckham, Richard" <BeckhamRE@cdm.com>

06/23/03 08:42 AM

To: 'Charlie LaCerra' <clacerra@emsl.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'William Longo' <wlongo@mastest.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, 'Shu-Chun Su' <scsu@delanet.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'brattin@syrres.com'" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>
cc:

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

From: "LaCerra, Charles" <CLaCerra@EMSL.com>
To: "Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>
Sent: Friday, July 18, 2003 5:57 AM
Attach: LB-000025_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-
Subject: FW: MODs: LB-000025, 26, 27 & 28

-----Original Message-----
From: Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]
Sent: Friday, July 18, 2003 7:53 AM
To: 'Beckham, Richard'; Autio, Anni
Cc: 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'
Subject: MODs: LB-000025, 26, 27 & 28

Richard,

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

-----Original Message-----
From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
Sent: Wednesday, July 16, 2003 5:30 PM
To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni
Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able
to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on
7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of 28
has not been prepared.

- Richard Beckham

-----Original Message-----
From: Raney, Mark
Sent: Wednesday, June 18, 2003 10:56 AM
To: 'Mahoney, Ron'
Cc: 'Anni Autio'; 'Mary Goldade'
Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e,. < 20).

Thanks,

Mark.

-----Original Message-----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Wednesday, June 04, 2003 9:27 AM
To: 'Mark Raney'

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF
Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of
the number of samples involved, and we agreed on < 20. The number is more
likely < 10, but we've deceided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

-----Original Message-----
From: Mary Goldade [mailto:mgoldade@peakpeak.com]
Sent: Wednesday, May 14, 2003 6:32 PM
To: Beckham, Richard; 'Charlie LaCerra'; jeanneorr@resienv.com; rdemalo@emsl.com; rmahoney@emsl.com; 'William Longo'; rhatfield@mastest.com; 'Bill Egeland'; Bob.Shumate@battaenv.com; 'Naresh C. Batta'; 'Shu-Chun Su'; corbin77@atc-enviro.com; 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; Raney@volpe.dot.gov; brattin@syrres.com; Goldade.mary@EPAmail.epa.gov; dmazzaferro@mastest.com; m_szynskie@resienv.com
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.
Ron-Do you already have in hand an estimate regarding the actual number of
samples this affects (i.e., are you able to quantify the term "few/limited"?)
Thanks,
Mary

----- Original Message -----
From: "Beckham, Richard" <BeckhamRE@cdm.com>
To: "'Charlie LaCerra'" <clacerra@emsl.com>; <jeanneorr@resienv.com>; <rdemalo@emsl.com>; <rmahoney@emsl.com>; "'William Longo'" <wlongo@mastest.com>; <rhatfield@mastest.com>; "'Bill Egeland'" <begeland@mastest.com>; <Bob.Shumate@battaenv.com>; "'Naresh C. Batta'" <ncbatta@battaenv.com>; "'Shu-Chun Su'" <scsu@delanet.com>;

<corbin77@atc-enviro.com>; "'Gustavo Delgado'"
<gdelgado77@atc-enviro.com>;
"'Garth B. Freeman'" <gfreeman@mastest.com>; "Autio, Anni"
<AutioAH@cdm.com>; <Raney@volpe.dot.gov>; <brattin@syrres.com>;
<Goldade.mary@EPAmail.epa.gov>; <dmazzaferro@mastest.com>;
<mgoldade@peakpeak.com>; <m_szynskie@resienv.com>
Sent: Wednesday, May 14, 2003 3:28 PM
Subject: MOD LB-000025

> This MOD impacts only EMSL. For your review and comment:
>
> <<LB-000025.doc>>
> - Richard Beckham

<<LB-000025_rev (MG 6-04-03 email).doc>> <<LB-000027 (MG 6-24-03).doc>> <<LB-000028 (MG 6-24-03).doc>>
>
>
>

Mary Goldade

07/29/03 01:57 PM

To: Anni Autio
cc: Mark Raney
cc:
Subject: LB-000027 & LB-000028 are signed and mailed

Anni & Joe,
I have mail you the original copiew of the mods LB-000027 & LB-000028.
Several of the email approval pages were not provided. I attached them.

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

(LB-000029a) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000029a

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All other TEM methods, including: SOP EPA-LIBBY-03, SOP EPA-LIBBY-07, and EPA/600/R-94/134 (EPA 100.2).

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 18 November 2003

Description of Modification:
Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses (all media).

Reason for Modification:
This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: 11/19/03 (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: W J Brattin Date: 11/19/2003
(Laboratory Manager or designate)

Project Review and Approval: [signature] Date: 11/20/2003
(Volpe: Project Technical Lead or designate)

Approved By: [signature] Date: 11/18/2003
(USEPA: Project Chemist or designate)

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Reprep	1%
Verified analysis	1%
Interlab	0.5%
Total	10%

Each laboratory should prepare and analyze lab blanks, recount (same, different and verified), and reprep samples selected at random in accord with this table. Samples for interlab comparisons will be designated on the COC sheets accompanying the samples.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: the procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Re-Analysis.

All re-analysis samples (same, different, interlab, and verified) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 2).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more errors, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Lab Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Re-Preparation.
Re-preparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 1. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% – 0.1%	0.2% - 0.5%	>0.5%
Recount samples	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrys vs amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Reprep	Concordance on LA count	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 1

STATISTICAL COMPARISON OF TWO POISSON RATES

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the re-preparation and re-analysis of a number of TEM grids for quantification of asbestos fiber concentrations in environmental media (air, dust, water, soil). Because of random variation, it is not expected that results from re-preparations samples should be identical. This appendix presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from the textbook entitled "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

Y1 = Fiber count in first evaluation
t1 = Number of grid openings in first evaluation
Y2 = Fiber count in second evaluation
t2 = Number of grid openings in second evaluation

The test is performed by following the following steps:

Step 1:

Calculate $Y = (Y1+Y2) / 2$
$t = (t1 + t2) / 2$
$\lambda = Y / t$

Step 2:

Calculate $Q = (Y1-Y)^2 / (\lambda \cdot t1) + (Y2-Y)^2 / (\lambda \cdot t2)$

Step 3:

Compare Q to the critical value of CHISQ(1-α,1) from the following table:

Alpha	CHISQ(1-α,1)
0.05	3.841
0.10	2.706
0.20	1.642
0.30	1.074

If Q is less than or equal to CHISQ(1-α,1), conclude that the two results are not statistically different at the 100(1-α)% confidence level.

If Q is greater than CHISQ(1-α,1), conclude that the two results are statistically different at the 100(1-α)% confidence level.

ATTACHMENT 2

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy-Version 2.0.

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

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Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. 1987, *52* (No. 210), 41826-41905.

[2]ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2-- The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5– The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6–4.5.7.

4.7 Complete the report form as described in items 4.7.1–4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6— The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
		F1 F2 F3 F4			
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

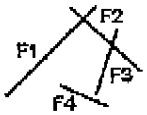
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

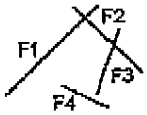
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

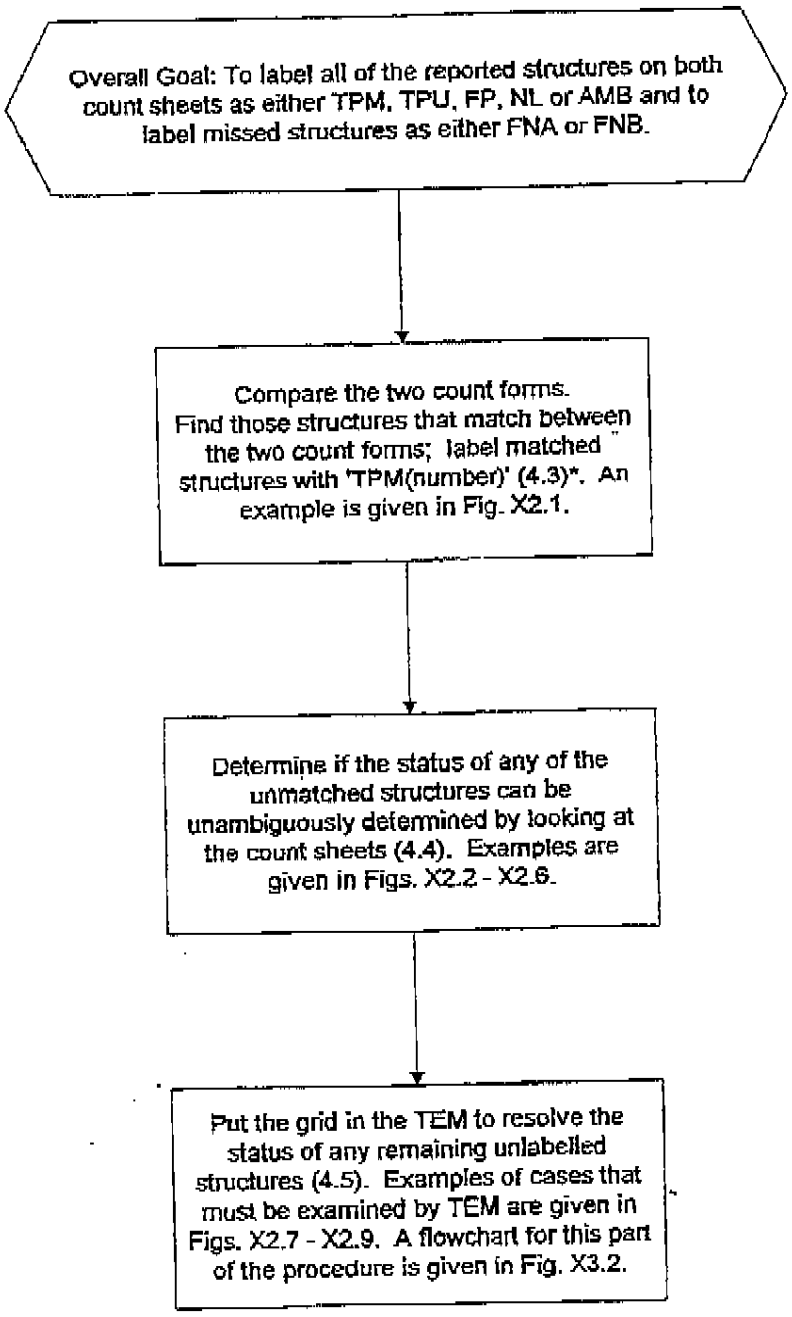


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.
*Numbers in parentheses in each block refer to the item number in the procedure.

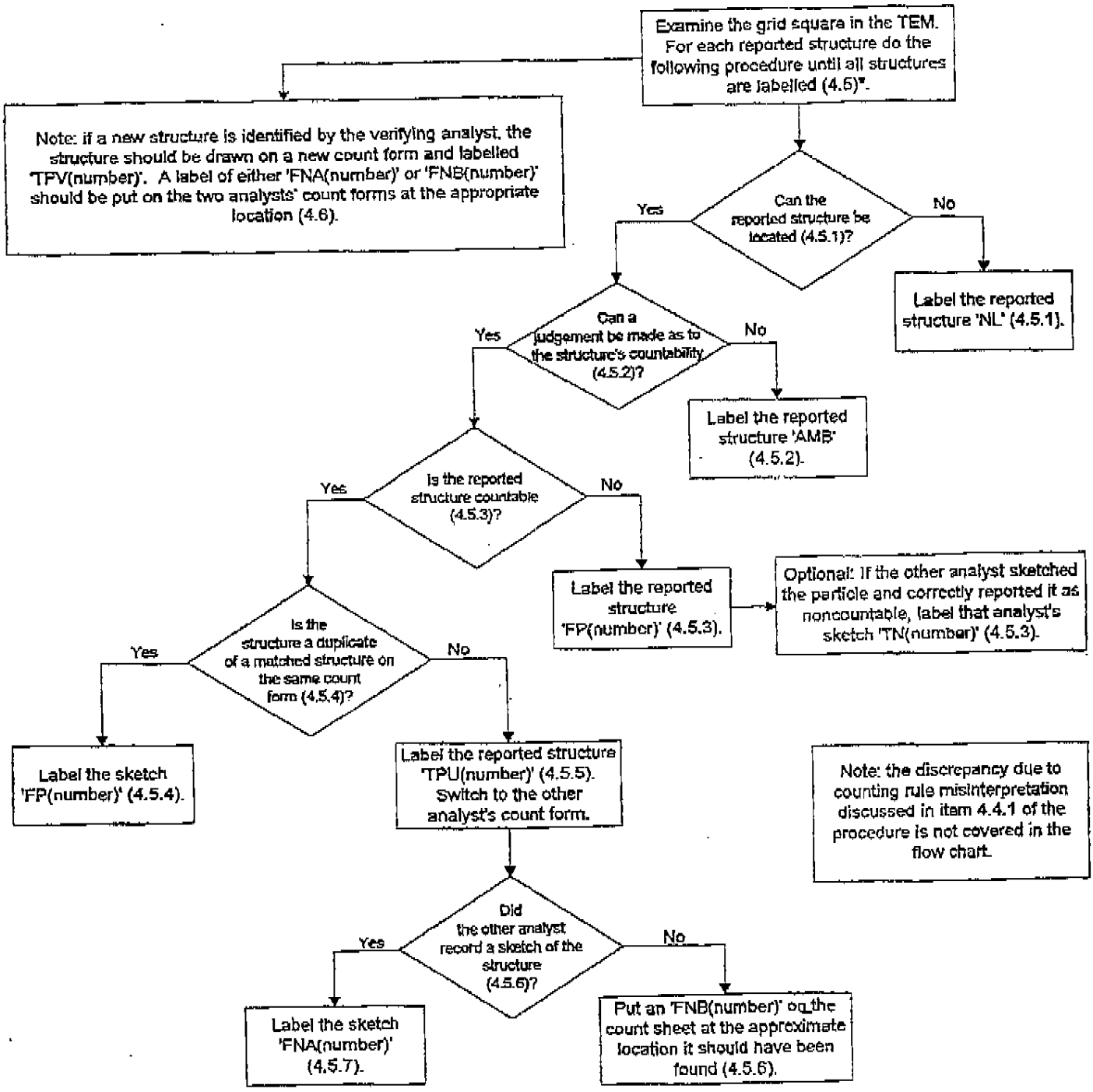


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

Mary Goldade

11/18/03 09:11 AM

To: William Brattin <brattin@syrres.com>
cc: Anni Autio <AutioAH@cdm.com>, Mark Raney <raney@volpe.dot.gov>
Subject: Re: EPA approved MOD LB-000029A

EPA approves MOD 29A w/ modifcations as attached.

LB-000029av1(MG 11-18-03).d

Mary Goldade
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William Brattin <brattin@syrres.com>

William Brattin <brattin@syrres.com >

11/04/03 02:28 PM

To: Mark Raney <raney@volpe.dot.gov>, "Mary Goldade (home)" <mgoldade@peakpeak.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, Anni Autio <AutioAH@cdm.com>
cc:
Subject:

Draft of Mod LB-000029a. I think it should go to labs for quick review before signing.

****
Bill Brattin
Syracuse Research Corporation
999 18th Street, Suite 1975
Denver CO 80202

Phone: 303-357-3121
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****

NISTIR 5351.pdf

"Raney, Mark" <RANEY@VOLPE.DOT.GOV>

11/20/03 11:19 AM

To: Mary Goldade/EPR/R8/USEPA/US@EPA, William Brattin <brattin@syrres.com>
cc: Anni Autio <AutioAH@cdm.com>, "Raney, Mark" <RANEY@VOLPE.DOT.GOV>
Subject: RE: EPA approved MOD LB-000029A

Bill,

Volpe concurs with Mary's comments to LB-000029a, please finalize and send through the signature process. I.e., make the necessary changes, print out, sign and date, retain a copy for your records, provide a copy to Anni and send the original for signature with all attachments including a hardcopy of Volpe and EPA's emailed approvals. Usually it goes to me first for signature, however since you are located with EPA provide it first to Mary, who will then forward to me for signature and I will close the loop.

Let me know if you have any questions.

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Tuesday, November 18, 2003 11:12 AM
To: William Brattin
Cc: Anni Autio; Mark Raney
Subject: Re: EPA approved MOD LB-000029A

EPA approves MOD 29A w/ modifcations as attached.
(See attached file: LB-000029av1(MG 11-18-03).doc)
(Embedded image moved to file: pic21954.gif)

William Brattin
<brattin@syrres.com>

11/04/03 02:28 PM

To: Mark Raney <raney@volpe.dot.gov>, "Mary Goldade (home)" <mgoldade@peakpeak.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, Anni Autio <AutioAH@cdm.com>
cc:
Subject:

Draft of Mod LB-000029a. I think it should go to labs for quick review before signing.

****
Bill Brattin
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e-mail: brattin@syrres.com
****
(See attached file: NISTIR 5351.pdf)

(LB-000029b) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical consultant
Company: Syracuse Research Corporation Date: December 7, 2006

Description of Modification:
Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

Reason for Modification:
This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: _____
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 4/25/07
(Volpe Project Technical Lead or designate)

Approved By: Mary Goldade Date: 4/25/07
(USEPA Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

QC Sample Type Definitions

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

Blanks

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Repreparations

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
Total	**10%**

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and repreparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and repreparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks.

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Recounts.

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Repreparations.
Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

ATTACHMENT 1

Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or repreparation.

For those laboratory jobs in which a recount or repreparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or repreparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). When selecting samples for repreparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with repreparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10th selection.

This selection procedure will ensure that the recount analyses and repreparations yield a dataset best suited to assess concordance[1].

Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.

2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

[1] It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

ATTACHMENT 2

Selection Procedure and Laboratory SOP for Interlabs (IL)

Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1st, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded[2].

2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 * 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.

3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated * the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.

4. Exclude samples in which the total number of GOs is more than 40 GOs[3]. Exclude any samples that have already been selected for interlab evaluation previously.

5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than to the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.

6. Submit list of selected interlab samples to the CDM Laboratory Manager.

7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

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[2] Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

[3] Because all interlabs will be reprepared, these interlab repreparation samples will also be evaluated for concordance with the original sample. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with repreparations.

Laboratory SOP

At the Originating Laboratory:

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.

2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).

5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby
EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.

10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).

11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

At the Interlab Laboratory:

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".

5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.

7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).

8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.

10. Ship the grid(s) back to the originating lab.

ATTACHMENT 3

Airborne Asbestos Method:
Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy-Version 2.0.
NIST (1994)

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

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Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. **1987**, *52* (No. 210), 41826-41905.

[2]ISO 10312 **1993**, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2-- The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator } 1) + TPU(\text{Operator } 2) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

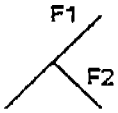
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

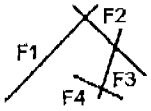
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

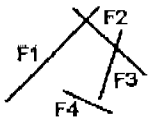
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

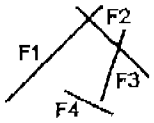
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

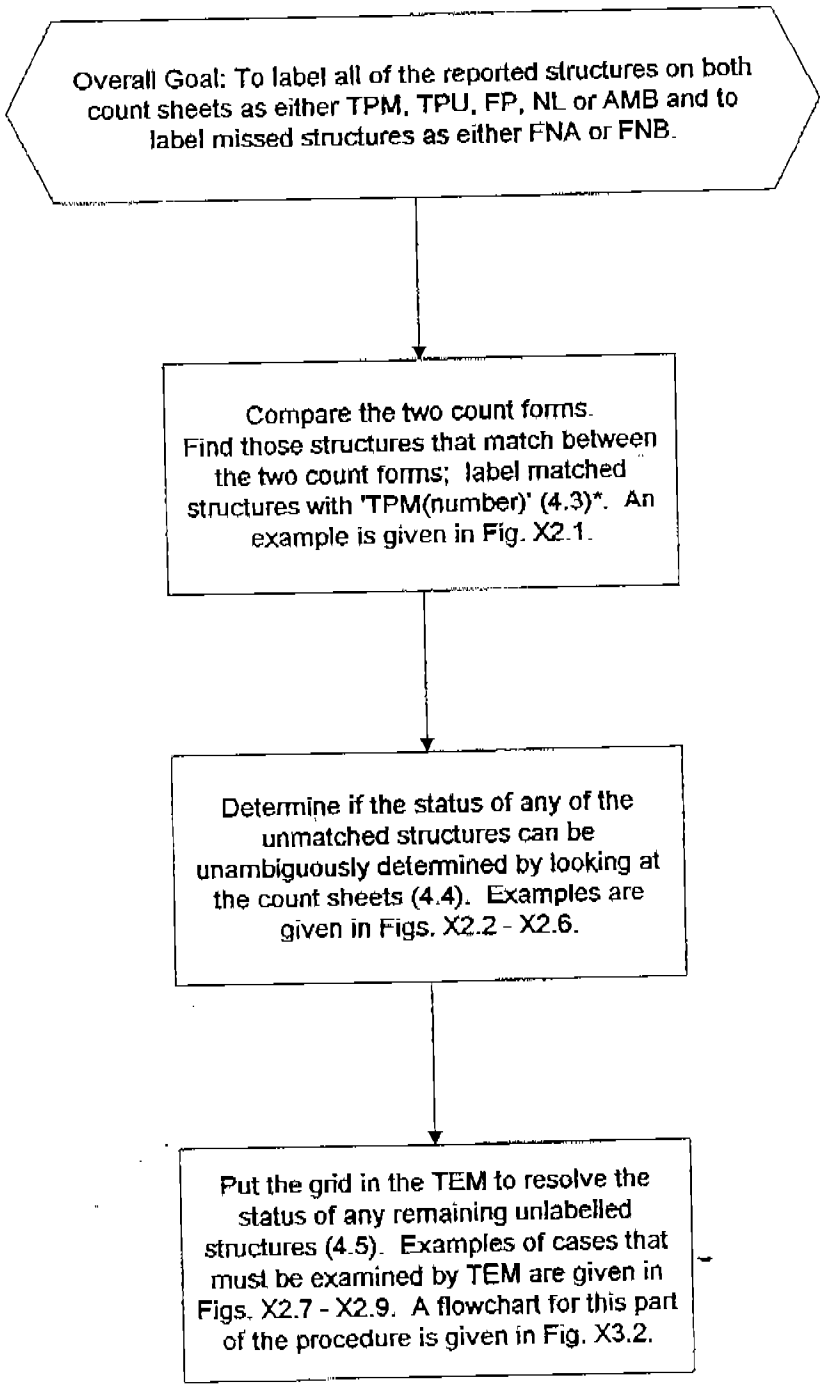


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.
*Numbers in parentheses in each block refer to the item number in the procedure.

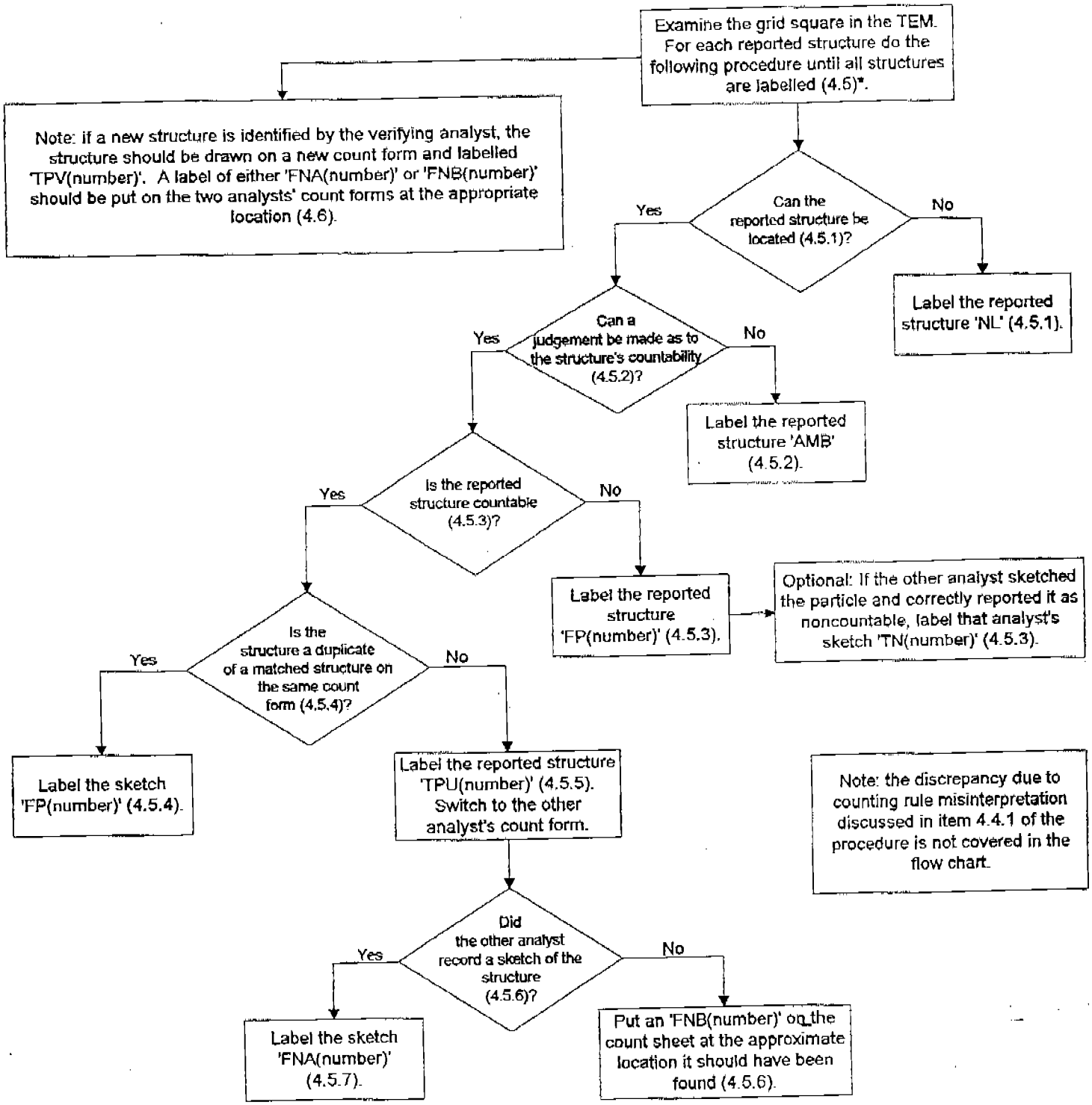


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

ATTACHMENT 4

Statistical Comparison of Two Poisson Rates

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

<u>Example:</u>

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 5

NVLAP Airborne Asbestos Proficiency Test 98-2:
Grid Orientation

Instructions for Form 1

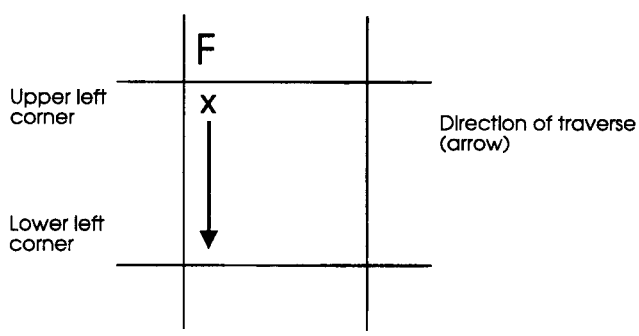
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1.* Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1.*

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1.* Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



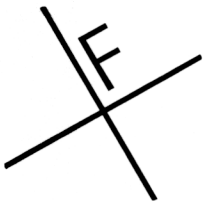
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

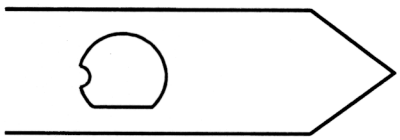
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



ATTACHMENT 6

Grid Opening Template for Sketching the Relative Position of Observed Structures

STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments:

(LB-000030) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology, and orientation relative to any nearby landmarks, if present.

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no loner need to be identified before analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: 8/14/03 (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: W J Brattin Date: 8/14/03
(Laboratory Manager or designate)

Project Review and Approval: [signature] Date: 8/14/03
(Volpe: Project Technical Lead or designate)

Approved By: Mary Goldade Date: 8/14/03
(USEPA: Project Chemist or designate)

Autio, Anni

From: Goldade.Mary@epamail.epa.gov
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: EPA Comments: LB-000030 (Draft for review/comment)

LB-000030 v0 (MG 08-07-03).doc... pic08313.gif (3 KB)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)

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To
Laboratory Activities
LB-000030

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Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: i

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): All Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted:

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

Autio, Anni

From: DeMalo, Robert [RDemalo@EMSL.com]
Sent: Thursday, August 07, 2003 11:20 AM
To: Goldade.Mary@epamail.epa.gov; Autio, Anni
Cc: Bob Shumate; LaCerra, Charles; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; EMSL Mobile Lab - Asbestos; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Richard Hatfield; Mahoney, Ron; Shu-Chun Su; Bill Longo
Subject: RE: EPA Comments: LB-000030 (Draft for review/comment)

I propose adding the word "morphology" as well into the description, as noted. I have no problem with including ISO to this procedure.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: EPA Comments: LB-000030 (Draft for review/comment)

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Autio, Anni

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Thursday, August 14, 2003 10:41 AM
To: 'Goldade.Mary@epamail.epa.gov'; Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Raney, Mark; Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: RE: EPA Comments: LB-000030 (Draft for review/comment)

LB-000030 v0 (MR 08-14-03).doc...

I concur with Mary's recommendations and mark-ups. The attached version also includes Rob Demalo's recommendation of adding morphology under the description section. Bill please finalize, sign and send it through the signature process. To expedite the process could you get Mary to sign before providing the original on for my signature. Let me know if you have any questions.

Thanks,

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
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Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

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Deleted: i

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): All Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted: _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

(LB-000031) Site-Specific SOP

Request for Modification
To
Laboratory Activities
LB-000031

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval. File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: _____

Requester: R.K. Mahoney Title: Senior Analyst / Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 27 January 2004

Description of Modification:
This is a clarification and expansion of TEM structure measurement and counting as expressed in the AHERA 40 CFR-Part 763 and ASTM D5755-95 methodologies.

Reason for Modification:
This clarification is intended to provide a basis for more consistent and uniform TEM results for the laboratories involved in the EPA Region 8, Libby, MT project.

Potential Implications of this Modification:
There are no negative potential implications of this clarification.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: Historic

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R.K. Mahoney Date: 27 January 2004
(Laboratory Manager or designate)

Project Review and Approval: [signature] Date: 1-27-04
(Volpe: Project Technical Lead or designate)

Approved By: [signature] Date: 2/5/04
(USEPA: Project Chemist or designate)

A Guide for Structure Measurement and Classification.
AHERA 40 CFR – Part 763 and ASTM D5755-95
US EPA Region 8, Libby, MT Project
Laboratory Modification LB-000031

Figure 1 Simple fiber – Record length and width. Structure must meet AHERA length and aspect ratio criteria.

Figure 2 Stepped fiber – Record length. Record width as a best estimate of the average width. Structure must meet AHERA length and aspect ratio criteria.

Figure 3 Bundle – Record length and width. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

Figure 4 Stepped bundle – Record length. Record width as a best estimate of the average width. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

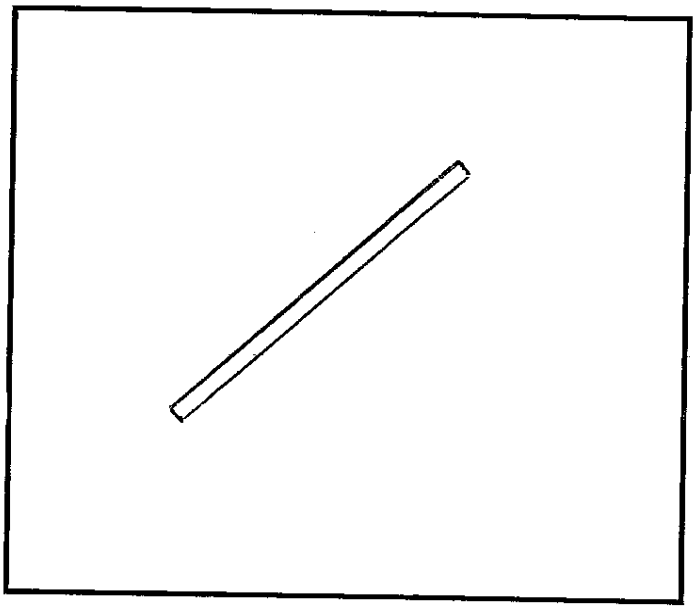
Figure 5 Matrix – Record longest exposed structure and its width. Structure must meet AHERA length and aspect ratio criteria.

Figure 6 Fiber with adhering matrix material - This structure does not fall into the matrix category as defined in that both ends are exposed (definition 14, AHERA) - Record length and width. Structure must meet AHERA length and aspect ratio criteria.

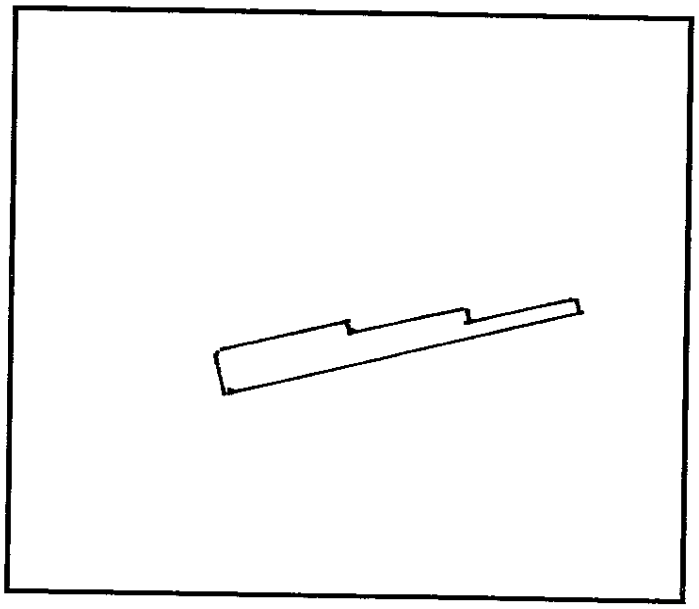
Figure 7 Structure with protrusions < 5:1 aspect ratio but an overall > 5:1 aspect ratio- Provided that the structure can be observed to be continuous through the adhering material, count as a fiber. Structure must meet AHERA length and aspect ratio criteria. If the structure cannot be observed to be continuous through the adhering material, do not count.

Figure 8 Cluster – Record the length of the longest observable structure. Record width as a best estimate of the average width of the overall structure. The aspect ratio of the overall structure is not a factor. There must be at least three intersections comprised of individual sub-structures that meet AHERA length and aspect ratio criteria to meet cluster definition.

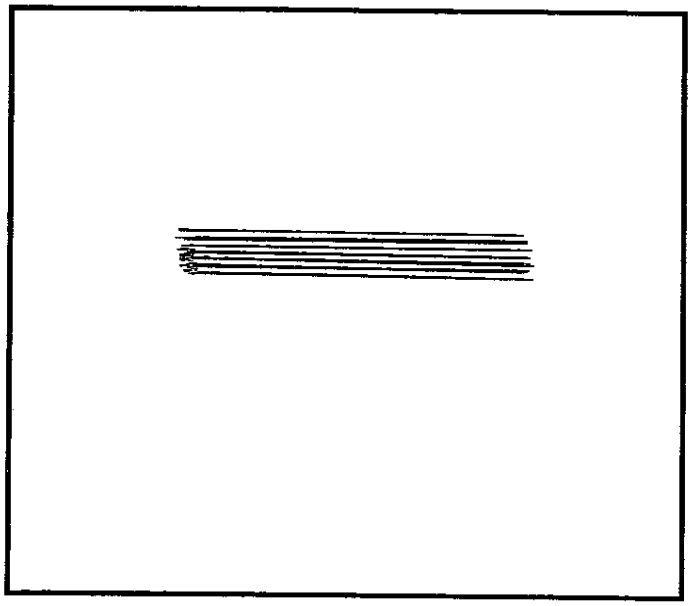
1



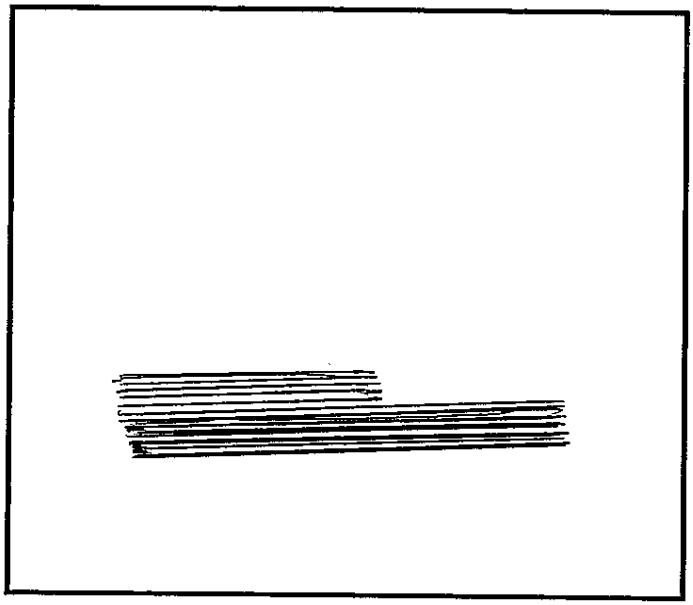
2



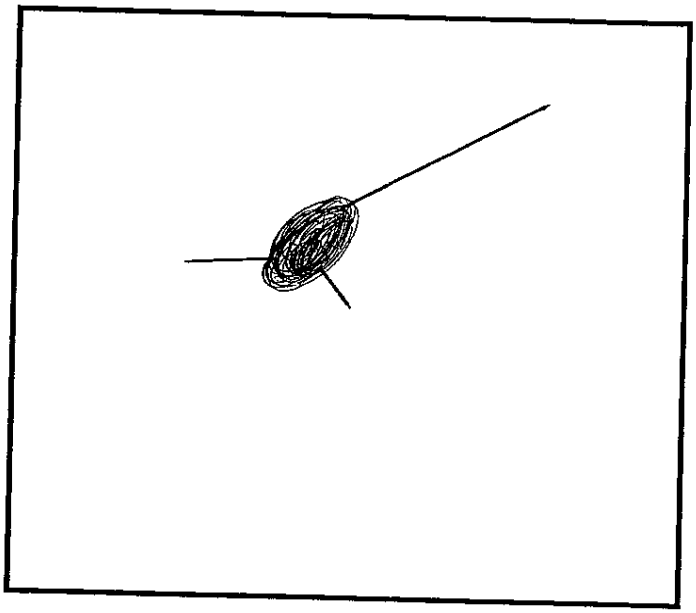
3



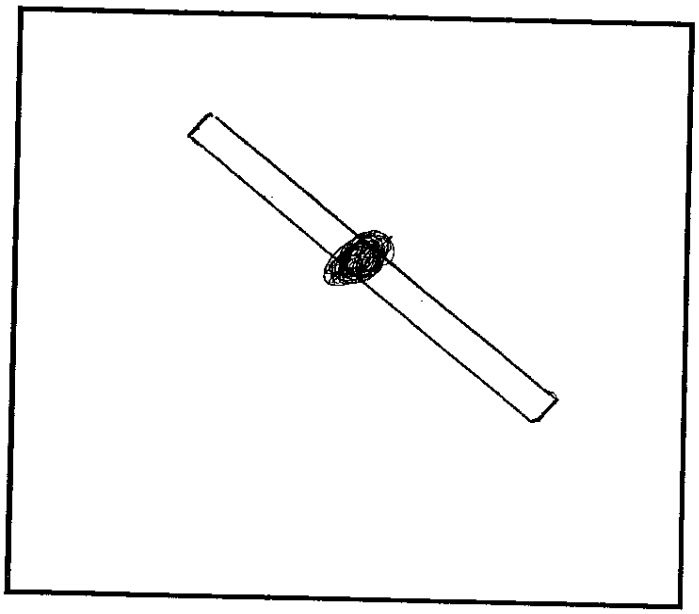
4



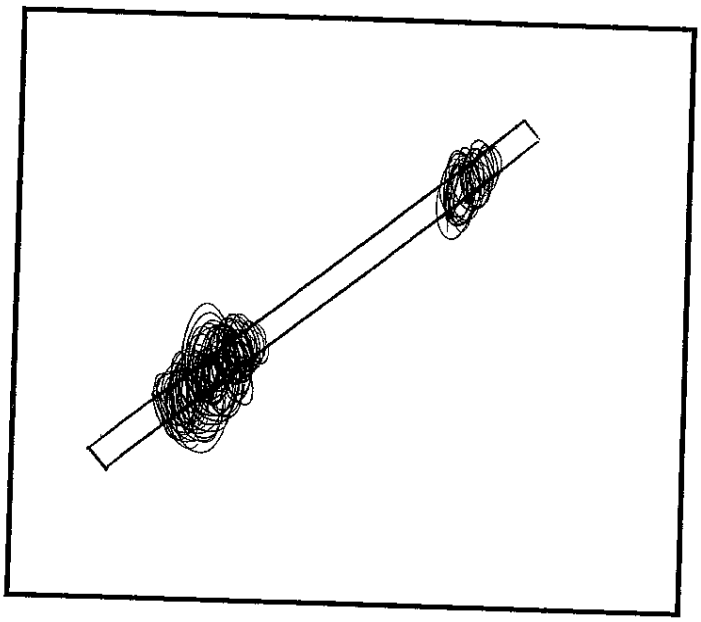
5



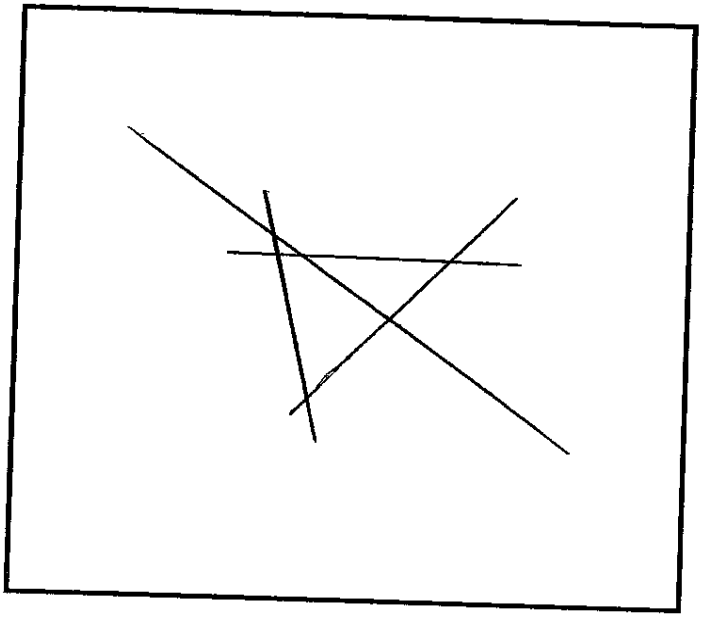
6



7



8



Kwiatkowski, Joseph

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Friday, October 10, 2003 12:59 PM
To: 'Goldade.Mary@epamail.epa.gov'; EMSL Mobile Lab - Asbestos
Cc: Autio, Anni; Kwiatkowski, Joseph
Subject: RE: LB-000031 discussion (With attachment this time)

I have reviewed the revised version of LB-000031 (as emailed by Ron M on 10/7/03) and it looks good. I therefore provide Volpe's approval, conditional to the following changes:

1. Permanent should be circled
2. The Effective Date should indicate "HISTORIC"
3. For the text associated with figures 2, 3, and 8 re-phrase the text such that "enough individual substructures" are defined. For example for the Figure 3 text say something like... "The aspect ratio of the overall structure is not a factor although the bundle must include at least three individual sub-structures that adhere to the AHERA length and aspect ratio criteria, in order to meet the AHERA definition for a bundle definition."

Let me know if you have any questions.

Thanks,

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Tuesday, October 07, 2003 3:17 PM
To: EMSL Mobile Lab - Asbestos
Cc: Anni Autio; Mark Raney
Subject: Re: LB-000031 discussion (With attachment this time)

Looks ok to me, Ron. Could you also attach the LB-000031 form to this email so we have it all together? I'll take a look at that and then send my approval.
Thanks,
mary

EMSL Mobile Lab - Asbestos <mobileasbestoslab@emsl.com>
10/02/03 03:41 PM

To: Mary Goldade/EPR/R8/USEPA/US@EPA, Mark Raney <Raney@volpe.dot.gov>, Anni Autio <autioah@cdm.com>
cc:
Subject: LB-000031 discussion (With attachment this time)

We'll try this again.

After a discussion with Bill on Tuesday, we deceided to modify the language
of the figure explanations of LB-000031 slightly. Let me know if this is OK
for you.

As soon as I get the go-ahead, I'll send it out.

R.

EMSL Mobile Asbestos Lab
107 W 4th St.
Libby, MT 59923
PH: (406) 293-9066
FAX: (406) 293-7016
http://www.emsl.com
(See attached file: LB-000031 discussion.doc)

Mary Goldade

02/05/04 10:47 AM

To: Anni Autio
cc: raney@volpe.dot.gov; mraney@adelphia.net (HOME)
Subject: LB-000024A & LB-000031 are in the snail mail

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 18th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

(LB-000045) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000045

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: Anni H. Autio Title: Laboratory Coordinator
Company: CDM Date: June 22, 2005

Description of Modification:
The TEM counting rules were defined in Attachment E of the Supplemental Remedial Investigation Quality Assurance Project Plan (USEPA June 2005) ; a copy is attached hereto.

Reason for Modification:
To document the counting rules, mineral classifications, and stopping rules applied to field samples collected for the Supplemental Remedial Investigation Quality Assurance Project Plan (USEPA June 2005)

Potential Implications of this Modification:
_____
_____

Laboratory Applicability (circle one): All Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: June 24, 2005
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
_____
_____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

LIBBY SUPERFUND SITE

ATTACHMENT E

TEM COUNTING RULES

All TEM analyses of air or dist samples for asbestos will be conducted in basic accord with the method and counting rules specified in ISO 10312 and all appropriated project-specific laboratory modification forms, in particular LB-000045. A brief summary of the counting rule changes outlined in LB-000045 are noted below:

1. All asbestos structures with a length greater than or equal to 0.5 um and an aspect ratio of 3:1 or greater shall be recorded.
2. The mineral type of each structure shall be classified as Libby Amphibole (LA), Other Amphibole (OA) or Chrysotile (C). When OA is selected, the mineral type is noted in the comments section.
3. Stopping rules for TEM analysis of LA structures in air and dust samples are specified in Attachment B. If a sample that is rich in chrysotile is encountered, recording of chrysotile structures may be stopped after completing the GO in which the 20th chrysotile structure is observed.

Source: Supplemental Remedial Investigation Quality Assurance Project Plan for Libby, Montana (USEPA . June 2005)

(LB-000053) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000053

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: Mark Raney Title: Volpe Libby Project Technical Lead
Company: US DOT Volpe Center Date: 25 April 2007

Description of Modification:
This laboratory modification relates to the project specific SOP for Indirect Preparation of Air and Dust Samples for TEM analysis (SOP # EPA-LIBBY-08). SOP # EPA-LIBBY-8 provides a standardized procedure for the indirect preparation of Libby air and dust samples that minimizes the loss of sensitivity and allows for the retention of a portion of the original filter for archive whenever possible. The SOP indicates two general indirect preparation procedures for samples, one that includes ashing of the primary filter and one that does not include ashing of the primary filter.

For air samples, whether ashing is required is dependant on whether the air sample is considered a project investigative or a non-investigative sample. Investigative air samples are defined as samples specifically collected to characterize concentration values in air for use in risk assessment and risk management decision making. In general non-investigative air samples are defined as samples that are intended only to help characterize the exposure level of EPA workers at the site and are used mainly to support health and safety assessment and monitor removal activities.

Table 1 of this laboratory modification provides a list of which sample prefix codes shall be identified as investigative and which shall be identified as non-investigative. In cases where there is a conflict regarding sample type between the sample prefix as defined by this modification and the chain of custody instructions, the chain of custody instructions take precedent. ALL investigative air samples shall require ashing as part of the sample indirect preparation procedures. Dust samples will NOT automatically require ashing regardless whether the sample is considered an investigative or non-investigative sample. For dust samples, ashing will be incorporated opportunistically as part of the indirect preparation procedures as determined necessary by the analyst.

Reason for Modification:
Air samples for which ashing may be warranted include indoor samples collected from properties with elevated levels of organic particulates (e.g., due to cigarette smoke or use of a wood-burning stove). In these samples, ashing may further reduce particulate loading, thus allowing for an improved analytical sensitivity. It was determined that for investigative air samples which require lower sensitivities the additional sample preparation time for incorporating ashing as part of the indirect preparation procedures was warranted. For Libby non-investigative samples, which typically require a faster analytical turnaround and higher target sensitivity, it was determined that the longer ashing procedures are not needed. It was also determined that ashing would be included only opportunistically for dust samples, since historically only a few dust samples have required ashing.

Potential Implications of this Modification:

Potential Implications of this Modification:
There are no known negative implications of this modification. Positive impacts to this modification are clarification of sample preparation steps.

Laboratory Applicability (circle one): All Individual(s)

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: December 12, 2006
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one): Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 12/08/06
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 12/7/06
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

TABLE 1 – Active Sample Prefixes
(October 5th, 2007 update)

Sample Prefix	Operable Unit	Prefix Description	Sampling and Analysis Plan Date	Investigative OR Non-Investigative?
1	4	Phase 1	Dec 99 (Rev 0) Jan 00 (Rev 1)	INVESTIGATIVE
2	4	Phase 2	Mar 01 (Rev 0)	INVESTIGATIVE
1R	4	Phase 1R	Sept 00 (Rev 1) Nov 2003	NON
CS	4	Contaminant Soil Screening Study	Apr 02 (Rev 0) May 03 (Rev 1)	INVESTIGATIVE
1D	4	Design Phase	Nov 03	INVESTIGATIVE
CE	4	Clean-up Evaluation	Nov 05	INVESTIGATIVE
SQ	4	SQAPP	Jun 05 (Rev 0) Aug 05 (Rev 1)	INVESTIGATIVE
CR	4	Cumulative Risk Pilot Study	Dec 2005	INVESTIGATIVE
AA	1,2,4,5,6	Outdoor Ambient Air	Dec 2006 July 2007 (addendum)	INVESTIGATIVE
DM	NA	Demolition Sampling	May 2007	NON
DP	4	Dust Sampling Pilot Study	May 2007	INVESTIGATIVE
TT	7	Troy Asbestos Project Evaluation	Jul 2007	INVESTIGATIVE
IN	4	Indoor Activity-Based Sampling	Jul 2007	INVESTIGATIVE
EX	4	Outdoor Activity-Based Sampling	Jul 2007	INVESTIGATIVE
P1	3	OU 3 Phase 1 Remedial Investigation	Oct 2007	INVESTIGATIVE
EP	1	Export Plant—Data Gaps and Collection	Sept 2007	INVESTIGATIVE
SL	5	Stimson Lumber Mill—Initial Soil Data Gap Investigation & Building Data Gap Sampling	Sept 2007 Nov 2007	INVESTIGATIVE
FC	4	Flower Creek Investigation	Aug 2007	INVESTIGATIVE

NOTE: If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead, the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.

Update Approvals:

Volpe Project Review and Approval: _____ Date: 1/17/08
(Volpe: Technical Lead or designate)

EPA Review and Approval: _____ Date: 2/4/08
(EPA: Project Chemist or designate)

TABLE 1 – Active Sample Prefixes
(May 11th, 2007 update)

Sample Prefix	Prefix Description	Investigative OR Non-Investigative?
1	Phase 1	INVESTIGATIVE
2	Phase 2	INVESTIGATIVE
1R	Phase 1R	NON
CS	Contaminant Screening Study	INVESTIGATIVE
1D	Design Phase	INVESTIGATIVE
CE	Clean-up Evaluation	INVESTIGATIVE
SQ	SQAPP	INVESTIGATIVE
CR	Cumulative Risk	INVESTIGATIVE
AA	Outdoor Ambient Air	INVESTIGATIVE
DM	Demo Sampling	NON
DP	Dust Pilot	INVESTIGATIVE
TT	Troy Asbestos Project Evaluation	INVESTIGATIVE
IN	Indoor Activity-Based Sampling	INVESTIGATIVE
EX	Outdoor Activity-Based Sampling	INVESTIGATIVE

NOTE: **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

Update EPA Approval: Mary Goldade 6/5/07

TABLE 1 – Active Sample Prefixes
(April 25th, 2007 update)

Sample Prefix	Prefix Description	Investigative OR Non-Investigative?
1	Phase 1	INVESTIGATIVE
2	Phase 2	INVESTIGATIVE
1R	Phase 1R	NON
CS	Contaminant Screening Study	INVESTIGATIVE
1D	Design Phase	INVESTIGATIVE
CE	Clean-up Evaluation	INVESTIGATIVE
SQ	SQAPP	INVESTIGATIVE
CR	Cumulative Risk	INVESTIGATIVE
AA	Outdoor Ambient Air	INVESTIGATIVE
DM	Demo Sampling	NON
TT	Troy Asbestos Project Evaluation	INVESTIGATIVE
IN	Indoor Activity-Based Sampling	INVESTIGATIVE
EX	Outdoor Activity-Based Sampling	INVESTIGATIVE

NOTE: **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

TABLE 1 – Active Sample Prefixes
(December 6th, 2006)

Sample Prefix	Prefix Description	Investigative OR Non-Investigative?
1	Phase 1	INVESTIGATIVE
2	Phase 2	INVESTIGATIVE
1R	Phase 1R	NON
CS	Contaminant Screening Study	INVESTIGATIVE
1D	Design Phase	INVESTIGATIVE
CE	Clean-up Evaluation	INVESTIGATIVE
SQ	SQAPP	INVESTIGATIVE
CR	Cumulative Risk	INVESTIGATIVE
AA	Ambient Air	INVESTIGATIVE
DM	Demo Sampling	NON

NOTE: **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

Raney, Mark

From: Raney, Mark
Sent: Friday, December 08, 2006 4:44 PM
To: Anni Autio (E-mail)
Cc: Lynn Woodbury (E-mail); 'Goldade.Mary@epamail.epa.gov'
Subject: RE: LAB MODs #53 EPA Approved

LB-000053 (MR 2-08-06 email)..

Anni,

Attached is a copy of the final approved version of Lab MOD #LB-000053. The attached modification will be considered effective immediately following discussion during Tuesday's lab call. Please distribute the attached to the laboratories for their review prior to Tuesday's call.

Note: I will be mailing the signed hardcopy to Mary for her signature. Mary will then forward the final/original signed hardcopy to CDM.

Let me know if you have any questions.

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, December 07, 2006 5:14 PM
To: Raney, Mark
Cc: Anni Autio (E-mail); Lynn Woodbury (E-mail)
Subject: Re: LAB MODs #53 EPA Approved

EPA approves Lab Mod 53 with changes as attached.
(See attached file: LB-000053 (MR 12-06-06 email) (MG 12-7-06).doc)

"Raney, Mark" <Mark.Raney@Volpe.dot.gov>

12/06/2006 03:31 PM

To
Mary Goldade/EPR/R8/USEPA/US@EPA, "Mary Goldade (E-mail 2)" <mgoldade@peakpeak.com>

cc
"Anni Autio (E-mail)" <autioah@cdm.com>, "Lynn Woodbury (E-mail)" <woodbury@syrres.com>

Subject
LAB MODs #53 and 29b

Mary,

Attached is a draft for your review of Lab Mod LB-000053 (to accompany the indirect SOP).

I have also reviewed Lab MOD LB-000029b. I have no comments to LB-000029b other than a formatting comment. The numbering format needs to be corrected for the steps on Attachment 2, under "At the Interlab Laboratory".

Let me know if you have any questions.

Mark.

Mark E. Raney, RTV-4E
Environmental Engineer / Project Manager
US DOT / RITA / Volpe Center
55 Broadway
Cambridge, MA 02142
raney@volpe.dot.gov
Office: 617-494-2377
Mobile: 617-694-8223
Fax: 617-494-2789

<<LB-000053 (MR 12-06-06 email).doc>> <<LB-000029b v7.doc>> [attachment "LB-000029b v7.doc" deleted by Mary Goldade/EPR/R8/USEPA/US]

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

Shirley Turner
Eric B. Steel

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards
and Technology
Microanalysis Research Group
Surface and Microanalysis Science Division
Chemical Science & Technology Laboratory
Gaithersburg, MD 20899

March 1994

U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. 1987, *52* (No. 210), 41826-41905.

[2]ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2– The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3– The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4– The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

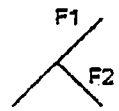
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

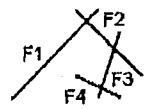
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

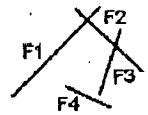
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

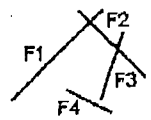
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

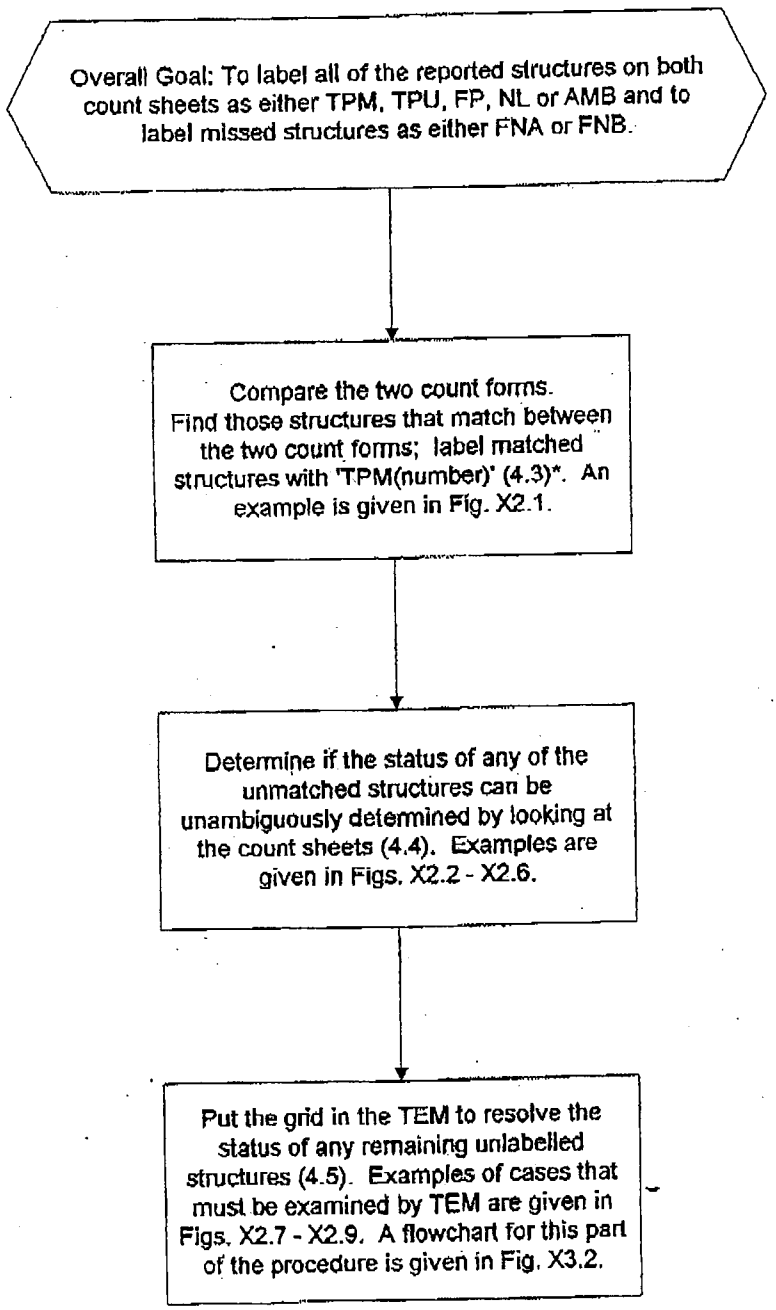


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst. *Numbers in parentheses in each block refer to the item number in the procedure.

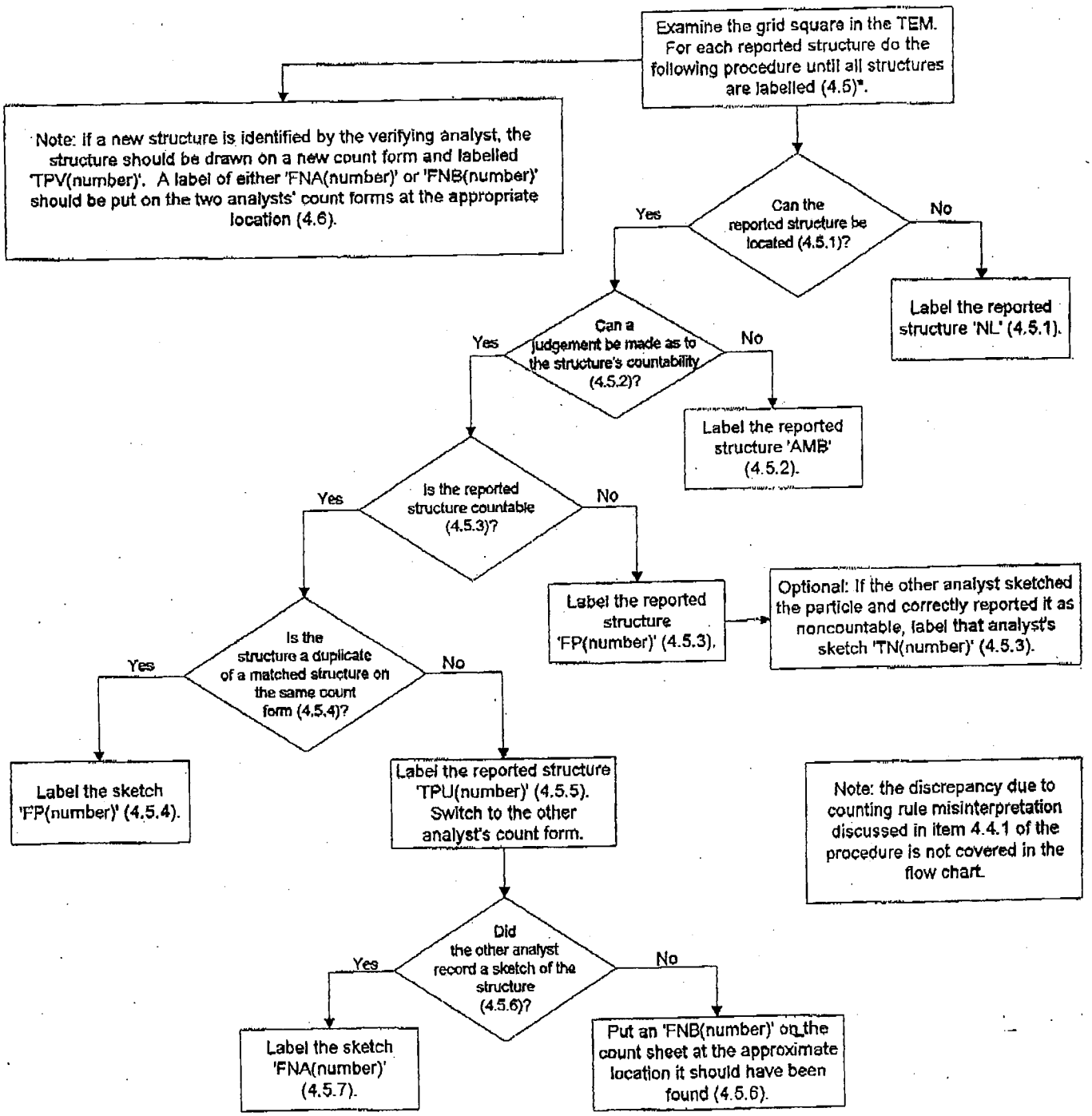


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

ATTACHMENT 4

Statistical Comparison of Two Poisson Rates

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

<u>Example:</u>

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 5

NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation

Instructions for Form 1

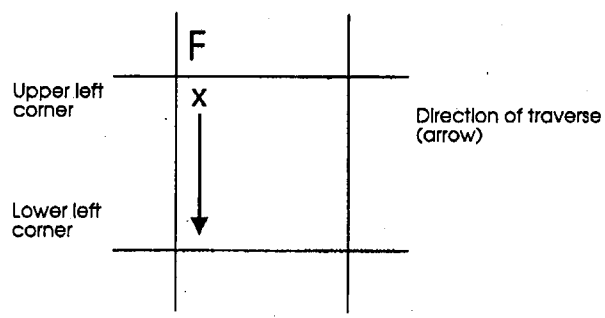
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1*. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1*.

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



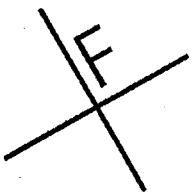
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

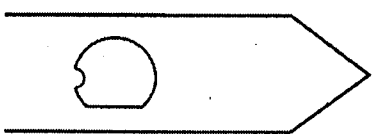
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



ATTACHMENT 6

Grid Opening Template for Sketching the Relative Position of Observed Structures

STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments:

(LB-000066c) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000066c

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: W. Brattin Title: Technical Consultant
Company: Syracuse Research Corporation Date: 09/11/2007

Description of Modification:
This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles,

Reason for Modification:
Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length ≥ 0.5 um, aspect ratio ≥ 3:1), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

Potential Implications of this Modification:
This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):

Temporary Date(s): 09/12/2007 until notified

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: _____

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Attachment 1

Note: This modification (LB-000066c) **supersedes** LB-000066b.

Technical Review: _____ Date: _____

(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 9/12/07

(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 9/11/07

(USEPA: Project Chemist or designate)

REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969.

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

1. Continue to classify structures as LA, OA, or C in accord with current procedures.

2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.

3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

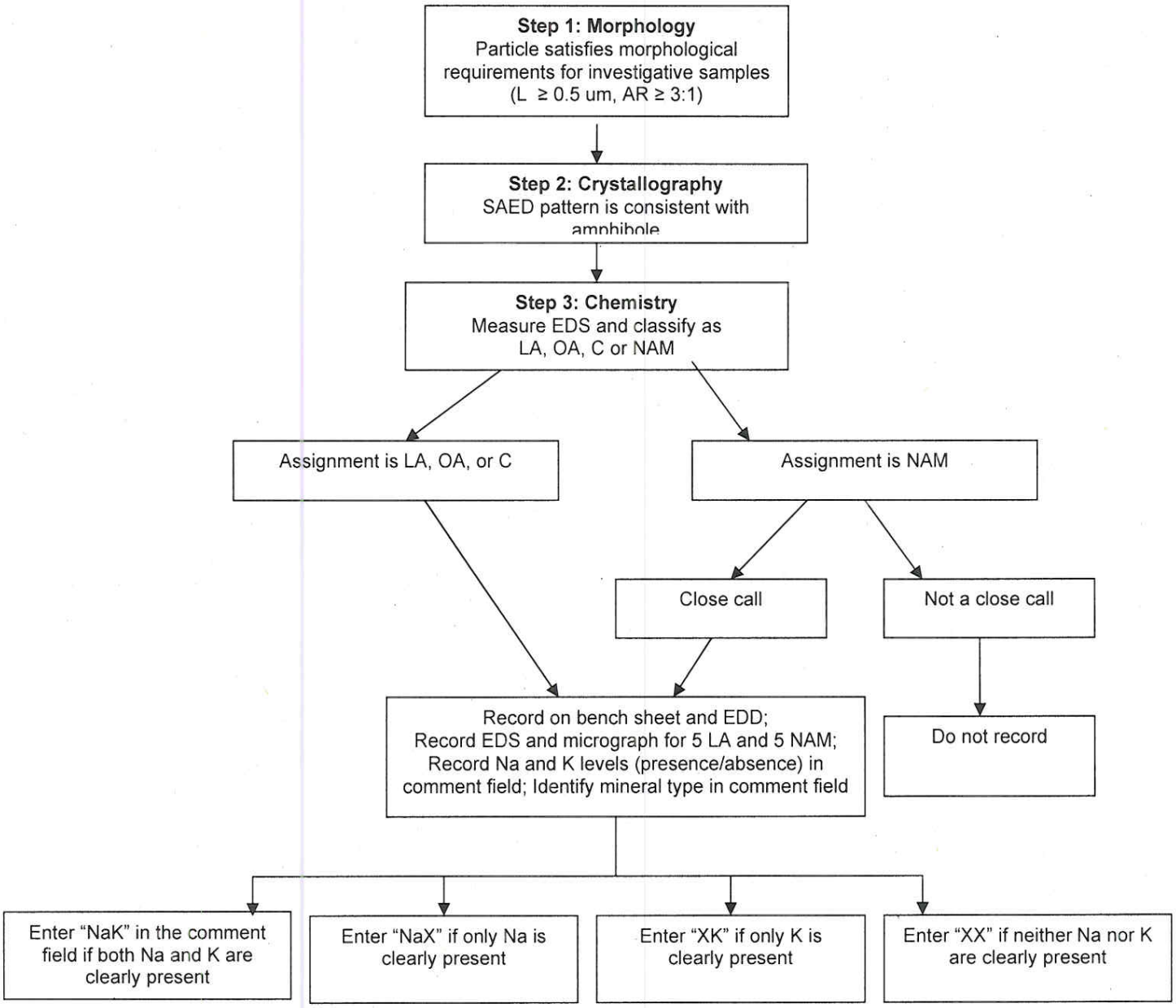
AC – actinolite
TR – tremolite
AT – actinolite/tremolite (too close to call)
AM – amosite
AN – anthophyllite
CR – crocidolite
PY – pyroxene
UN – Unknown

5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.

6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.

7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

FIGURE 1
FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION



(LB-000084) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000084

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: EPA-Libby-03, EPA-Libby-07, EPA/600/R-94/134 (100.2, Mod 20)

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator
Company: EMSL Analytical, Inc. Date: 29 January 2008

Description of Modification:
The purpose of this modification is to modify the counting rules for all TEM analysis methodologies as they pertain to the presence of abundant chrysotile. Note, this modification replaces **(supersedes?)** modifications LB-000016a and LB-000017a and pertains to all TEM methodology clarifications associated with abundant chrysotile. Enumeration of chrysotile structures may be terminated after a minimum of 50 structures are counted and recorded. If abundant chrysotile is present, when feasible the chrysotile count may be terminated at the end of the grid opening in which the 50th chrysotile structure is counted. In situations where the 50th chrysotile is encountered prior to completion of a grid opening and excessive chrysotile remains unenumerated within the GO, enumeration of chrysotile may be terminated with the completion of the vertical traverse in which the 50th chrysotile is encountered. A decimal estimation of the percentage of the grid opening enumerated will be calculated by measuring the linear portion of the horizontal grid bar enumerated as compared to that not enumerated. This area estimate will be recorded on Data Entry 2 (of the EDD) in the column entitled "Fraction of GO counted". The grid opening where the enumeration of chrysotile was terminated prior to the completion of the grid opening will be followed by an "!" (e.g., J6!). A qualifier will be added to the chrysotile concentration to indicate it is an estimated calculated value based on the recorded estimated GO fraction counted. The analysis will continue recording amphibole structures only until the remaining grid openings to be analyzed are completed as required to satisfy the desired analytical sensitivity. The grid opening designations will be followed by an "*" to indicate the grid openings where only amphibole structures were recorded (e.g., J8*).

Reason for Modification:
In that chrysotile is not the analyte of interest in the project, the decision by EPA was that it was an inappropriate expenditure of time and effort to enumerate more that approximately fifty chrysotile structures.

Potential Implications of this Modification:
Truncated analysis of excessive numbers of chrysotile has the potential to generate greater uncertainty regarding the calculated chrysotile concentrations.

Laboratory Applicability (circle one): All Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 29 January 2008

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

_____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

(LB-000085 rev) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000085

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: All TEM and SEM Methods supporting Libby site investigative or Libby Action Plan (LAP) sample analysis

Requester: Mary Goldade Title: Senior Environmental Scientist/Chemist
Company: Environmental Protection Agency, Region 8 Date: April 2, 2008

Description of Modification:
Laboratories conducting transmission electron microscopy (TEM) or scanning electron microscopy (SEM) analysis in support of either the Libby Site (all operable units, including Troy) or Libby Action Plan shall perform analysis of a reference standard to calibrate the energy dispersive x-ray spectrometry (EDS) analysis. The reference standard, a glass material referred as BIR-1G, was created by the USGS. It is recommended for use for Libby Amphibole analysis because it contains sodium (Na) and potassium (K) at known levels. Na and K are important elements used in Libby Amphibole identification by EDS. The BIR-1G standard was freezer-milled by EMSL to create particles for EM analysis. While generation of thin sections of the BIR-1G using a microtome was not feasible due to the expense, analysis of the BIR-1G in particulate form is useful in standardizing the elemental measurements of the EDS and understanding the inherent variability in the EDS measurements.

The BIR-1G shall be tested at the beginning of each analytical run and must meet acceptance criteria prior to analysis of any field samples. Laboratories shall record the calibration information in accord with Attachment 1. As seen, not only does Attachment 1 provides the details for populating the electronic disk deliverable (EDD) used in recording the calibration information, but Attachment 1 also describes the process for generating acceptance criteria for the BIR-1G standard for each individual instrument.

Reason for Modification:
The modification provides for a standardized process for performing and recording calibration standards for EDS during Libby Amphibole analysis.

Potential Implications of this Modification: There are no negative implications to this modification. Positive impacts include a standardized process for: (1) daily calibration of a standard for the EDS used in Libby Amphibole identification; (2) reporting results of BIR-1G measurements; and (3) generating acceptance criteria for the BIR-1G standard over time.

Laboratory Applicability (circle one): All Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s):_____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: April 23, 2008
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

_____

Technical Review: _____N/A_____Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____Date:_____
(Volpe: Project Technical Lead or designate)

Approved By:_____Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

Analyzing the BIR-1G Standard

- The BIR-1G standard shall be tested at the beginning of each analytical run or daily (whichever is more frequent), prior to analyzing any field samples.
- Set up TEM instrument and orient for typical Libby field samples.
- Record the TEM instrument details in the BIR-1G Electronic Data Deliverable (EDD) spreadsheet (see most recent version of Excel file "BIR-1G EDD.xls"). Note: Use one spreadsheet per TEM instrument.
- For each BIR-1G evaluation, select one particle and record the measured weight % for each element ***as oxide weight %*** in the BIR-1G EDD. Note: When recording oxide weight %, enter results as a percentage not fractions (i.e., for 30%, enter 30 not 0.3).
- When selecting particles for analysis:
    - Choose particles in the middle of the grid opening and in the center of the grid.
    - Particles should not be in close proximity to the grid bar or neighboring particles.
    - Randomly select particles within different grid openings for each analysis.
- For selected particles, focus the beam on the thin edge, not the center of the particle.
- Continue analysis until a count of 1,000 is achieved for silicon (Si). This total Si count should be sufficient to achieve optimum instrument testing conditions. It is recognized that this total Si count may not be equivalent to typical analytical conditions for field samples.
- On a monthly basis, the EDD for each TEM instrument should be provided to EPA (or designated contractors).

Acceptance Criteria

- Acceptance criteria will be TEM instrument- and element-specific and will be derived from measured results.
    - Results that are within ± 1 standard deviation of the nominal will be ranked as acceptable.
    - Results that are outside ± 1 standard deviation but within ± 2 standard deviations of the nominal will be ranked as within the warning level.
    - Results that are outside ± 2 standard deviations of the nominal will be ranked as a failure.
- The potential bias of measured results will be assessed based on a frequency evaluation of results above and below the nominal.
- As needed, EPA will re-evaluate and revise the acceptance criteria to optimize program goals.

Corrective Action

In the event that analysis results of the BIR-1G fall outside of the acceptance criteria, there should be a structured, progressive response. First, confirm that the detector/x-ray system has satisfied the acceptance criteria in the past. Next, confirm that the settings for the x-ray analysis software are correct (e.g. bias, scale). Finally, de-ice the LN2 dewar (unless it is a dry system) and carefully clean the window.

If these actions fail to rectify the problem, it will probably be necessary to send the detector/x-ray out to be serviced. The actions taken by the servicing company may include such things as baking the detector, renewing the vacuum in the dewar, checking the pre-amp or actual x-ray system for hardware defects, or replacing the crystal and/or FET (field effect transistor). In most instances the fault will not lie in the window unless the integrity of the window is compromised.

Upon the return and re-installation of the detector, re-run the BIR-1G standard to confirm that corrective action measures have resolved analysis issues.

(LB-000086) Site-Specific SOP

Request for Modification
to
Laboratory Activities
LB-000086

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator
Company: EMSL Analytical Inc. Date: 22 April 2008

Description of Modification:
All samples analyzed by SRC-Libby-03 (PLM-VE) shall be referenced by the use of a concatenation of the Index ID, Suffix ID, and the Suffix # (e.g. 1D-00827-FG2).

Reason for Modification:
This will allow for the identification of different sample aliquots resulting from the cone and quarter process conducted at the CSF facility in Denver to generate the VE samples that will be very useful in the QC reanalysis process.

Potential Implications of this Modification:
There are no negative implications resulting from this process.

Laboratory Applicability (circle one): All Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 22 April 2008
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

_____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

(ISSI-LIBBY-01) Site-Specific SOP

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Date: May 6, 2004 SOP No. ISSI-LIBBY-01 (Rev. 8)

Title: SOIL SAMPLE PREPARATION

Author: William Brattin Syracuse Research Corporation(a)

SYNOPSIS: A standardized method for preparation of soil samples for asbestos analysis is described.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		_____
Syracuse Research Corp.	_____	

Revision Number	Revision Date	Reason for Revision
1	1/7/00	Incorporation of sieving to the sample preparation.
2	7/12/00	Revision in sieve size, other minor edits.
3	5/7/02	Incorporate minor edits

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Revision Number	Revision Date	Reason for Revision
4	8/1/02	Modify sieving procedure, add grinding step
5	3/6/03	Incorporate modifications to the procedure and documentation requirements
6	3/24/03	Incorporate modifications to the logsheets to conform with electronic data storage requirements and add grinder blank requirements.
7	8/5/03	Incorporate modifications to drying and sample storage procedures
8	5/4/04	Incorporate modifications to drying batch size and recording of preparation information

(a) This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by Syracuse Research Corporation (SRC).

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for preparation of soil samples for asbestos analysis. This procedure will be used by employees of United States Environmental Protection Agency (USEPA) Region 8 and by contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby, Montana site. Site-specific deviations from the procedures outlined in this document must be reviewed and approved within a Request for Modification by the Volpe Center Technical Lead or Libby Project Manager and the USEPA Region 8 Remedial Project Manager or Regional Chemist.

The contents of this SOP have been specifically designed for the Libby Asbestos site. For example, the particle size of 250 µm was selected in an attempt to balance two opposing goals: 1) grinding the sample to a small enough particle size to obtain homogeneous soil samples; and 2) keep the particle size distribution of sufficient size to accommodate analyses by several methods including polarized light microscopy-visual area estimation (PLM-VE), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It is possible that for methods such as the TEM, further preparation at the laboratory may be necessary. If so, these additional steps will be addressed at the level of the TEM SOP.

Procedures outlined in this SOP have been designed with the intent to prepare soil samples having a target concentration greater or equal to approximately 0.1-0.2% (weight percent) total Libby amphibole (LA) material.

2.0 RESPONSIBILITIES

The Preparation Laboratory Project Leader (PL2) may be an USEPA employee or contractor who is responsible for overseeing the soil sample preparation activities. The PL2 is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Close Support Facility Soil Preparation Plan, Libby Asbestos Site, Operable Unit 4, Libby, Montana (CSF SPP). It is the responsibility of the PL2 to communicate with the Preparation Laboratory personnel regarding specific collection objectives and anticipated situations that require any deviation from the respective Project Plans. It is also the responsibility of the PL2 to communicate and document the need for any deviations from the

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Project Plans with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

Personnel preparing Libby soil samples are responsible for adhering to the applicable tasks outlined in this procedure and conducting all sample handling and preparation activities in the ventilation hood.

3. 0 EQUIPMENT

General purpose laboratory oven - must be capable of maintaining a constant temperature of approximately 89-91°C.

Analytical balance - calibrated and accurate to tolerance limits indicated on Attachment 2, range of 0.1 g to at least 2000 g

Riffle splitter - with 3/4 inch chutes to split samples

Plate Grinder - capable of accepting soil particles of approximately 1/4 inch diameter and grinding to produce particle of approximately 250 µm

Metal (other than plastic) scoop or spoon - for transferring samples

1/4 inch metal (other than plastic) sieve and catch pan - for coarse sieving samples

60 mesh (250 µm) and 200 mesh (74 µm) metal (other than plastic) sieves - for verification of the plate grinder settings

Clean quartz sand - required for preparation of grinding and drying blank samples (Sections 6.2, 9.2, 12.1 and 12.3) and for decontamination of grinder (Section 9.4)

Clean soil - sufficient aliquot required for calibration of grinder (Section 9.1)

Drying Pans - pans used during the sample drying process

Sample containers - plastic ziplock bags (pint and gallon size)

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless

Field clothing and Personal Protective Equipment - as specified in the Health and Safety Plan (Appendix E of the CSF SPP, December 2003)

Field notebook -used to record progress, any problems or observations and deviations

Sample Drying Log Sheets - (Attachment 1) - used to record all sample drying information

Sample Preparation Log Sheets (Attachment 1)- used to record all sample preparation information (splitting, sieving and grinding)

Three-ring binder books - binders will contain:

Analytical Balance Calibration and Maintenance Log (Attachment 2)
Grinder Calibration and Maintenance Log (Attachment 3)
Ventilation Hood Calibration and Maintenance Log (Attachment 4)
Vacuum Maintenance Log (Attachment 5)
Oven Temperature Calibration and Maintenance Log (Attachment 6)
Sample labels

Trash Bags - used to dispose of gloves, wipes and other investigation derived waste

Indelible Marking Pen - used to record sample information onto plastic ziplock bags and to record logbook information
Ballpoint Pen - used to record field logsheet information

4.0 METHOD SUMMARY

Figure 1 provides an overview of the steps in this procedure. Soil samples are dried in a standard laboratory oven and split into a preparation sample and an archive sample. The

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preparation sample is sieved to separate coarse material (> 1/4 inch) from fine material (< 1/4 inch). The fine material is ground to a standard particle size of about 250 µm for subsequent asbestos analysis. The coarse material is examined by stereomicroscopy to determine if any large particles of asbestos are present (EPA SOP SRC-LIBBY-01).

5.0 SOIL STORAGE

Upon receipt of samples, samples will be grouped in an inventory batch of approximately 120 samples. Samples will be archived according to the inventory batch they are assigned to and filed by the inventory ID noted on the Sample Drying Log Sheet and Sample Preparation Log Sheet (Attachment 1). This box number will be automatically assigned by the electronic Libby Asbestos Sample Tracking Information System (eLASTIC) when the inventory batch is created in the database.

6.0 BULK SOIL DRYING

Prior to drying, samples will be grouped in a drying batch and assigned a drying batch number. The following sections detail all activities and procedures related to drying samples.

6.1 Calibration

Samples will be weighed prior to and following drying activities. The analytical balance used for drying activities will be calibrated on days when samples are loaded into, or unloaded from, the oven. Before weighing samples, calibrate the balance using S-1 class weights and record all measurements, any required maintenance, and the balance number on the Analytical Balance Calibration and Maintenance Log (Attachment 2).

All drying activities will be performed under a negative pressure HEPA filtered hood. Prior to loading the oven, the ventilation hood will be calibrated to ensure that the ventilation system is operating properly. Ventilation hood calibration and any required maintenance will be documented on the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. Vacuum calibration will be performed daily, prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded on the Vacuum Maintenance Log (Attachment 5).

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Oven temperature calibration will be performed on a weekly basis. Oven temperature calibration and any required maintenance will be documented on the Oven Temperature Calibration and Maintenance Log (Attachment 6).

6.2 Drying Blanks

A drying blank will be created and associated with each drying batch prior to loading samples into the oven. A drying batch will consist of approximately 15 samples. The drying blank will consist of approximately 100-200 grams of clean quartz sand, placed in a drying pan and assigned an index ID (see Section 6.1). Each drying blank will be identified in the notes section of the Sample Drying Log Sheet (Attachment 1) and will be prepared using the same methodology as other soil samples. Following preparation, whenever possible, each blank will be shipped with its associated batch samples. See Section 12.1 for more details regarding drying blanks.

6.3 Drying Procedure

Samples will be loaded into the drying oven using the following steps:

Record the SOP and Revision Number used to prepare the samples on the Sample Drying Log Sheet (Attachment 1). Record the oven number used to dry the samples on the Sample Drying Log Sheet (Attachment 1).

Prior to unsealing and drying each sample, record the sample mass to the nearest 0.1 g on the Sample Drying Log Sheet (Attachment 1), the technicians initials and the date. See Section 6.1 for calibration details.

Set the oven temperature to 90 ± 1°C. For every sample drying batch, check the oven temperature to verify that proper temperature has been reached and document the start date/time and temperature on the Sample Drying Log Sheet (Attachment 1).

Transfer each sample to be dried from its zip top storage bag into a clean drying pan. Each sample should be transferred to its respective drying pan under the negative pressure HEPA filtered hood. Label each drying pan with its respective Index ID. Place each sample in the oven.

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Leave the samples in the oven for approximately 24-48 hours or until completely dry. Verify that each sample is dry, by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness. Once it is confirmed that samples are dry, record the technician's initials, and the date and time of completion, on the Sample Drying Log Sheet (Attachment 1).

Turn off the oven and allow the samples to cool in the oven. Once the samples are cooled, unload each sample and transfer each sample volume to a clean zip top bag, re-bag the sample with another clean zip top bag and identify the dried sample with the index ID. All samples should be transferred to zip top bags under the negative pressure HEPA filtered hood to prevent potential exposure to fibers that might be released from the sample.

Record the sample mass of each bagged sample to the nearest 0.1 g on the Sample Drying Log Sheet (Attachment 1), the technician's initials and the date.

6.4 Decontamination

Decontaminate the inside of the hood and the inside of the oven by HEPA vacuuming and wet wiping all surfaces before loading a new batch for drying.

Decontaminate all sample drying pans under the ventilation hood using compressed air or a HEPA vacuum to remove any residual organic material left on the pans. Wet wipe or brush off any visible material that is not removed from the air blast or vacuum. All pans will be decontaminated between samples.

7.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

Prior to sieving and grinding, samples will be divided into a portion for archive and a portion for preparation. The sections below describe the sample splitting procedure.

7.1 Calibration

Prior to any splitting, sieving, or grinding activities, calibrate the ventilation hood to ensure that

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the ventilation system is operating properly. Document ventilation hood calibration and any required maintenance on the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

7.2 Procedure for Sample Division

Samples will be divided using the following steps:

Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.

Splitting must be performed in the hood to prevent potential exposure to fibers that might be released from the sample. Place one pan on each side of the riffle splitter. Divide the sample into two equal sub-parts by removing the sample from its original plastic bag and loading the dry material into the splitter.

After splitting, set aside one part for sample preparation as described below (if the volume of the portion left for preparation is still too large for processing, split the sample again so that 3/4 of the original sample will be archived and 1/4 will be set aside for processing).

Place the remaining split portion into a clean, zip top bag, re-bag the sample in another clean zip top bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the index ID and the suffix “A” (for archive fraction). Record the technician’s initials and date on the Sample Preparation Log Sheet. Store the archive portion in the numbered inventory box noted on the Sample Preparation Log Sheet (Attachment 1).

7.3 Duplicate Samples

One preparation duplicate sample will be processed for every 20 field samples prepared. A preparation duplicate is a sample split of material that is prepared in the same fashion as the parent sample (preparation split) and will be submitted to the laboratory blind. The preparation duplicate will be assigned a unique and random index identification number. For both samples, the corresponding index ID will be indicated in the notes section of the Sample Preparation Log Sheet (Attachment 1). If a preparation duplicate is not being prepared for a particular sample, proceed to Section 7.4.

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Following the division of a sample for preparation and archive. Divide the designated sample into two equal sub-parts using a riffle splitter (as described in section 7.2). Retain one portion as the parent sample and assign the other portion the duplicate index ID. Record the technician's initials, and the date of creation on the Sample Preparation Log Sheet (Attachment 1), when the duplicate sample is prepared. Prepare each portion according to the processes outlined below. For further information on preparation duplicates, refer to Section 12.2.

7.4 Decontamination

The splitter will not be decontaminated following this step provided the fine ground sample will be split again into four fractions in Section 10.0. If for any reason the same sample is not immediately split further, the riffle splitter must be decontaminated as follows.
Use a HEPA vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

8.0 PREPARATION SAMPLE SIEVING

All samples will be sieved prior to grinding to separate out the coarse and fine fractions. The sample sieving procedure is described in the sections below.

8.1 Calibration

All sieving activities will take place in the hood. Refer to Section 6.1 for details regarding the frequency of ventilation hood calibration.

Samples will be weighed during sieving activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded on the Analytical Balance Calibration and Maintenance Log (Attachment 2).

8.2 Sample Sieving Procedure

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Conceptually, sample sieving may generate a coarse and a fine fraction sample. Samples will be sieved using the procedure outlined below.

Coarse Fraction

A 1/4 inch stainless steel screen with catch pan will be used to divide the fine and coarse fractions using the following procedure:

Pour the sample through the 1/4 inch stainless steel sieve and give the screen a shake to ensure all particles < 1/4 inch in size are allowed to pass through the screen. In addition, a pestle may be used to break up any remaining soil clumps to ensure all particles <1/4 in size pass through the screen.

Pour all material which does not pass through the screen (> 1/4 inch) into a new, tared, sample bag and identify the coarse sample with the index ID and the suffix "C" (for "coarse fraction").

Record the mass of the coarse fraction to the nearest 0.1 g on the Sample Preparation Log Sheet (Attachment 1) and record the technician's initials and the date.

Double-bag the coarse sample portion and identify the sample with the index ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

Fine Fraction

Tare an empty sieve pan, to account for the weight of the pan containing the fine sample, and weigh the fine material that passed through the sieve. Record the mass of the fine fraction to the nearest 0.1 g on the Sample Preparation Log Sheet (Attachment 1). If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction on the Sample Preparation Log Sheet.

Whenever possible, immediately process the fine material that passes through the screen in accord with the approach described in Section 9.3 (below). If processing cannot occur immediately, pour the fine material which passed through the sieve into a new plastic ziplock bag and identify the fine sample material with the index ID and the suffix "F" (for "fine

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fraction"). Double-bag the sample and identify the sample with the index ID and suffix on the outside of the bag.

8.3 Decontamination

Decontaminate all sieves, pans and the pestle under the ventilation hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual organic material left on the sieve pans. All pans and sieves will be decontaminated between samples.

9.0 FINE SAMPLE GRINDING

The fine sieved sample will be ground to produce a material of about 250 µm. The final sample will be packaged and shipped to the laboratory for asbestos analysis. The procedure for grinding the fine sieved sample is outlined below.

9.1 Calibration

All grinding activities will take place in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.
A HEPA vacuum will be used to decontaminate the hood and processing equipment, following the preparation of each sample. Vacuum calibration will be performed daily, prior to grinding activities. All system checks, required maintenance and the vacuum number will be recorded on the Vacuum Maintenance Log (Attachment 5).

A standard BICO vertical plate grinder will be used to process samples. The grinder will be calibrated daily or after any adjustments are made to the plates. To verify proper particle size (approximately 250 µm), and demonstrate that samples will not be over-processed, grind a sample of clean soil (rather than quartz sand) and sieve using stacked sieves. Clean soil will be provided by the United States Geological Survey (USGS). Unlike the coarseness of quartz sand, soil will more accurately approximate the typical grain size and texture of the Libby samples being processed and will reduce the chance of over-processing. Note that the particle size is cited as “approximately 250 µm”. This is due to the nature of grinding asbestos material. Some material that is longer than 250 µm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

the vertical grinder plates will be ground to <250 µm.

The grinder is adjusted acceptably if all material passes through a 60-mesh (250 µm) screen and is substantially retained by a 200-mesh (74 µm) sieve. If the appropriate amount of material does not pass through the stacked sieves, adjust the plates of the vertical grinder until all material processed passes through the aforementioned sieve sizes. Document the grinder number, verification of acceptable adjustment and any observations in the Grinder Calibration and Maintenance Log (Attachment 3).

Following the calibration activities, the stacked sieves will be decontaminated using a HEPA vacuum, compressed air and an aliquot of approximately 20 g of quartz sand will be passed through the grinder before the next sample is processed.

Samples will be weighed following grinding activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded on the Analytical Balance Calibration and Maintenance Log (Attachment 2).

9.2 Grinding Blanks

A grinding blank will be prepared daily, per grinder used, and will be associated with all samples prepared per day, per grinder. The grinding blank will consist of approximately 100-200 grams of clean quartz sand, and will be processed on days that field samples are ground. Each grinding blank will be identified in the notes section of the Sample Preparation Log Sheet (Attachment 1) and will be processed according to the direction of Section 9.3. Grinding blanks will be included with daily shipments to the laboratory. For further information on grinding blanks refer to Section 12.3.

9.3 Grinding of Fine Field Samples

The sample portion that was sieved to < 1/4 inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine sample set aside in Section 8.2, load the grinder hopper, and allow the fine sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of

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grinding, and grinder number on the Sample Preparation Log Sheet (Attachment 1).

9.4 Decontamination

When grinding is complete, do not move the plates for decontamination (this would require re-calibration). Decontaminate the hopper and catch pan by using a HEPA vacuum, followed by a blast of high pressure air. Set the catch pan aside and clean the grinder with several blasts of compressed air. Pay special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps). Reattach the catch pan to the grinder. Pass an aliquot of approximately 20 g of quartz sand through the grinder to clean out any residual soil. Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

10.0 SPLITTING OF THE FINE GROUND SAMPLE

The fine ground soil sample should be distributed into four approximately equal subsamples using a splitter. All splitting activities will be performed in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

10.1 Splitting Procedure for Fine Ground Sample

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997):

Set up one receiving pan on each side of the splitter. Load the soil from the grinder catch pan (Section 9.3) into the splitter, collecting the sample in two receiving pans.

Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.

Empty each receiving pan into the grinder catch pan and sieve catch pan, respectively. Set the sieve pan aside; this portion of fine ground sample will be split again later.

Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample and re-load the contents into the splitter as detailed above. Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to

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uniformly distribute the sample. The resulting splits are the “FG1" and “FG2" portions on the Sample Preparation Log Sheet (Attachment 1).

Take these two portions and carefully transfer each into a clean, tared, zip top sample bag. Re-bag one sample portion in another clean zip top sample bag and identify this fine ground sample with the index ID, the suffix "FG" (for "fine fraction, ground") and the fraction number 1, (ex. CS-12345-FG1 for fine ground fraction #1). Identify the bagged second portion with the Index ID, the suffix “FG” and the fraction number 2 and set aside to be re-bagged with the following fine ground portions:

Place the two empty receiving pans from the “FG1" and “FG2" portion next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the “FG3" and “FG4" portions.

Take the remaining “FG3" and “FG4" portions and carefully transfer each into a clean, tared, ziplock sample bag, identify each remaining fine ground sample with the index ID as noted above.

Weigh each sample portion (FG1 through FG4), and record each mass along with the technician’s initials and date on the Sample Preparation Log Sheet (Attachment 1).

Combine all bagged portions (archive, coarse and fine) into one large clean, zip top sample bag.

Fine ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, the “FG1" fraction will be sent first. If further analyses are required for the fine ground portion, the subsequent fractions will be double bagged and sent (i.e., FG-2 then FG-3, etc.). All archived fine ground portions will be filed in the appropriate inventory archive box noted on the Sample Preparation Log Sheet (Attachment 1).

10.2 Decontamination

Use the vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

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11.0 DOCUMENTATION

Index ID numbers are recorded on the Sample Drying Sheet, Sample Preparation Log Sheet (Attachment 1) and on all sample containers. Sample Drying Sheets and Sample Preparation Log Sheets will be filed under their associated dry batch and preparation batch number. If revisions to the Sample Drying Sheet and/or Sample Preparation Log Sheet are necessary, the appropriate parties will be notified of the changes, however, these changes will not necessitate revision to the current standard operating procedure, a modification form will be filled out to document the revisions.

As mentioned above, the following equipment calibration and maintenance logs will also be maintained:

daily analytical balance calibration using S-1 class weights (Attachment 2)
daily grinder setting verification for calibration check and/or post-adjustment verification, grinder maintenance as necessary (Attachment 3)
daily ventilation hood operating condition verification (i.e., inline filter checks, changes) (Attachment 4)
HEPA vacuum maintenance and bag changes (Attachment 5)
weekly oven temperature calibration, oven maintenance as necessary (Attachment 6)

In addition, a field notebook will be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected:

date
time
personnel
PPE
Governing Plan (CSF SPP, February 2004) and TSOP including revision number
descriptions of any deviations to the SOP, the reason for the deviation and/or any modification forms being followed
summary of laboratory activities (including number of samples prepared, and equipment

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calibrated and used)

12.0 QUALITY ASSURANCE

All quality control sample results will be monitored for potential contamination. If samples results indicate cross-contamination, the PL2 will be notified. The PL2 will then identify the affected samples and notify the appropriate parties of these samples. Laboratory procedures will also be re-assessed and appropriate changes will be made and documented accordingly by the PL2.

12.1 Drying Blanks

At least one drying blank will be processed with each batch (per oven) of approximately 15 field samples (Section 6.2). Results from each drying blank will determine if cross-contamination occurred during the drying process. The drying blank, consisting of clean quartz sand, will be assigned a random and unique index identification number and will be submitted to the laboratory blind. Detection of asbestos fibers in any drying blank (at the practical quantitation limit of about 0.1-0.2% LA) should be taken as a sign of potential cross-contamination, and steps should be taken to identify and address the source of the cross contamination.

12.2 Preparation Duplicates

One preparation duplicate sample (Section 7.3) will be processed for every 20 field samples prepared. Results from duplicate samples serve to evaluate the precision of the sample preparation process and of the laboratory analysis. A preparation duplicate is prepared by using a riffle splitter to divide the sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are prepared in the same fashion. The preparation duplicate is assigned a unique and random index identification number, and is submitted to the laboratory blind. Inconsistent sample results should be taken as an indication of variability in sample preparation, and steps should be taken to identify and address the source of the variability in sample preparation.

12.3 Grinding Blanks

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

One grinding blank (Section 9.2), consisting of clean quartz sand, will be processed once per day, per grinder, on days that field samples are ground. Each grinder used in the lab will be assigned a number and all samples processed will be associated with the grinder used for preparation. The grinder number used for each sample will be noted on the Sample Preparation Log Sheet (Attachment 1). Grinding blanks will not be dried, split for archive, or sieved, a grinding blank will only be ground and split into four fine ground samples. Results from the grinding blank will determine if decontamination procedures of laboratory equipment are adequate in preventing cross-contamination of samples during sample grinding and fine ground sample splitting processes only. The grinding blank is assigned a random and unique index identification number and is submitted to the laboratory blind. If asbestos fibers are detected in any grinding blank the PL2 will be notified. The PL2 will identify all samples that were processed on the day the grinding blank was prepared, and the grinder that was used to process the grinding blank. Detection of asbestos fibers in any drying blank (at the practical quantitation limit of about 0.1-0.2% LA) should be taken as a sign of potential cross-contamination, and steps should be taken to identify and address the source of the cross contamination.

13.0 DECONTAMINATION

All non-disposable equipment used during sample preparation must be decontaminated prior to use. Scoops or spoons, splitters, sieves and drying pans that are re-used must be decontaminated with a HEPA vacuum, compressed air, wet-wiping and/or by brushing off any residual material. If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean.

Detailed decontamination procedures for specific equipment are noted in Sections 6.4, 7.4, 8.3, 9.4, and 10.2.

14.0 GLOSSARY

HEPA - High Efficiency Particulate Air

Project Plan - The written document that spells out the detailed site-specific procedures to be followed by the Project Leader and the Preparation Lab Personnel.

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

15.0 REFERENCES

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

CDM. 2004. Close Support Facility Soil Preparation Plan, Libby Asbestos Site, Operable Unit 4, Libby, Montana. February.

USEPA. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

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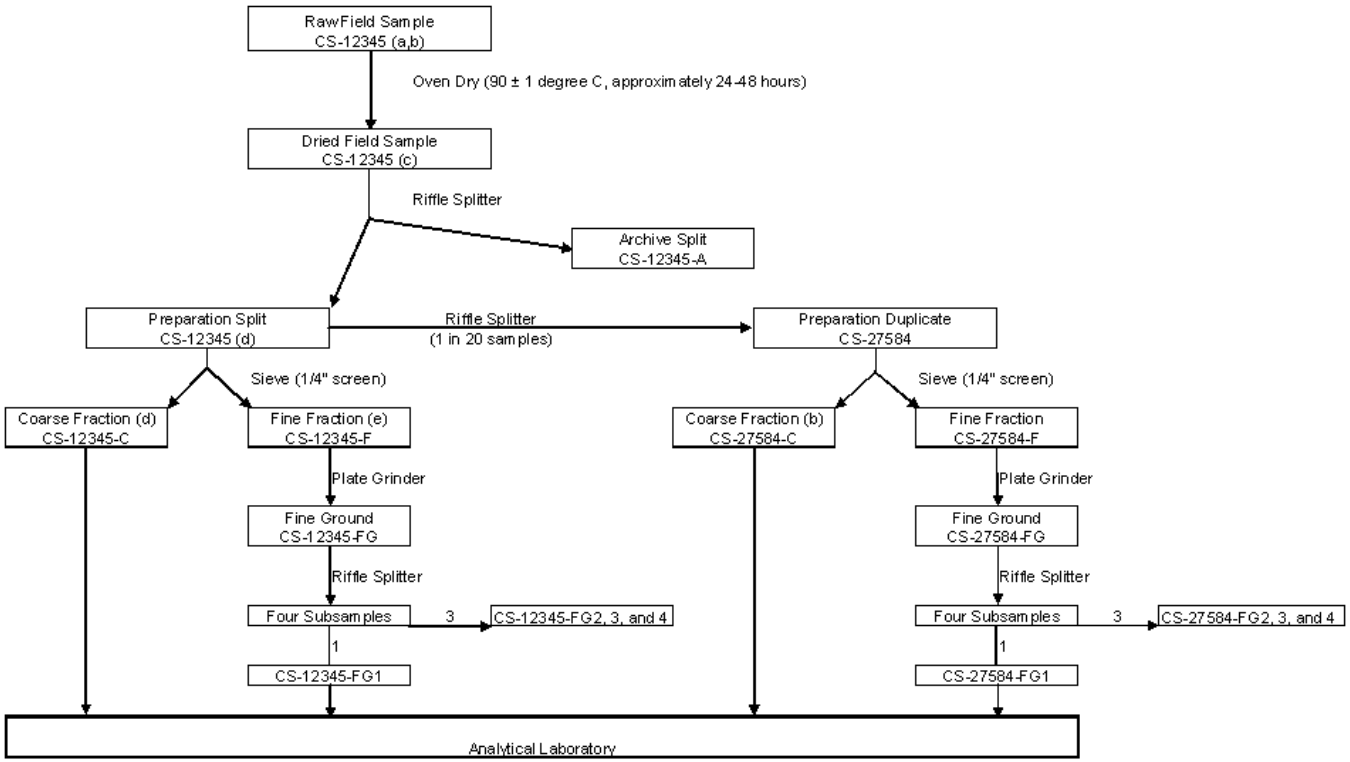
Figure 1 Soil Sample Preparation Flow Diagram



(a) Example sample number shown to illustrate naming conventions
(b) Drying blanks, created with clean quartz sand (Section 5.2) will be processed with each batch using the same sample processing procedures outlined in ISSI-Libby-01 (Rev 7)
(c) If the sample is designated as a duplicate, the sample will follow the duplicate splitting process defined below. If the sample is not a designated duplicate, it will proceed to the sieving step defined below.
(d) Coarse sample will be returned to CDM CSF for archive after laboratory analysis
(e) Grinding blanks (Section 8.3), created with clean quartz sand, will be ground and split into four fine ground samples using the same procedures outlined in ISSI-Libby-01 (Rev 7)

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 1

SAMPLE DRYING AND SAMPLE PREPARATION LOG SHEETS

TECHNICAL STANDARD OPERATING PROCEDURE

ISSI-LIBBY-01, Revision 8: SOIL SAMPLE PREPARATION

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Sample Drying Log Sheet

10-23-2003, Revision 0

Dry Batch ID _____ Sheet No.: CSF- _____

Date/Time Drying Begun _____

Date/Time Drying Completed _____

Oven Number _____

Oven Temp (C) _____

Index ID	Inventory ID Number	SOP and Rev Number	Mass (g)			Original Sample Identification and Notes (Indicate if dry blank sample)	QC
			Before Drying	After Drying	Initials and Date		Initials and Date

The following preparation steps require Technician Initials and Date to document activity: Sample Mass, Data Entry QC

Page 1 of 1

TECHNICAL STANDARD OPERATING PROCEDURE

ISSI-LIBBY-01, Revision 8: SOIL SAMPLE PREPARATION

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Sample Preparation Log Sheet

10-23-2003, Revision 2

Prep Batch ID _____

Sheet No.: CSF- _____

Index ID	SOP and Rev Number	Inventory ID	Dry Batch ID	Archive Sample Splitting	Duplicate Sample Splitting	Sieving			Sample Grinding		Sample Splitting					Original Sample Identification and Notes (Indicate if grind blank, dry blank, or duplicate pair. For duplicate pair enter the parent/child ID)	QC
						Sample Mass (g)	Sample Mass (g)				Sample Mass (g)						
				Initials and Date	Initials and Date	Fraction >1/4"	Fraction <1/4"	Initials and Date	Initials and Date	Grinder #	FG1	FG2	FG3	FG4	Initials and Date		Initials and Date

The following preparation steps require Technician Initials and Date to document activity: Archive Sample Splitting, Preparation Duplicate Splitting, Sieveing, Sample Homogenization, Sample Splitting, and Data Entry QC

Page 1 of 1

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 2

ANALYTICAL BALANCE CALIBRATION AND MAINTENANCE LOG

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Balance # = _____

Measurement Number		S - 1 Class Weight Measurements				Measurement within range? Yes or No	If "No" Recalibrate	Technician Initials	QC check initials
	Calibration Weights	0.1 g	1 g	10 g	100 g				
	Tolerance Limit Range	0.05 - 0.15 g	0.90 - 1.10 g	9.75 - 10.25 g	99.00 - 101.00 g				
	Date								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
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16									
17									
18									
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21									
22									
23									
24									
25									

The analytical balance calibration will be verified daily.
All tolerance limits are standard tolerance limits for Class S-1 weights.
After 20 measurements, the tolerance range will be evaluated for reasonableness.
Weights falling outside the range require that the balance be recalibrated using all S-class weights

BBY-

Sheet No.: Balance - _____

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 3

GRINDER CALIBRATION AND MAINTENANCE LOG

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Grinder # = ____________________

Date		Sieve Test				
	Activity Type (D, A, M)	60 mesh acceptable Y or N	200 Mesh acceptable Y or N	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

D = daily calibration
A = Adjustment Calibration Verification
M = Maintenance Only
For the 60 mesh sieve test to be acceptable all of the clean soil must pass through the sieve.
For the 200 mesh sieve test to be acceptable a substantial portion of the ground soil must be retained on the sieve.
Failure of either sieve test requires adjustment of the plates followed by adjustment verification prior to grinding samples.

Sheet No.: Grinder - ______________

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 4

VENTILATION HOOD CALIBRATION AND MAINTENANCE LOG

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Ventilation Hood # = _______________

Date	In-line HEPA indicators acceptable?	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

Ventilation system operating conditions should be verified daily
In-line system should indicate the velocity/negative flow is 100 ft/min. If not, the problems with the system must be corrected prior to processing samples. Note actions or maintenance taken.

Sheet No.: Hood - ____________

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 5

VACUUM MAINTENANCE LOG

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Vacuum # = ________________

Date	System Check Acceptable? Yes or No	HEPA filter clean? Yes or No	Bag fill level acceptable? Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Physically check the vacuum suction and note audible change in motor daily. If significant reduction in the vacuum suction or motor "strain" is audibly noted then the system check is unacceptable. If unacceptable, perform and document the HEPA filter and bag maintenance checks above and perform maintenance.

Sheet No.: Vacuum - ______________

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

ATTACHMENT 6

OVEN TEMPERATURE CALIBRATION AND MAINTENANCE LOG

TECHNICAL STANDARD OPERATING PROCEDURE
ISSI-LIBBY-01, Revision 8: SOIL SAMPLE PREPARATION

NOTE: This SOP was designed for use on Libby Project Soils. For any other use the SOP needs to be re-evaluated based on specific project objectives

Oven # = _____

Date	Certified Temperature Reading (°C)	Thermometer ID	System Check Acceptable? 90°C (+/- 1°C) Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Sheet No.:Oven - _____ -LIBBY-

Appendix B

Health and Safety Plan

VOLUME V

HEALTH AND SAFETY PLAN

BNSF RAILWAY MAINTENANCE OF WAY

ACTIVITY BASED SAMPLING

LIBBY, MONTANA

EMR PROJECT 5539-020

Prepared for:
Mr. David Smith
Manager Environmental Remediation
The Burlington Northern and Santa Fe Railway Company
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Helena, Montana 59601

Prepared by:

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Duluth, Minnesota 55802

SEPTEMBER 2008

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1.0 INTRODUCTION

The following Health and Safety Plan (HSP) has been developed to provide a safe operational framework in which Activity Based Sampling (ABS) can be conducted. This HASP will describe the following

- Scope of Work (Section 1.1)
- Work Environment (Section 1.2)
- Personal Protective Equipment (Section 2.1)
- Personnel Training Requirements (Section 2.2)
- Daily Safety Procedures (Section 3.0)
- Decontamination and Dust Suppression Procedures (Section 4.0)
- Accident Prevention Plan (5.0)

1.1 SCOPE OF WORK

The focus of this project will be the collection of data, through ABS, to assess the human health risk associated with maintenance activities along portions of the BNSF Kootenai River Subdivision in the vicinity of Libby, Montana. ABS will be conducted during planned track structure maintenance consisting primarily of rail replacement and surfacing activities. This phase of the project will include sample collection from the following media and or receptors:

- personal air monitoring on BNSF maintenance personnel;
- personal air monitoring on simulated trespassers;
- perimeter air monitoring;
- soil sampling adjacent to the Right-of-Way (ROW).

The work will be closely coordinated with BNSF maintenance forces to ensure that the collected data meets the Data Quality Objectives and the work is completed without any Health and Safety incidents. The work will be performed in accordance with applicable Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA), BNSF and EMR health and safety requirements. A site-specific Accident Prevention Plan is included in Section 5.0. A copy of this Health and Safety Plan will be present on Site for the duration of the work activities.

1.2 WORK ENVIRONMENT

The project site (Site) is located along a portion of BNSF Kootenai River Subdivision, a mainline that runs from Whitefish, Montana to Sandpoint, Idaho. The work will occur between Mileposts (MP) 1314 and 1339 which traverses through Libby and Troy, in Lincoln County, Montana.

The Site consists of approximately nine (9) individual areas where BNSF will be performing maintenance. The sites, which are subject to change, are named as follows:

- MP 1314
- MP 1316
- MP 1327
- MP 1329
- MP 1330.3
- MP 1330-1331.8
- MP 1333
- MP 1336
- MP 1338

Each work area has unique characteristics, but the following is a general description of the Site. The BNSF mainline follows a deeply incised valley created by several processes including erosion by the Kootenai River. Through the entire Site the BNSF mainline is within 500 feet of the south bank of the Kootenai. Although public roads parallel the BNSF tracks the distance to these roads varies from less than 100 feet to more than 1,000 feet. Between Libby and Troy US Highway 2 is the primary route from which the BNSF mainline can be accessed. Much of the Site is lightly populated, with the exception of the areas immediately adjacent to Libby and Troy. It should be noted that cell phone coverage is intermittent at best, outside of Libby and Troy.

This sampling effort will be conducted in correlation with BNSF maintenance activities that includes: ribbon rail removal and replacement; ballast regulation and tamping; and possibly tie removal and replacement. Numerous machines, personnel and vehicles will be part of the work, creating a unique set of hazards for sampling personnel. No formal designation of Exclusion Zones (EZ) or Contamination Reduction Zones (CRZ) will be attempted during this sampling effort. In place of EZ and CRZ designations, Work Zone (WZ) will be used to designate the area on and within 25 feet of the center line of the mainline where rail maintenance work is being conducted or sampling is taking place.

2.0 PERSONAL PROTECTIVE EQUIPMENT AND TRAINING REQUIREMENTS

2.1 PERSONAL PROTECTIVE EQUIPMENT

All personnel entering the WZ will have appropriate Personal Protective Equipment (PPE). All workers entering the WZ will be required to wear protective equipment including but not limited to:

- Disposable Tyvek® or equivalent coveralls with hood;
- Half face or full face air purifying respirator with P100 HEPA cartridges;
- Disposable Nitrile or equivalent gloves;
- Hearing Protection in areas where noise levels exceed 85db;
- Hard hat with reflective tape;
- Orange reflective vest;
- Safety glasses (if no full face respirator is being worn); and
- Steel-toed boots.

All BNSF contractor personnel (EMR representatives or subcontract laboratory visitors) and EPA Representatives (CDM, EPA or Volpe Center) will be required to wear baseline BNSF PPE while on BNSF property. Baseline BNSF PPE consists of a hard hat, orange reflective vest, safety glasses with side shields and steel toed work boots.

2.2 TRAINING/CERTIFICATION REQUIREMENTS

All Contractor Personnel will be required to have successfully completed an approved BNSF Contractor Orientation coarse and must have security clearance through the Erailsafe program.

Different training requirements are required depending on tasks within the WZ. At a minimum, all Contractor Personnel will be required to have 40 hour Hazardous Waste Operations (HAZWOPER) training (29 CFR 1910.120). All Contractor Employees entering the WZ to conduct air monitoring will be required to have 32 hour EPA asbestos worker or 40 hour EPA asbestos supervisor training or accompanied by certified personnel. At least one 40 hour asbestos supervisor will be on site during all air monitoring activities.

Any project management personnel entering the but not conducting or assisting in rail, tie or soil removal will be required to have two hour EPA asbestos awareness training, respirator training and fit test, and 40 hour HAZWOPER training. Air monitoring personnel working outside the WZ are required to have HAZWOPER training at a minimum.

3.0 DAILY SAFETY PROCEDURES

All Contractor Personnel will be required to read, understand and sign this HSP.

All Contractor Personnel and EPA Representatives will be required to attend the daily BNSF Safety Briefing. Attendance of this meeting is critical to ensure that Contractor Personnel and EPA Representatives understand of the rail maintenance activities that will be conducted and potential safety hazards posed by these activities. Since Contractor Personnel will be working in close proximity to BNSF maintenance equipment and personnel it is imperative that Contractor Personnel have a working knowledge of the day's activities and anticipated hazards. All Contractor Personnel should leave the meeting with a knowledge of emergency procedures.

Following the BNSF Safety Briefing, all Contractor Personnel will conduct an additional daily health and safety briefing. These daily health and safety briefings will discuss the hazards expected to be encountered each day during work activities including, at a minimum, the hazards of asbestos, heavy equipment, and heat or cold related health hazards. Additional Contractor Personnel health and safety briefings will be conducted when there are changes in work location and assignments, weather conditions, or when other conditions warrant additional briefings. The Contractor Personnel health and safety briefings will be documented on the form found in Appendix A.

4.0 DECONTAMINATION AND DUST SUPPRESSION REQUIREMENTS

4.1 DECONTAMINATION PROCEDURES

Contractor vehicles are required to be outside of the WZ. Contractor equipment required for the project will be allowed to enter the WZ. However, equipment within the WZ will be minimized to decrease the potential for contamination tracking and to minimize activity within the WZ. No restraints will be placed on the placement of BNSF equipment within the since BNSF activity is focus of the sampling event. No efforts will be made to decontaminate vehicles, equipment or machines operated by BNSF or Contractors. Personal decontamination will be conducted as described below.

4.1.1 Personal Decontamination

All Contractor Personnel working in the WZ will decontaminate upon leaving the Work Zone. Personal decontamination will consist of the following procedure:

- Remove Tyvek suit;
- Gross removal of soil from boots with scrub brush;
- Water boot rinse using Hudson-type (positive pressure) sprayer;
- Remove gloves;
- Remove respirator.

All used PPE (except respirators) will be placed in a suitable container, labeled and stored for disposal. Respirators will be wet wiped, disinfected, dryed and placed in a clean plastic reclosable bag. Duct tape will be placed over the HEPA cartridge inlet between uses.

4.1.2 Personal Hygiene

No eating, drinking of beverages, use of tobacco, or applying of cosmetics will be allowed in the Work Zone. None of these activities may be done during the work day unless the worker is outside the Work Zone.

4.2 AIR SAMPLING EQUIPMENT DECONTAMINATION

Air sampling equipment (e.g., pumps, cassette holders, etc.) that is used within the Work Zone will be decontaminated prior to removal. Air sampling pumps, tubing, sampling stands, and rotometers will be cleaned with wet disposable wipes and dried with clean paper towels. All disposable wipes and towels will be properly stored and disposed of with used, disposable PPE.

4.3 DUST SUPPRESSION PROCEDURES

Soil wetting will not be permitted during soil sampling activities as per the Sampling and Analysis Plan (SAP). Wetting will not be allowed in order to evaluate moisture content of

the soils during sampling. Active wetting will not be conducted by BNSF during maintenance activities.

5.0 ACCIDENT PREVENTION PLAN

An Accident Prevention Plan has been prepared. Accidents will be prevented by reviewing hazards, conducting daily safety briefings, and being aware of on-site activities at all times. Phone numbers and directions of emergency services will be posted in the personal decontamination trailer. Tailgate safety meeting forms with attached egress routes and closest hospital information are attached as Appendix A.

5.1 GENERAL INFORMATION

Project Name: BNSF Railway Maintenance of Way Activity Based Sampling

EMR Project No.: 5539-020

Project Manager: Scott Carney

Project Field Manager: David L. Welch

Location: Libby, Montana

Prepared by: Scott Carney **Date prepared**: 9/8/08

Revised by: Dave Welch **Date prepared**: 9/9/08

Approval by: Chuck Hendrix **Date**: 9/9/08

Scope/Objective of Work: Soil and air sampling will be conducted to evaluate the magnitude of potential risk associated with the disturbance of residual Libby Amphibole (LA) caused by railroad maintenance activities. Personal air samples will be collected to determine exposures to both worker and simulated trespassers. Stationary air monitoring samples will be collected to determine the ambient air quality during railroad maintenance activities. Soil sampling will be conducted in order to potentially correlate LA soil concentrations with fiber content in air samples. Personal air samples will be analyzed using a combination of Phase Contract Microscopy (PCM) and Transmission Electron Microscopy (TEM). Soil samples will be analyzed by Libby amphibole (Tremolite/Actinolite Series) Method 9002, Issue 2.

Proposed Date(s) of Field Activities: Between September 17, 2008 and September 22, 2008, weather permitting.

Background Information: ___ Complete _X_ Preliminary (analytical data incomplete)

Documentation/Summary:

Overall Chemical/Waste hazard: __ Serious __ Moderate X Low __ Unknown

Overall Physical hazard: __ Serious X Moderate __ Low __ Unknown

5.2 SITE/WASTE CHARACTERIZATION

Waste Type(s): __ Liquid X Solid __ Sludge __ Gas/Vapor X Aerosols (Dust)

Characteristics:

__ Flammable __ Volatile __ Corrosive __ Acutely toxic (LBP)

X Carcinogen (ACM) __ Reactive __ Explosive __ Radioactive

Physical Hazards:

X Overhead ___ Confined Space ___ Below Grade X Trip/fall

__ Puncture __ Burn X Cut __ Splash X Noise

Other: Work will be conducted in close proximity to BNSF rail-based maintenance equipment. Rail placement and ballast regulation will occur during the sampling and introduces sprung metal and flying debris hazards Though freight and passenger trains are a recognized hazard, BNSF personnel will provide track protection during work activity and communicate train hazards to contractor personnel and EPA representatives on a daily basis.

Site History/Description and Unusual Features: The majority of work will be conducted during rail replacement and surfacing activities. This work will include numerous rail-based machines as well as several support vehicles. All Contractor Personnel will need to be aware of the activities occurring around them. In addition the work areas will likely shift on a daily basis, if not more frequently.

Locations of Chemicals/Wastes:
The WZ may or may not have visible hydrated biotite (vermiculite) that would potentially contain LA. Railroad right-of-way soils and ballast in OU6 that contains LA will be disturbed by the rail-based maintenance equipment, resulting in potential airborne asbestos.

Estimated Volume of Chemicals/Waste: Unknown

Sites currently in operation: X Yes __ No

5.3 HAZARD EVALUATION

Prior to each day of work, a "tailgate" safety meeting will be held to review the overall Health and Safety Plan, specific physical and chemical/waste hazards for the Site, and railroad safety and track protection. Since the work site will change on a daily basis it will be especially important to discuss routes of egress from the Site and routes to the nearest hospital in case of accident. Following the meeting, site personnel will sign a sheet with the current date and the date that they reviewed the Health and Safety Plan and discussed specific hazards and routes of egress for the Site. The sign-off sheet for each day will identify the specific work planned for that day. Cellular telephone reception will be evaluated during the tailgate safety meeting to determine whether it will be available in case of an emergency. It is likely that cell phone reception will not be available at all of the work sites. In the case of emergency, there will be two possible scenarios:

Scenario 1: Non-life threatening: Personnel will be escorted out of WZ and assisted in decontamination prior to delivery of injured worker to hospital. BNSF personnel will be immediately notified and first aid will be offered to the injured.

Scenario 2: Life-threatening: EMR personnel will contact BNSF personnel, call 911 or other means of communication, and inform emergency personnel how to access the site. If possible, EMR personnel will meet emergency personnel at the nearest access point to expedite arrival of emergency personnel at the incident site. Emergency personnel will don PPE, attend to and stabilize victim, bring the victim out of the W Z, and decontaminate the victim and themselves according to decontamination procedures outlined in Section 4.1.1at and deliver victim to hospital. If delay will threaten the victim's life, medical personnel and the site supervisor may weigh exposure risk as it relates to the effect of delay caused by PPE and decontamination.

5.3.1 Physical Hazard Evaluation/Control:

1. Hazard: Work around rail maintenance equipment mechanical hazards.

   Control: Daily tailgate meeting to identify hazards. Worker in the WZ must be aware of maintenance work being conducted around Contractor Personnel.

2. Hazard: Work around long lengths of welded rail.

   Control: Contractor Personnel must be aware of the work activities being conducted around them. Contractor Personnel will not be in the WZ during rail removal or placement.

3. Hazard: Slip, trip and fall hazards will be a constant in the Work Zone.

   Control: Daily tailgate meeting to identify hazards in the Work Zones. Prior to entering the Work Zone, Contractor Personnel will discuss any

additional slip, trip and fall hazards that may have been created since the tailgate safety meeting

5.3.2 Chemical/Waste Hazard Evaluation/Control

(Source: NIOSH pocket guide to chemical hazards-June 1997)

Chemical (Mineral) Hazard:

COMPOUND	PEL/TWA	ROUTE OF EXPOSURE	ACUTE SYMPTOMS	ODOR THRESHOLD	ODOR DESCRIPTION
Asbestos	0.1 fiber/cc	Inhalation Ingestion Contact	None	None	None

Control:

Personal protective equipment will be the primary control since wetting will not permitted during soil sampling and general wetting of the WZ will not be possible. Air monitoring will be used to determine airborne fiber content.

5.4 SITE SAFETY WORK PLAN

Site Control:

__ Perimeter identified __ Site secured X Work area designated

__ Zones of contamination identified

Personnel Protection: Tyvek® suit (or equivalent), hardhat, steel-toed boots, safety glasses, reflective safety vest, hearing and respatory protection.

Anticipated Levels of Protection: Modified Level C

Modifications: Half-face air purifying cartridge respirators with P100 HEPA cartridges.

Action Levels for Evacuation of Work Zone: N/A (air monitoring detection will result in a change in the methods and/or procedures).

Air Monitoring Required: Personnel and stationary monitoring will conducted as per the Sampling and Analysis Plan

Decontamination Solutions and Procedures for Equipment, Sampling Gear, etc.: water solution

Personnel Decontamination Protocol: See Section 4.1.1

Decontamination Solution Monitoring Procedure, if applicable: Not Applicable

Special Site Equipment, Facilities, or Procedures (sanitary facilities, lighting, etc.): Portable toilets and a water supply will not be available on site. The only traffic expected during sampling activities will be railroad maintenance equipment, train traffic will not be possible during the maintenance window.

Site Entry Procedures and Special Considerations: Site entry will be variable since the work area will change on a daily basis. Scouting of the best entry route will be conducted as necessary. Local railroad personnel will be the best source of access information. The primary public roads that will be used to access the Work Zones is the Champion Haul Road (east of Libby) and US Highway 2 (west of Libby).

Work Limitations (time of day, weather conditions, etc.) and heat/cold stress requirements:

- Work will be conducted during daylight hours.
- Adequate breaks will be taken during the day to avoid cold or heat stress. It is not anticipated that Contractor Personnel will be required to spend extended periods of time within the WZ. Contractor Personnel will be allowed to decontaminate

hands, respirators and faces at the edge of the WZ and consume liquid for hydration.

- Work will stop for 15 minutes for each audible or visual lightning strike. Work will begin after 15 minutes pass without any record of strikes.

General Spill Control, if applicable: NA

Investigation-Derived Material Disposal (i.e. expendables, decontamination waste, cuttings): Expendables will be bagged for appropriate disposal.

Sample Handling Procedures, including protective wear: disposable gloves, safety glasses, respirators.

5.5 EMERGENCY INFORMATION

Ambulance:..... 911 or BNSF Radio

Hospital Emergency Room: St. Johns Lutheran Hospital
350 Louisiana Avenue
Libby, MT 59923
(406) 293-0100

Poison Control Center:..... 1-800-542-6319
Police/Sheriff: 911
FIRE Department: 911

Agency contacts (EPA, state, local):

EPA Regional Office, Emergencies	(406) 293-6194
MDHE	(406) 444-2544

EMR Contacts:

Scott Carney Work: (218) 625-2331 Cellular (218) 393-0936 Home: (218) 724-7685
Paul Fowler Work: (785) 842-9013 Cellular (785) 764-0186
Tom Patnode Work (785) 842-9013 Cellular (425) 417-8814
Dave Welch Work (425) 861-451 Cellular (425) 512-5510
Amanda Thornton Work: (425) 861-4561
John Starr Work (785) 842-9013 Cellular (785) 766-7003

Water Supply Source: BNSF water supply, or portable water containers will be used. No significant source of potable water will be available at the work sites.

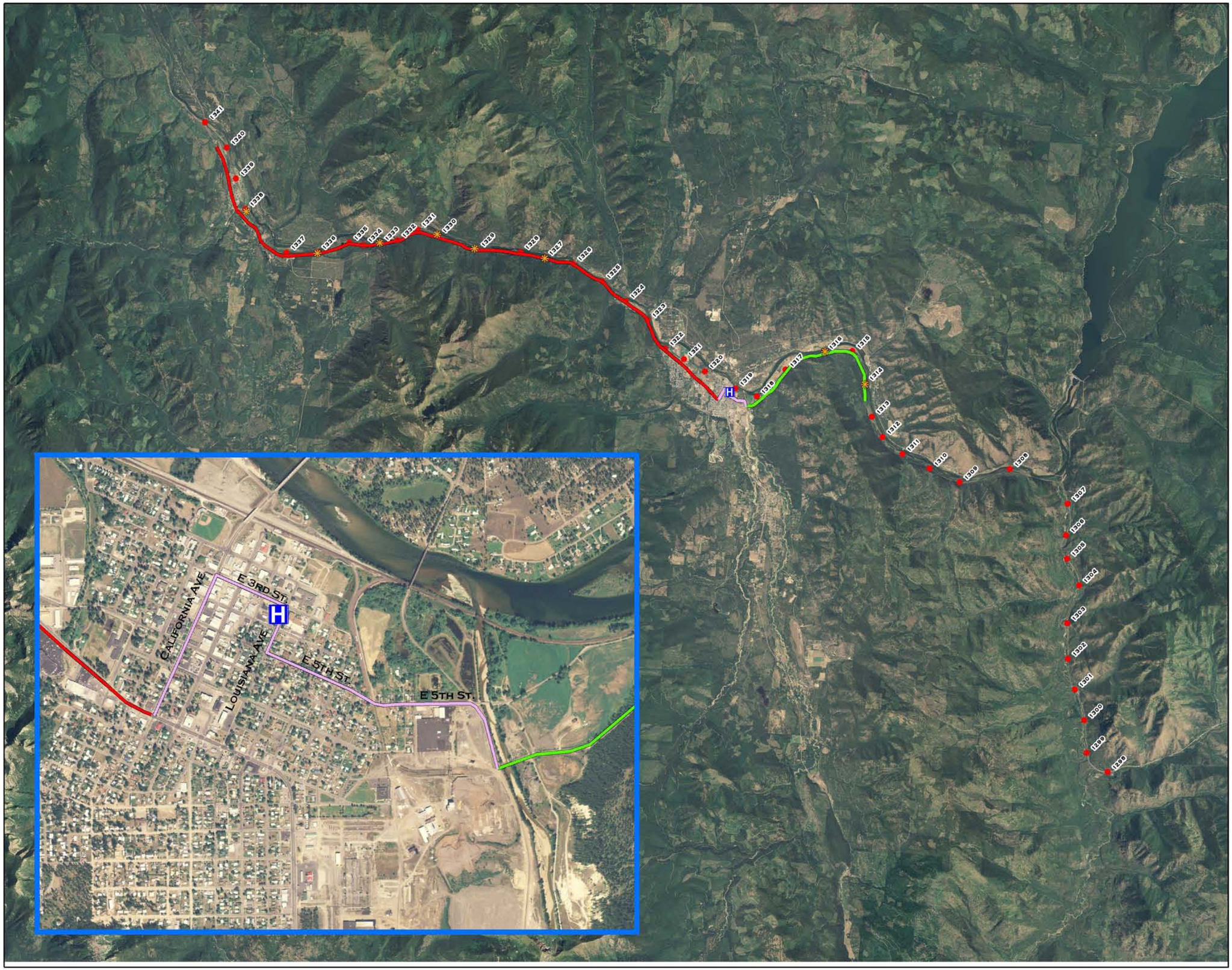
Hospital Address: St. Johns Lutheran Hospital, 350 Louisiana Avenue, Libby, Montana

Directions: To be attached to each "tailgate" safety meeting sign-off sheet.

Emergency Egress Routes for Site Evacuation: Exit site to nearest public road which will vary according to the specific work location. The primary public access roads will be the Champion Haul Road (east of Libby) and US Highway 2 (west of Libby). (See attached pages).

APPENDIX A

ROUTE TO HOSPITAL MAP



N
W E
S

Route to Hospital Map
BNSF Kootenai River Sub

St. Johns Lutheran Hospital
350 Louisiana Avenue
Libby, MT 59923
(406) 293-0100

Legend

- Steel Gang Work Sites
- St Johns Lutheran Hospital
- Route to Hospital
- Champion Haul Road
- Highway 2
- Approximate Milepost Locations

0 7,500 15,000 30,000
Scale In Feet

0 1 2 4
Scale In Miles

EMR
INCORPORATED

APPENDIX B

DAILY TAILGATE SAFETY FORM

DAILY TAILGATE SAFETY MEETING FORM

DATE:_____**INSTRUCTOR**: _____
PROJECT NAME/LOCATION: _____
TODAYS ACTIVITIES: _____
CHEMICALS/HAZARDS PRESENT: _____

SAFETY TOPICS DISCUSSED

Protective Clothing/Equipment Required: A B C D
Describe: _____
_____

Hazards of Chemicals Present: inhalation dermal ingestion contact
Describe: _____
_____

Physical Hazards: mechanical electrical pneumatic hydraulic weather
slips trips and falls heavy equipment noise
Describe: _____
_____

Heavy Equipment on Site (List):_____
Attach daily equipment inspections to this sheet.

Startup Procedures: hot line wind direction radio/communication check
air monitoring site notifications PPE checked
Describe:_____
_____

Other Hazards: confine spaces hot worktraffic flow
Describe_____
_____

Track Protection Required?: Yes / No (circle one)
Type of Track Protection (Form B, flagman, etc):_____

Name of Person Providing Protection: _____

Limits of Track Protection:_____

Expiration Time of Track Limits: _____

Communication/Warning Methods:_____

ATTENDEES

Name (printed)	**Signature**

APPENDIX C

DOCUMENT SIGNATURE PAGE

HEALTH AND SAFETY DOCUMENT SIGNATURE PAGE

I have read and understand the safety rules described in the Health and Safety Plan.

NAME	SIGNATURE	COMPANY	DATE

Appendix C

Field Change Order (FCO) Form

Field Change Order (FCO)

DATE:
ADDRESS: BNSF ROW
PROJECT NAME: Libby Asbestos Project

DESCRIPTION OF CHANGE AND RATIONALE

SIGNATURE APPROVALS

EPA MANAGEMENT: _____ DATE: _____

EMR MANAGEMENT: _____ DATE: _____

Appendix D

Field Sample Data Sheets

Sheet No.: PA- **00«seq»**

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR PERSONAL AIR

Field Logbook No: _____ Page No: _____ Sampling Date: _____

Address: _____ Owner/Tenant: _____

Business Name: _____

Land Use: Residential School Commercial Mining Roadway Other ( )

Sampling Team: CDM Other _____ Names:_____

Person Sampled: _____ SSN:_____ Task:_____

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)
Matrix Type (circle)	Indoor Outdoor	Indoor Outdoor	Indoor Outdoor
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8
Flow Meter Type (circle)	Rotometer DryCal NA	Rotometer DryCal NA	Rotometer DryCal NA
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes NA	No Yes NA	No Yes NA
MET Station onsite?	No Yes NA	No Yes NA	No Yes NA
Sample Type	TWA EXC NA	TWA EXC NA	TWA EXC NA
Field Comments Cassette Lot Number: _____	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No
Entered (LFO) _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by	QC by

vs 10507

Sheet No.: SA-008609

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR STATIONARY AIR

Field Logbook No: _____ Page No: _____ Sampling Date: _____

Address: _____ Owner/Tenant: _____

Business Name: _____

Land Use: Residential School Commercial Mining Roadway Other ( )

Sampling Team: CDM Other _____ Names: _____

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS FB-(field blank) LB-(lot blank) DB-(prep-dry blank)	FS FB-(field blank) LB-(lot blank) DB-(prep-dry blank)	FS FB-(field blank) LB-(lot blank) DB-(prep-dry blank)
Matrix Type (circle)	Indoor Outdoor NA	Indoor Outdoor NA	Indoor Outdoor NA
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8
GPS Status (circle)	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected-not required for sample	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected- not required for sample	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected- not required for sample
GPS File (fill in or circle)	Filename:_____ NA	Filename:_____ NA	Filename:_____ NA
Flow Meter Type (circle)	Rotometer DryCal NA	Rotometer DryCal NA	Rotometer DryCal NA
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes NA	No Yes NA	No Yes NA
MET Station onsite? (circle)	No Yes NA	No Yes NA	No Yes NA
Sample Type (circle)	Pre Post Clear 2nd Clear 3rd Clear NA	Pre Post Clear 2nd Clear 3rd Clear NA	Pre Post Clear 2nd Clear 3rd Clear NA
Field Comments Cassette Lot Number:_____	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No
Entered (LFO): _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by:	QC by:

vs 10507

Sheet No.: S- **00«seq»**

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR SOIL

Field Logbook No: _____ Page No: _____ Sampling Date: _____

Address: _____ Owner/Tenant: _____

Business Name: _____

Land Use: Residential School Commercial Mining Roadway Other ( )

Sampling Team: CDM Other _____ Names:_____

Data Item	Sample 1	Sample 2	Sample 3
Index ID			
Location ID			
Sample Group			
Location Description (circle)	Back yard Front yard Side yard Driveway Other _____	Back yard Front yard Side yard Driveway Other _____	Back yard Front yard Side yard Driveway Other _____
Category (circle)	FS FD of _____ EB LB	FS FD of _____ EB LB	FS FD of _____ EB LB
Matrix Type (Surface soil unless other wise noted)	Surface Soil Other _____	Surface Soil Other _____	Surface Soil Other _____
Type (circle)	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____
GPS Status (circle)	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected-not required for sample	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected-not required for sample	Collected Previously Collected Not Collected-no signal (3 attempts) Not Collected-not required for sample
GPS File (fill in or circle)	Filename:_____ NA	Filename:_____ NA	Filename:_____ NA
Sample Time			
Top Depth (inches below ground surface)			
Bottom Depth (inches below ground surface)			
Field Comments *Note if vermiculite is visible in sampled area*	BD-_____	BD-_____	BD-_____
Entered (LFO) _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by:	QC by:

vs 10507

Appendix E

Libby Asbestos Project Record of Modification Form

Record of Modification

to the

Libby Sampling and Quality Assurance Project Plan

Field Activities

LFO-0000__

Instructions to Requester: **Fax to contacts at bottom of form for review and approval.**
File approved copy with Data Manager at the Libby Field Office (LFO).
Data Manager will maintain legible copies in a binder that can be accessed by LFO personnel.

Project QAPP (circle one): Phase I (approved 4/00) Phase II (approved 2/01)
Removal Action (approved 7/00) Contaminant Screening Study (approved 5/02)
Other (Title and approval date):_____

SOP (Number and Revision No.):_____
_____

Other Document (Title, Number/Revision): _____

Requester: _____ Title: _____
Company: _____ Date: _____

Description of Modification (attach additional sheets if necessary; state section and page numbers of SQAPP that are affected by the proposed modification):_____
_____
_____

Field logbook and page number where Modification is documented (or attach associated correspondence):
_____

Potential Implications of Modification: _____
_____
_____

Duration of Modification (circle one):

Temporary Date(s):_____
Resident address(es): _____
_____

- If appropriate, attach a list of all applicable Index Identification numbers.

Permanent (Proposed Text Modification Section) Effective Date:_____

Proposed Text Modifications in Associated Guidance Document (attach additional sheets if necessary):____
_____
_____

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Technical Review and Approval: _____ Date:_____
(Volpe Project Manager or designate)

EPA Review and Approval: _____ Date:_____
(USEPA RPM or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Appendix F

Analytical Requirements Summary

SAP ANALYTICAL SUMMARY # XXXXX-0000
SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS FOR ASBESTOS

SAP Title: _____

SAP Date (Revision): _____

EPA Technical Advisor: _____
(contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: _____
_____
_____
_____
_____

Index ID Prefix: _____

Medium-Specific TEM/PCM Preparation and Analytical Requirements for Field Samples:

Medium Code	Medium, Sample Type	Preparation Details				Analysis Details			Applicable Laboratory Modifications
		Investigative? (a)	Indirect Prep? (a,b)		Filter Archive? (b)	Method	Recording Rules	Analytical Sensitivity/ Stopping Rules	
			With Ashing (b)	Without Ashing (b)					

(a) See LB-000053 for additional details
(b) See most current version of EPA-LIBBY-08 for preparation details

TEM/PCM Preparation and Analytical Requirements for Quality Control Samples:

Medium Code	Medium, Sample Type	Preparation Details			Analysis Details			Applicable Laboratory Modifications
		Indirect Prep?		Filter Archive?	Method	Recording Rules	Stopping Rules	
		With Ashing	Without Ashing					
	Field Blank	No	No					
	Lot Blank	No	No					

PLM Preparation and Analytical Requirements:

Medium Code	Medium, Sample Type	Preparation Method	Analysis Method	Applicable Laboratory Modifications

Laboratory Quality Control Frequencies:

TEM: Lab Blank – ____%
Recount Same – ____%
Recount Different – ____%
Verified Analysis – ____%
Repreparation – ____%
Interlab – ____%

PLM: Lab Duplicate – ____%
Interlab – ____%

Requirements Revision:

Revision #:	Effective Date:	Revision Description

Analytical Laboratory Review Sign-off:

- ☐ Batta [sign & date: _____]
- ☐ EMSL-Libby [sign & date: _____]
- ☐ EMSL – Westmont [sign & date: _____]
- ☐ EMSL – Beltsville [sign & date: _____]
- ☐ ESAT [sign & date: _____]
- ☐ Hygeia [sign & date: _____]
- ☐ MAS [sign & date: _____]
- ☐ RESI [sign & date: _____]

[Checking the box and initialing above indicates that the laboratory has reviewed and acknowledged the preparation and analytical requirements associated with the specified SAP.]