LIBBY OU3
PILOT STUDY DESIGN

STUDY 3A
EVALUATION OF FIELD FILTRATION OF WATER SAMPLES

October 20, 2010

APPROVAL PAGE

This Libby OU3 Pilot Study Design, Study 3A, Evaluation of Field Filtration of Water Samples is approved for implementation.

Bonita Lavelle
Remedial Project Manager, Libby OU3

10/20/10
Date

1.0 PROJECT OVERVIEW

1.1 Purpose of This Document

This document provides the design of a Pilot Study that is needed to select and optimize a method for the collection and analysis of total and free Libby Amphibole (LA) fibers in water samples that may be needed as part of the Remedial Investigation and Feasibility Study for Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site.

1.2 Project Management and Organization

Project Management

The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Dick Sloan. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC) for performance of a Remedial Investigation/Feasibility Study (RI/FS) at OU3 of the Libby Asbestos Site. Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

Technical Support

EPA will be supported in this study by SRC, Inc. SRC has provided support to EPA in the development of this study design, and will provide additional support in the evaluation and interpretation of the data.

LA Test material

The LA test material used in this study has been prepared by the US Geological Survey (USGS) who is working with EPA under an inter-agency cooperative agreement. The lead scientists at USGS on this project include Greg Meeker and Steve Wilson.

Sample Preparation and Analysis

All sample preparation and analysis tasks required by this study design will be performed laboratories selected and approved by EPA. The laboratory that will be utilized for preparation and analysis of Study 3A water samples is EMSL located in Libby.

Data Management

The data generated by this study will be managed by Lynn Woodbury of SRC. She will be responsible for sample tracking, uploading new data, performing data verification and error checks to identify incorrect, inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed.

2.0 PROJECT BACKGROUND

EPA is currently collecting data needed to support an ecological risk assessment at Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site. One important part of the ecological risk assessment at OU3 is an investigation of the potential effects of Libby Amphibole asbestos (LA) in surface water on aquatic organisms (fish, benthic macroinvertebrates, and amphibians) that reside in the water. This investigation requires the ability to measure the concentration of LA in water with sufficient accuracy to support the characterization of exposure.

This task is complicated by the finding that if the water is not completely sterile, organic matter associated with microbial contamination tends to form, which tends to cause the fibers to clump together. This causes two effects: a) asbestos fibers[1] that are clumped together are difficult to observe and count using transmission electron microscopy, and this may lead to a decrease in estimated concentration of LA in the water, and b) fibers within clumps of organic matter tend to adhere to the walls of the sample bottles, thus decreasing the concentration of fibers in the water. The magnitude of these effects is time-variable, and depends on the amount of organic matter present and the time the sample is held before filtering. Both phenomena (fiber clumping, fiber adherence to container walls) have been observed in studies performed to date by EPA at the Libby OU3 site, including a juvenile rainbow trout toxicity test performed using site waters in

[1] Asbestos structures that are present in water may include fibers, bundles, clusters, and matrix particles. See ISO 10312:1995(E) (ISO 1995) for counting rules for each class of asbestos structure. For simplicity in this document, the word "fiber" is used to refer to all countable asbestos structures.

2009, and analysis of surface water samples collected at stream sampling station LRC-06 in July 2009.

EPA developed Analytical Method 100.1 (EPA 1983) for the analysis of asbestos in water. This method involves treating the water sample with ozone, ultraviolet light, and sonication before filtration. This treatment oxidizes organic material that is present in the water or on the walls of the sample bottle, destroying the material that causes clumping and binding of fibers. Based on studies performed by EPA, this treatment allows good recovery of fibers under a variety of starting conditions.

EPA also performed several studies to investigate whether the treatment resulted in any alterations in fiber size distribution. Studies were performed using water that was contaminated with chrysotile. Based on these studies, the authors concluded that the fiber length distributions in water were not changed by various treatments.

A pilot study "Study 1A, Effect of Treatment on LA Fiber Integrity" will be conducted in support of the Libby OU3 remedial investigation and ecological risk assessment to investigate whether the treatments utilized in EPA Analytical Method 100.1 (sonication and ozone/UV treatment), alone or in combination, results in a significant increase in fiber concentrations or skewed fiber size distribution when applied to suspension of LA in water.

If Study 1A results support the conclusion that EPA Analytical Method 100.1 allows successful analysis of the total amount of asbestos present, that method will be utilized in the analysis of total LA in water samples from OU3. However, in some cases, it may also be desirable to measure the concentration of free fibers that are present. This may be done simply by filtering the water sample without treatment, and counting only free fibers that are present on the filter.

In this approach, the details of how the water is handled before filtration may be important. For example, if the water sample is placed in a bottle for transport to the laboratory for filtration, it is possible that additional clumping might occur and/or that binding of clumps to the bottle wall might occur before filtration occurs. If so, this could yield results that are not representative of the true condition of the water at the time of sampling.

One way to avoid this potential problem is to filter the water sample directly at the site of collection, and never place it in a bottle for transport to the laboratory. However, preparation of filters that are suitable for analysis is not simple, even under laboratory conditions. Care must be taken to exclude potential sources of contamination, and to ensure that particle loading is even and uniform across the filter. Achieving these goals under field conditions is even more difficult. The purpose of this study is to investigate the feasibility of field filtration of water samples from streams and ponds in OU3.

3.0 STUDY OBJECTIVES

This study is designed to evaluate an approach for direct filtration of water samples in order to allow measurement of free LA fibers as well as organic clumps in samples of water. This method may be applicable both to field samples collected from OU3, and also to samples associated with laboratory-based toxicity testing.

4.0 STUDY DESIGN

This study will be performed in two phases as described below.

Phase 1 – Laboratory Test

Test Material

The LA test material to be utilized in this study was collected from the Libby mine by USGS in 2007. Samples of LA from the mine were mixed, crushed, and ground to a "raw" sample by USGS in the laboratory at the Federal Center in Denver, Colorado. This material is referred to as "Libby 2007 Raw".

The USGS will ship three aliquots of this material, each approximately 1 gram in mass, to the following laboratory:

EMSL Analytical
107 West 4th St.
Libby, MT 59923

Phone: 1-406-293-9066

Laboratory Protocol

Once the test material is received by the laboratory, the study will be performed as follows:

1. Prepare a sterile stock solution of LA by weighing approximately 10 mg test material and adding it to 1 L of ozone-treated laboratory water in a glass cylinder. Mix thoroughly by stirring with a glass rod. Allow the suspension to settle for approximately 30 minutes. Remove 10 mL from the upper portion of the cylinder and dilute this to 500 mL using ozone-treated laboratory water. Mix thoroughly. It is expected that the concentration in this dilution will be approximately 60 million fibers per liter (MFL). However, the exact concentration is not critical, and may be either higher or lower.

2. While stirring the dilution with a magnetic stir bar, remove 10.0 mL and filter through a 25 mm polycarbonate (PC) filter[2] with 0.1 um pore size using a standard laboratory vacuum filtration apparatus, in basic accord with the filter preparation step of EPA Method 100.1 (Section 6.3).

3. While stirring the dilution with a magnetic stir bar, fill a 15 mL syringe with the LA suspension. Expel any air bubbles and adjust the volume to 10.0 mL. Filter the entire 10.0 mL aliquot through a 25 mm diameter syringe filter equipped with a PC filter with a pore size of 0.1 µm. (Note that collection of a water sample using a syringe with a filter disk attached rather than using a pipette and vacuum apparatus is thought to be more easily implemented under field conditions and was suggested by USGS.)

4. Repeat steps 2 and 3 two additional times, resulting in a total of three "standard" filters and three "syringe" filters. The loading on each filter is expected to be approximately 1,500 fibers per mm^2 (about 15 fibers per grid opening).

5. Prepare one filter blank by filtering 10.0 mL of laboratory water through a 25 mm PC filter with 0.1 um pore size using a standard laboratory vacuum filtration apparatus and one blank by filtering 10.0 mL of laboratory water through a 25 mm diameter syringe filter equipped with a PC filter with a pore size of 0.1 µm.

6. For each filter (a total of eight), prepare three transmission electron microscopy (TEM) grids from different parts of each filter in accord with Method 100.1, except that the filter dissolution procedure shall utilize the revised method developed by Chatfield (provided in Attachment A).

7. Analyze a total of 5 grid openings from each of the three grids from each of the three filters in basic accord with ISO Method 10312 (ISO 1995), as modified by Libby-specific laboratory modifications LB-000016A, LB-000019, and LB-000030 (provided in Attachment B).

   Counting Rules

   All amphibole structures (including not only LA but all other asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 µm and an aspect ratio (length:width) ≥ 3:1, will be recorded on the site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheet developed for Libby OU3 to record TEM analysis results for water (provided as Attachment C).

[2] PC filters are used because a) Method 100.1 calls for use of PC filters, and b) based on the experience of the TEM analyst at the EMSL laboratory in Libby, PC filters tend to yield better grid preparations for TEM analysis than MCE filters and are recommended.

Record the structure type and dimensions of each countable amphibole structure. Data recording for chrysotile, if observed, is not required.

Phase 2 – Simulated Field Test of Syringe Filters

If the syringe filters prepared under optimal conditions (as described above) are either unevenly loaded and/or yield results (loadings) that are not equivalent to standard laboratory filters (see data analysis methods in Section 7), proceed to Phase 3 (below). If the syringe filters prepared under optimal conditions are evenly loaded and yield results that are equivalent to standard laboratory filters, the next step is to determine if evenly loaded filters with accurately known volumes of water can be prepared under simulated field conditions. This will be done by collecting 3 syringe filters and one blank syringe filter using the same approach as described above, except as follows:

1. The person collecting the samples must be dressed in the full level of personal protective equipment (PPE) that will be worn by field teams in OU3.

2. The water sample must be placed on the floor before syringe samples are withdrawn.

After preparing three syringe filters and one blank syringe filter under simulated field conditions, analyze and evaluate each filter as described in steps 6-8 above.

Phase 3 – Use of Vacuum Filtration in the Field

If the testing of syringe filters in Phase 1 or Phase 2 indicates that use of syringe filters will provide reliable results, Phase 3 will not be needed. However, in the event that results from either Phase 1 or Phase 2 testing indicates that that use of syringe filters will not provide reliable results, it will then be necessary to test the use of a more traditional vacuum filtration device in the field. The details of a Phase 3 study to test the ability to generate reliable filters in the field using vacuum technology will be developed at a later time, if needed.

5.0 QUALITY CONTROL

There are two types of laboratory-based quality control analyses for TEM that will be performed during this pilot study.

Laboratory Blank –As noted above (see Step 5 of the test protocol), there will be one filter blank prepared by filtering 10.0 mL of laboratory water through a 25 mm PC filter with 0.1 um pore size using a standard laboratory vacuum filtration apparatus and one filter blank prepared by filtering 10.0 mL of laboratory water through a 25 mm diameter syringe filter equipped with a PC filter with a pore size of 0.1 µm. A total of 5 grid openings from each of three grids (15 grid openings total) should be analyzed for each of these two filters using the same procedure as used

for investigative samples. There shall be no asbestos structure of any type detected in an analysis of 15 grid openings of either filter. 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 - A recount is an analysis where TEM grid openings are re-examined after the initial examination. A *Recount Different* (RD) is a re-examination of the specified grid openings by a different microscopist within the same laboratory than who performed the initial examination. For the purposes of this pilot study, two RD analyses should be performed by randomly selecting one sample filtered using a standard laboratory vacuum filtration apparatus and one sample filtered using a syringe equipped with a PC filter. LB-000029B (provided in Attachment A) summarizes the acceptance criteria (and corrective action) for RD analyses.

6.0 SAMPLE DOCUMENTATION

Results for each sample will be recorded on the site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheet developed for Libby OU3 to record TEM analysis results for water (provided as Attachment B). All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

The laboratory will also maintain a logbook. This logbook will provide information on any deviations from the study design, as well as information on instrument preparation, calibration, and maintenance logs, and any analytical difficulties or quality control (QC) issues associated with the samples.

Following completion of the study, the laboratory will prepare a data summary report. This report shall include a case narrative that briefly describes the number of samples, the analyses performed, and any issues identified during the study. This report will also include the raw (hand-written) laboratory bench sheets, instrument printouts of sample results (e.g., spectra, micrographic photos, and diffraction patterns), and laboratory QC sample results.

All TEM EDDs and a scanned copy of the study logbook and data summary report will be submitted to EPA's technical contractor (SRC) electronically by e-mail to LibbyOU3@srcinc.com.

7.0 DATA EVALUATION

The evenness of filter loading on each filter will be evaluated using the statistical chi-square test described in Annex F2 of ISO (1995). (Note: The EDD spreadsheet provided in Attachment B is designed to perform this test automatically.)

The filter loading (s/mm^2) on the standard filters will be compared to the loading on the syringe filters using the Poisson ratio test (Nelson 1982) to determine if there are any meaningful differences.

8.0 REFERENCES

EPA. 1983. Analytical Method for Determination of Asbestos in Water. U.S. Environmental Protection Agency, Environmental Research Laboratory, Office of Research and Development, Athens GA. EPA-600/4-83-043. September, 1983.

ISO. 1995. International Organization for Standardization. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

Nelson WB. 1982. Applied Life data Analysis. Wiley Interscience, Hoboken NJ, pp. 441-443.

LIBBY OU3
WATER PILOT STUDY 3A

ATTACHMENT A

IMPROVED METHOD FOR PREPARATION OF TEM GRIDS FROM POLYCARBONATE FILTERS

This page intentionally left blank to facilitate double-sided printing.

CHATFIELD TECHNICAL CONSULTING LIMITED

2071 Dickson Road
Mississauga, Ontario
CANADA L5B 1Y8
Telephone: (905) 896-7611
Fax: (905) 896-1930

Presentation
Inter/Micro-94

An Improved Method for Preparation of TEM Specimen Grids from Polycarbonate Membrane Filters Used for Particulate and Fiber Analysis

Eric J. Chatfield
Chatfield Technical Consulting Limited
2071 Dickson Road
Mississauga, Ontario
Canada L5B 1Y8

Direct-transfer preparation of TEM grids from polycarbonate (PC) filters is a simple procedure in which a carbon extraction replica is produced from the filter surface. Particles are deposited on the surface of the filter by filtration. A portion of the filter is then placed in a carbon evaporator, and a thin film of carbon is deposited on the filter surface. A small area of the carbon-coated filter is placed on a TEM support grid, and the filter medium is dissolved away leaving the original deposit trapped in the carbon film on the TEM grid.

Excessive heating of the surface of PC filters during carbon deposition has been found to render the surface layers of the polymer insoluble in the commonly-used solvents, such as chloroform. Over the last 10 years, PC filters have also varied in their resistance to complete dissolution. It has become clear that the majority of TEM specimen preparations produced by chloroform dissolution of PC filters have a residual layer of undissolved polymer, which reduces particle image contrast and limits the visibility of electron diffraction patterns. Both effects increase the strain on the TEM operator and

introduce the potential for a negative bias in the analysis.

The top micrograph shows an example of a TEM specimen recently prepared by a laboratory accredited by NVLAP for analysis of asbestos in air samples by TEM. Using specimen grids of this quality, the laboratory proceeded with the analysis and reported results. A chrysotile fiber can be found in the area shown in this micrograph, but the thickness of this specimen imposes an unacceptable limitation on particle visibility. This illustrates an extreme example of incomplete filter polymer dissolution, but almost all preparations using chlorinated hydrocarbon solvents show some degree of residual filter polymer, the amount depending on the carbon coating technique and the procedures used for filter dissolution. Assuming that no excessive heating of the filter surface occurred during carbon evaporation, use of a Jaffe washer for 2 hours, followed by a 20 minute treatment in a condensation washer has usually been found to produce TEM grids of acceptable quality. After this treatment, there will be very little further removal of filter polymer even if the condensation washing is extended significantly. The TEM specimens, however, almost always exhibit the "connected pore" phenomenon, which is caused by undissolved polymer.

A new dissolution procedure has been developed which is based on the use of a mixture of 20% 1-2-diaminoethane and 80% 1-methyl-2-pyrrolidone in a Jaffe washer of the conventional wire mesh bridge design. This solvent mixture completely removes the PC filter polymer in a period of 10 minutes. It yields ideal specimen grids, even from filters that have been excessively heated during carbon evaporation. This TEM specimen preparation procedure allows the use of much thinner carbon films, than is the case for the conventional chloroform dissolution procedure, because the distortion of the PC filter observed during dissolution using chloroform does not occur. The new procedure has been incorporated in the International Organization for Standardization Method ISO 10312 (Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy procedure (Draft) 1993) and in a

new draft method for determination of asbestos fibers in parenteral medicines (Proposed Analytical Method (Draft) Der Verband der Chemischen Industrie, Parenteral Medicines - Determination of asbestos fibres - Direct-transfer transmission electron microscopy procedure, 1994). The bottom micrograph shows an example of a TEM specimen prepared from a polycarbonate filter by the new procedure.

The new dissolution procedure is as follows:

1. prepare a Jaffe washer consisting of a stainless steel mesh bridge in a glass petri-dish;

2. add to the Jaffe washer a sufficient volume of a pre-prepared 20% 1-2-diaminoethane and 80% 1-methyl-2-pyrrolidone mixture, such that the meniscus touches the horizontal underside of the stainless steel bridge;

3. place each TEM grid with a portion of carbon-coated PC filter, carbon side facing upwards, on to the Jaffe washer mesh. Cover the dish and allow to stand for approximately 10 minutes;

4. transfer the stainless steel mesh with the grids to a second, empty petri-dish;

5. to the second petri-dish, add either distilled water or reagent alcohol (ethanol) until the meniscus touches the horizontal underside of the stainless steel bridge. Allow to stand for approximately 10 minutes;

6. remove the stainless steel bridge from the Jaffe washer, place it on a paper towel, and allow to dry. The drying process can be accelerated by absorbing the excess water or ethanol from the underside of the mesh using paper towel.

The choice of distilled water or reagent alcohol as the final washing

solvent depends on the application or the preference of the analyst. If it is required to retain water-soluble particle species such as gypsum on the final TEM specimens, then alcohol must be used. For water sample analysis, or for indirect TEM specimen preparations from air samples in which water has been the dispersal medium, either water or reagent alcohol may be used. For direct-transfer air sample preparations in which it is desired to remove gypsum fibers from the preparation, water should be used. In analyses of air samples for asbestos, thin gypsum fibers may be present in large numbers, and these can slow down the TEM examination because each fiber must be identified. Selective removal of gypsum from such samples is advantageous, in that the TEM examinations proceed more rapidly after non-asbestos fibers have been removed, even if carbon replicas of the original particles still remain visible in the image.

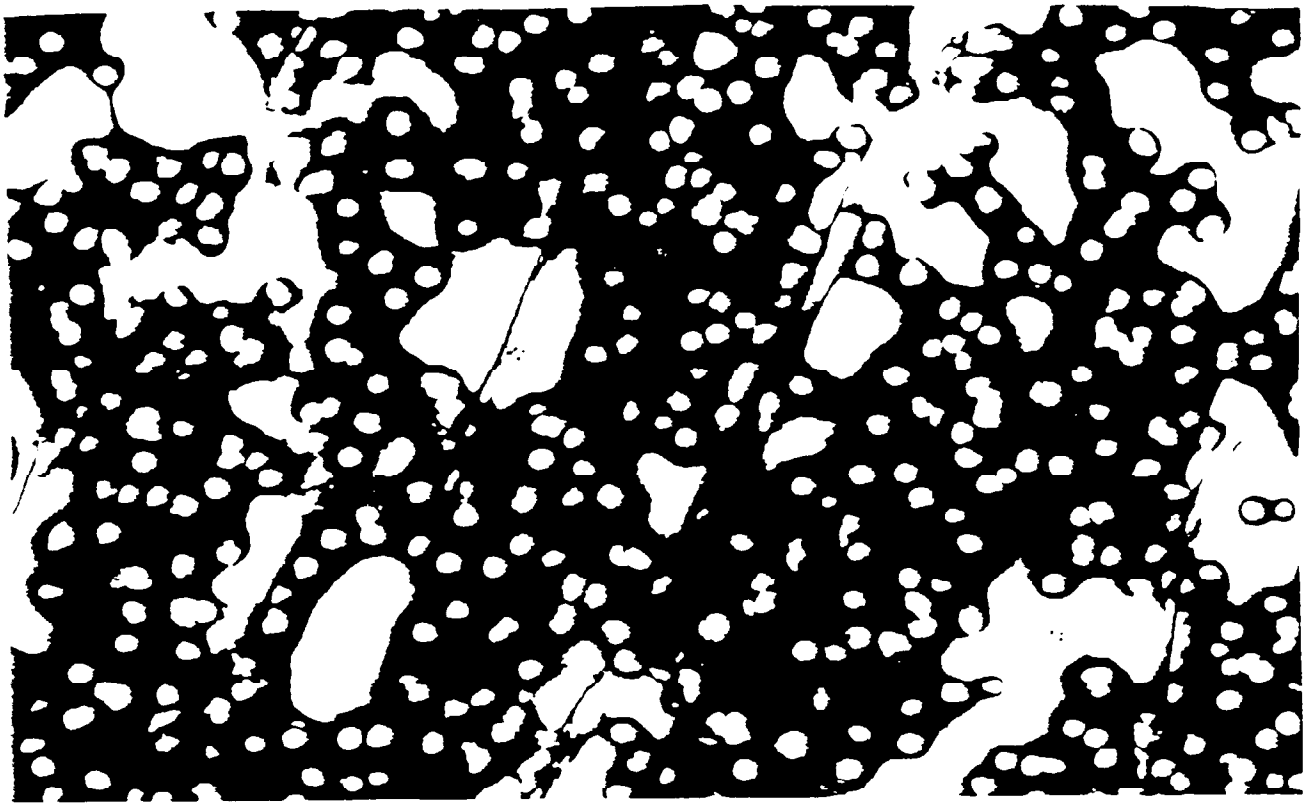
Some types of copper grid are slightly attacked by the solvent. It is not recommended, nor is it necessary, to allow grids to remain in contact with this solvent for more than approximately 10 minutes, and substantially less dissolution time may be sufficient in some cases. A slight blue color in the solvent after use will indicate if any chemical attack on the grids has occurred, but this has not presented any problems. If this attack is of concern, or if for operational reasons the grids must remain in contact with the solvent for periods of time longer than approximately 15 minutes, gold grids may be used instead of copper and are not attacked by the solvent.

Using the new procedure, dissolution occurs so rapidly that interference colors, created by the rapidly thinning polymer layer, can be observed only a few seconds after dissolution commences. No distortion of the shape of the carbon-coated PC filter portion is seen during dissolution. Starting from a PC filter, ideal TEM specimens can be available for TEM analysis in a period of less than 30 minutes. This is a significant improvement over the conventional methods for preparation of TEM specimens from PC filters. It also represents considerable savings of time over the MCE preparation methods,

bearing in mind the additional time requirements for collapsing of the MCE filter and plasma etching.

Figures: Top: TEM micrograph showing the appearance of a specimen prepared from a 0.2 micrometer pore size PC filter on which chrysotile fibers had been collected. This shows the effects of overheating during carbon evaporation, and incomplete dissolution of the filter polymer by chloroform. Magnification 11,000

Bottom: TEM micrograph showing the appearance of a similar specimen prepared using the new dissolution procedure. Magnification 11,000



LIBBY OU3
WATER PILOT STUDY 3A

ATTACHMENT B

LIBBY-SPECIFIC ANALYTICAL LABORATORY MODIFICATIONS

LB-000016A
LB-000019
LB-000029B
LB-000030

This page intentionally left blank to facilitate double-sided printing.

Request for Modification
to
Laboratory Activities
LB-000016A

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: April 10, 2008

Description of Modification:
Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air and dust samples using ISO 10312. The purpose of the attached is to document historic modifications & clarifications, and provide additional permanent clarifications.

Reason for Modification:
To optimize the efficiency of air and dust 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. Negative implications - comparisons of the Total # of LA structures between historical results and current results may be biased (high or low) due to differences in recording rules with regard to aspect ratio criteria. Positive implications - consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): [All] Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **[SUPERCEDES]** **LB-000016**

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.

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 attached sheets for the description of the TEM-ISO clarifications/modifications.

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.

ISO 10312 MODIFICATIONS AND CLARIFICATIONS

1. **Overloading Modification:**

   The ISO method requirement states that 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. **Indirect Preparation of Air Samples Modification:**

   ISO 10312 is a direct preparation method. If the sample is visibly overloaded or contains loose debris, it will be prepared indirectly according to procedures provided in SOP EPA-Libby-08. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations will be adjusted to contain a dilution factor. This indirect preparation procedure will enable the capture of data from samples that otherwise would be rejected.

3. **Stopping Rule 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.

4. **Abundant Chrysotile Modification:**

   If abundant chrysotile is present, the chrysotile count may be terminated in accordance with the counting rules specified in LB-000039.

5. **Structure Counting and Recording Modifications and Clarifications:**

   a. Non-asbestos material (NAM) structures are not being recorded, unless identified as a "close call" (see LB-000066 for details). This project-specific modification stems from the need only to quantify levels of contaminants of concern (i.e., asbestos) at a given sample location.

   b. Recording rules will be as described in the ISO method except that the aspect ratio requirement will depend upon the classification of the sample as "investigative" or "non-investigative", as specified in LB-000053. If samples are classified as investigative, the aspect ratio requirement will be 3:1, rather than 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise. Thus, fibers (either individual fibers or fibers within disperse matrices or clusters) shall only be recorded if the length is greater than or equal to 0.5 um and the aspect ratio is greater than or equal to the appropriate criterion. Bundles shall only be recorded if they contain individual constituent fibers with an aspect ratio greater than or equal to the appropriate criterion. The aspect ratio criterion does not apply to compact clusters, compact matrices, or residuals. The overall aspect ratio of a bundle, compact cluster, compact matrix, or residual may have any value.

   c. The definition of a PCM equivalent (PCME) structure is as follows: Any fiber, bundle, matrix, or cluster with an aspect ratio of 3:1 or greater, length longer than 5 um, and width greater than or equal to 0.25 um.

   d. The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual sub-structures associated with the matrix or cluster will be recorded as described in the ISO method.

   e. Structures that intersect a non-countable grid bar (i.e., top and left grid bars) will be recorded on the count sheet but excluded from the structure density and concentration calculations. These non-countable structures will be denoted with a zero in the Total column.

   f. 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.

g. If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

h. See Attachment A for detailed examples of how to record specific structure types that may be encountered in Libby samples.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

OVERVIEW OF HISTORICAL RECORDING CRITERIA

At the beginning of the Libby project, analytical laboratories (primarily EMSL and RESI) were following the ISO method with regard to structure recording (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1).

Approximately the time of the Phase 2 Investigation (late Spring 2001), project laboratories were instructed by Chris Weis (EPA, Region 8) to record all structures regardless of minimum length or aspect ratio. This recording rule change enabled data users to gain a better understanding of the dimension attributes for structures at the Libby site and allowed for the calculation of PCM equivalent (PCME) structures. In the ISO report generated by the TEM EDD spreadsheet, structures with an aspect ratio less than 5:1 were counted in Bin A and structures with a length less than 0.5 um were counted in Bin B. Also at this time, the TEM EDD spreadsheet was modified to allow for the capture of the raw structure data, as entered from the laboratory bench sheet, into the Libby site database.

Although it is uncertain exactly when the recording rules changed after the Phase 2 Investigation, based on analyst interviews, project laboratories reverted back to following the ISO method (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1) beginning approximately December 2001, unless specifically requested otherwise in project-specific SAPs and/or QAPPs (e.g., the Supplemental Remedial Investigation samples collected under the SQAPP specified an aspect ratio criterion of greater than or equal to 3:1).

Laboratory modifications LB-000016B through 16F (provided as Attachment B) document the historical laboratory and analyst-specific deviations in recording/counting rules for ISO based on analyst interviews conducted in August and September 2006.

Beginning August 29, 2006, all project laboratories began utilizing an aspect ratio criterion of 3:1, unless specifically requested otherwise.

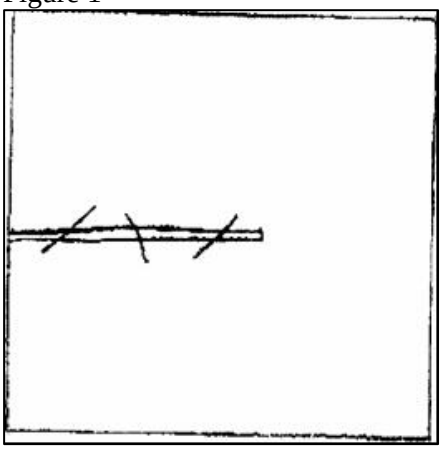
Preparation techniques and recording rules were further refined as part of LB-000053 (effective date: December 21, 2006), whereby all Libby samples were classified as “investigative” and “non-investigative”. Samples classified as “investigative” were to utilize an aspect ratio criterion of 3:1, and samples classified as non-investigative were to utilize an aspect ratio criterion of 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise.

Because of the differences in recording rules for ISO analyses across time, data users should be cautious when making comparisons across samples based on the total number of LA structures. The binned metric of total number of LA structures may differ depending upon the recording rule in place at the time.

ATTACHMENT A

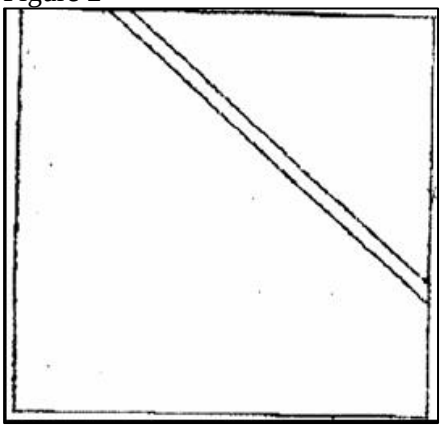
STRUCTURE-SPECIFIC EXAMPLES OF DATA RECORDING

Figure 1



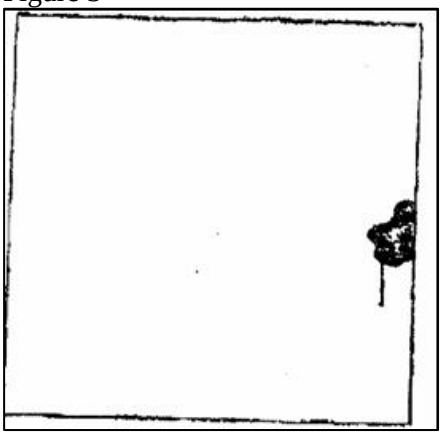
Count as three fibers (F). The large structure is excluded because it crosses a non-countable grid bar (left grid bar).

Figure 2



Count as one fiber (F). Record the length as that observed without doubling.

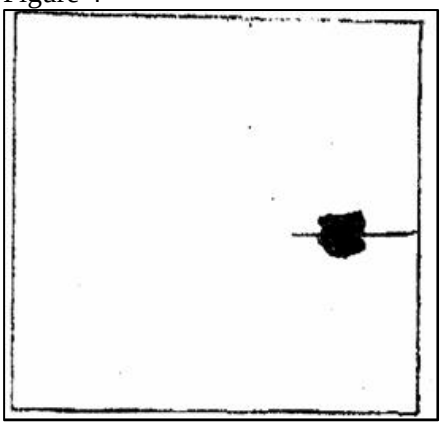
Figure 3



Count as disperse matrix, consisting of one fiber longer than 5 um.

Record as MD11, followed by one fiber (MF). When recording the MF, do not double the length.

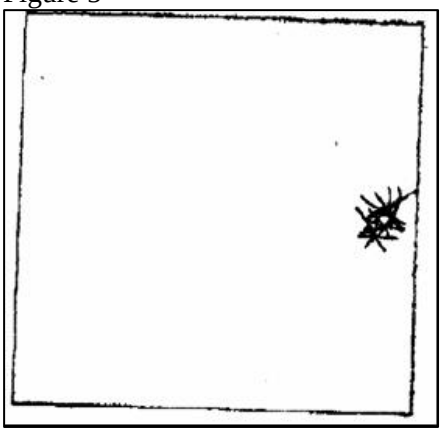
Figure 4



Count as disperse matrix, consisting of one fiber longer than 5 um.

Record as MD11, followed by one fiber (MF). When recording the MF, double the length of the observed fiber.

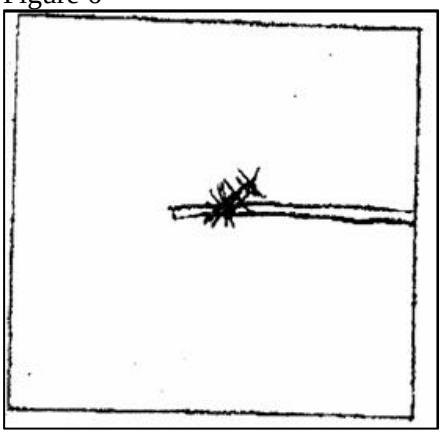
Figure 5



Count as one compact cluster containing more than 9 fibers, which includes one fiber that is longer than 5 um.

Record as CC+1. When recording the CC, record the length of the cluster as double the length of the observed fiber longer than 5 um.

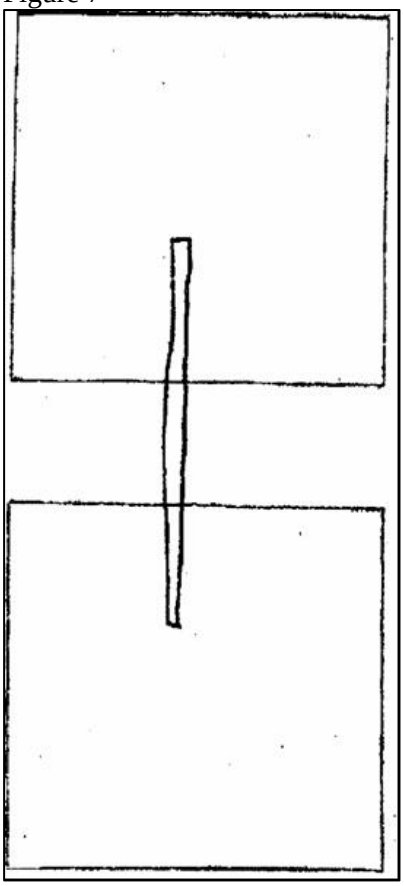
Figure 6



Count as disperse cluster, consisting of one fiber which is longer than 5 um and one compact cluster residual containing more than 9 fibers.

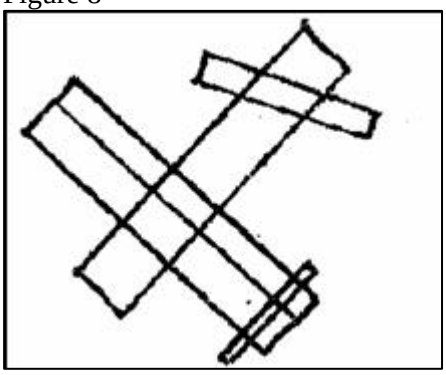
Record as CD+1, followed by one CF and one CR+0. When recording the CF intersecting grid bar, double the length.

Figure 7



Count as one fiber (F). Record the actual length, including protrusion into adjacent grid opening.

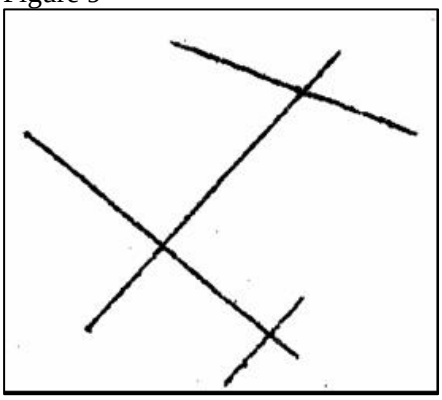
Figure 8



Count as disperse cluster, consisting of four fibers each longer than 5 um.

Record as CD44, followed by four CFs.

Figure 9



Count as disperse cluster, consisting of four fibers each longer than 5 um.

Record as CD44, followed by four CFs.

ATTACHMENT B

LABORATORY AND ANALYST-SPECIFIC DEVIATIONS IN ISO 10312 RECORDING AND COUNTING RULES PRIOR TO AUGUST 2006 (LB-000016B through 16F)

LB-000016B - Batta
LB-000016C - EMSL
LB-000016D - Hygeia
LB-000016E - MAS
LB-000016F - RESI

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
>
>
>

This page intentionally left blank to facilitate double-sided printing.

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.

---

[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

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

---

[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

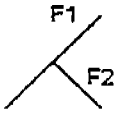
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

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

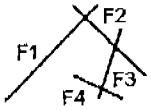
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

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

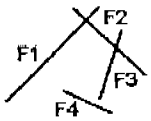
c

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

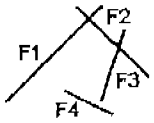
a

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

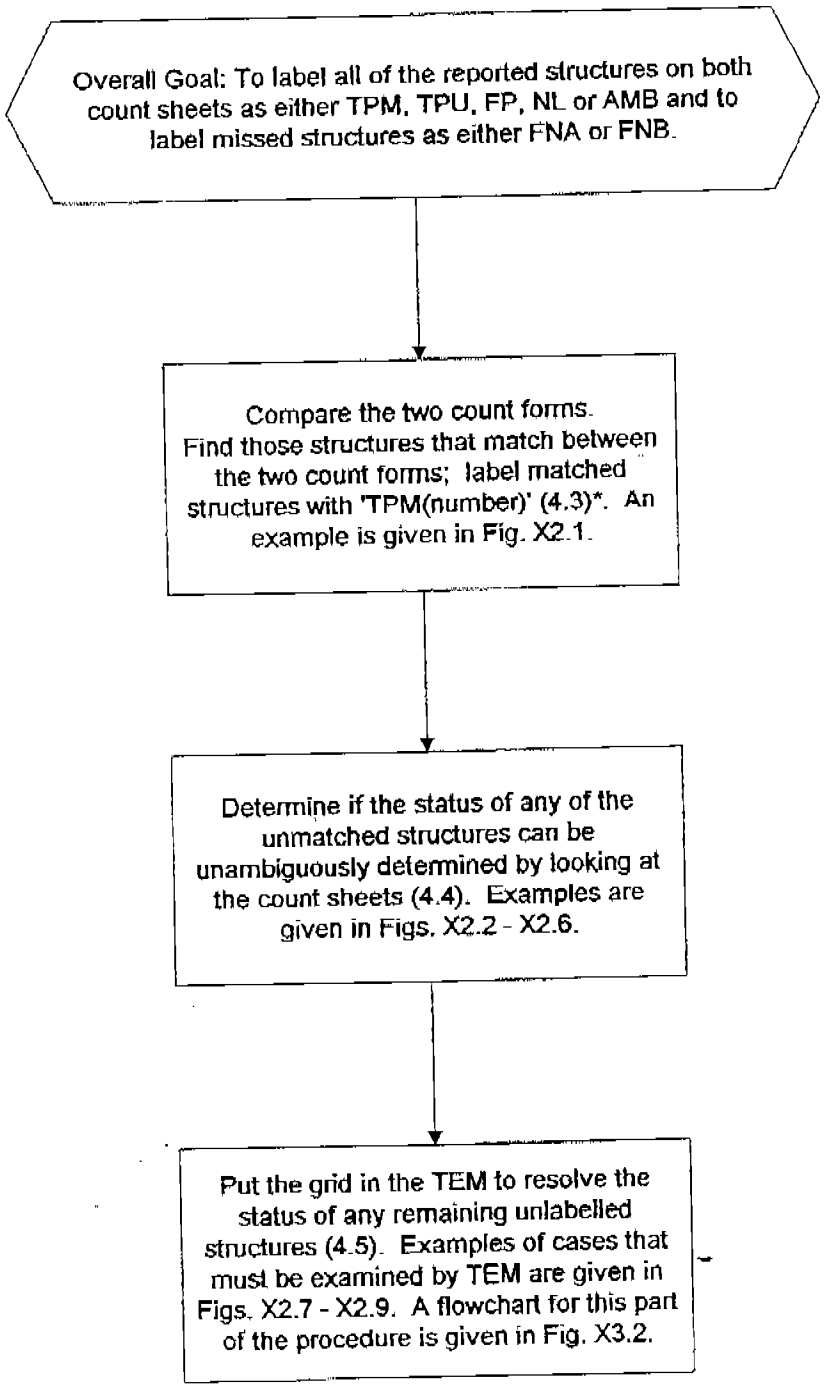


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.
*Numbers in parentheses in each block refer to the item number in the procedure.

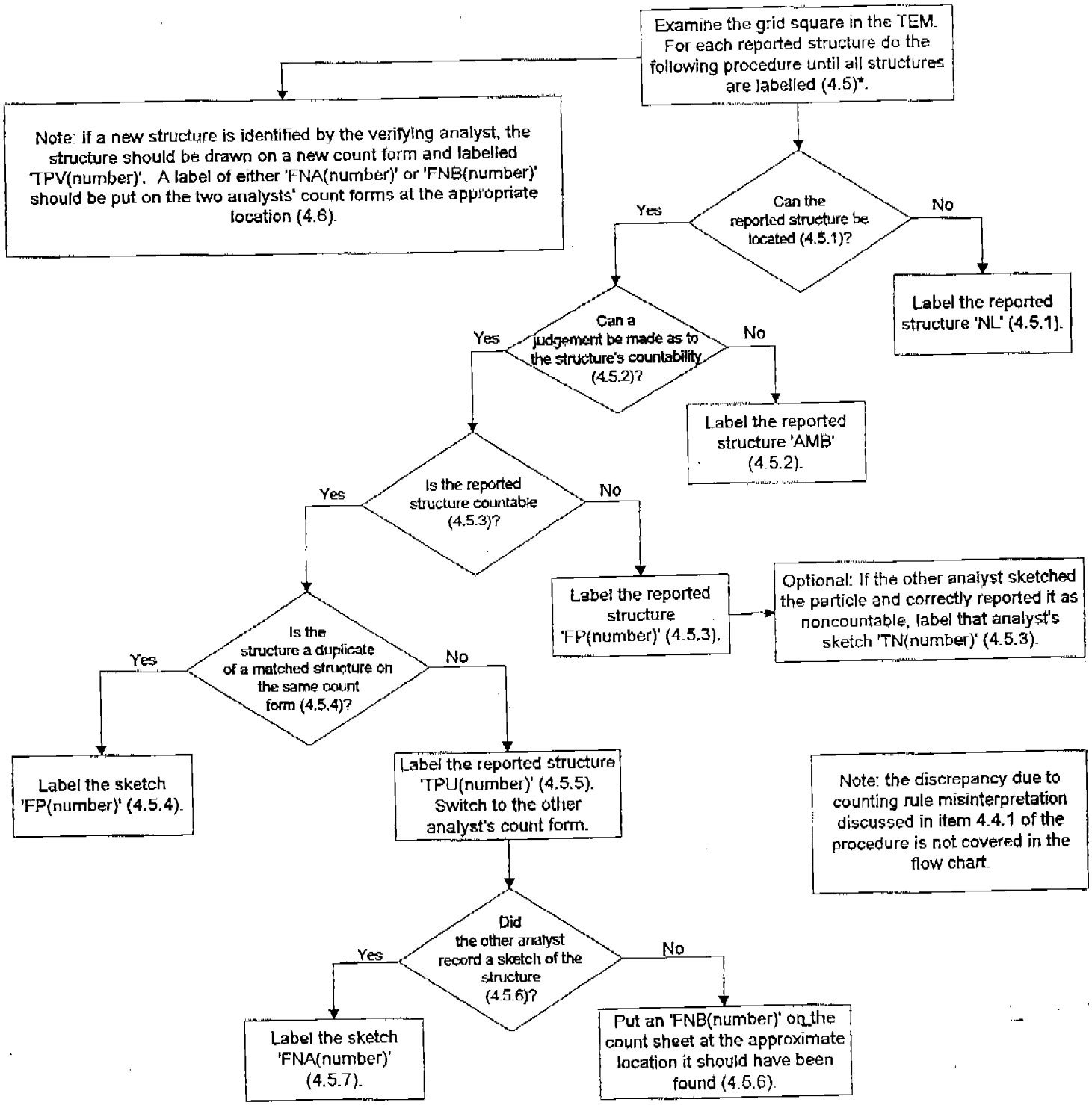


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

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

This page intentionally left blank to facilitate double-sided printing.

Instructions for Form 1

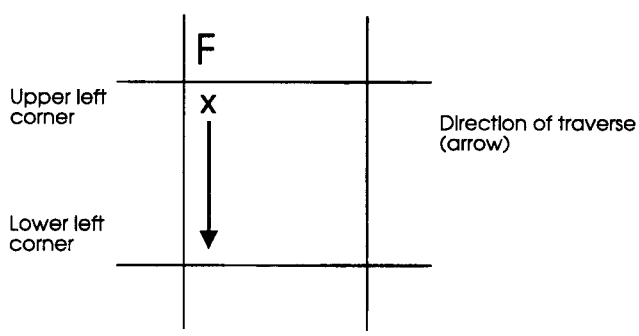
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

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):



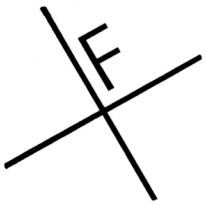
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

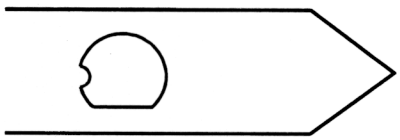
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

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:

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)

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.

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)

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)

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
Subject: EPA Comments: LB-000030 (Draft for review/comment)

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)

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.

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)

LIBBY OU3
WATER PILOT STUDY 3A

ATTACHMENT C

ELECTRONIC DATA DELIVERABLE (EDD) SPREADSHEET FOR TEM ANALYSIS OF WATER SAMPLES

(see file "Water TEM v6_OU3.xls")